

White Paper on NRIM

March, 2000

Volume 2 Research Activities

National Research Institute for Metals

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PREFACE

Research work in NRIM has been carried out based on the long-term plan which has been revised every 5 years. The 5th Long-term Plan started from April 1997. According to the Plan, the promotion of R&D for fundamental technologies and materials characterization in materials science is selected as one of the most important policies. The research organization for this R&D is called as the Research Core Branch which consists of 5 Divisions, 5 Research Groups and 4 Research Teams.

The research in advanced physical fields, which needs large-scale research facilities, is also stressed in the 5th Long-term Plan. The research has been carried out at the Center for Advanced Physical Fields, which consists of 3 Stations. In the Center, the COE Project and cooperative research have been carried out using the large scale research facilities.

In the 5th Long-term Plan, “Research on the Structural Material for the New Century” has been selected as a social-needs-oriented research project. The project has been carried out in cooperation with industry and universities at the Frontier Research Center for Structural Materials, which consists of 3 Stations. The task force system has been employed to promote the R&D efficiently, and the system has been carried out by cooperation among the Stations.

The promotion of the research for constructing databases of fundamental materials information is also stressed in the 5th Long-term Plan. With the databases we are constructing a knowledge foundation for materials science, accessible through the Internet.

This booklet on the Research Activities in the NRIM shows the details of research activities of all the Research Divisions, Research Groups, Research Teams, Stations in Centers and all the additional research projects from April 1, 1997 to March 31, 1999. Their goals, engaged members and outputs are also included.

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CHAPTER 1: Research for Materials Science

The fundamental research for materials science has been promoted at the five Divisions in the Research Core Branch which was established on April 1, 1997. They consist of the Materials Physics Division, the Physical Properties Division, the Computational Materials Science Division, the Mechanical Properties Division and the Materials Processing Division. Research cooperation with the Stations in the Frontier Research Center for Structural Materials and the Center for Advanced Physical Field has been extensively carried out in each Division. Collaboration with other research organizations has been mainly supported by the Special Coordination Funds for Promoting Science and Technology.

1.1 Materials Physics Division

Muneyuki Amano (Director)

1.1.1 Research work

Following the 5th Long-term Plan, the R&D of high techniques for materials characterization have been promoted at the Materials Physics Division. Efforts for introducing new technologies for materials characterization and advanced research facilities for physical and chemical analyses have been made. Examples of the output are as follows.

- (1) It is possible to map out elemental distributions in alloys in a three-dimensional real space with a near atomic scale resolution by using a three-dimensional atom probe.
- (2) The physical properties and structural transition of various compounds under ultra-high pressure can be measured by using a diamond-anvil technique.
- (3) The surface structure and electronic properties of semiconductors and quasicrystals can be studied by using angle-resolved photoelectron spectroscopy.
- (4) High precision chemical analysis of metallic elements in ppb order is possible by using skillful sample preparation methods and ICP-MS.

The members of this Materials Physics Division, as at March 31, 1999, consisted of 31 researchers, 2 STA fellows, 2 domestic research fellows, 2 special technical staff, 2 technicians, 1 graduate student, 2 secretaries and 5 guest researchers.

The output of the research activities has been markedly increased since the start of the 5th Long-term Plan. The number of publications, including submitted papers, was

140, and the number of patent applications was 7 between April 1, 1977 to March 31, 1999. The details are shown in the research activities of each Laboratory.

Support work in physical and chemical analyses is carried out in the 5th Laboratory. For the support of physical analyses, facilities such as EPMA, TEM, SEM and XRD are kept in good condition by 4 researchers, 2 technicians, and 1 special technical staff. Researchers can use these facilities with the help of support staffs. For the support of chemical analyses, 11 researchers and 1 special technical staff have been engaged, by using ICP-AES, ICP-MS, GF-AAS, GD-MS, XRFA, etc. The number of samples for chemical analyses requested from researchers in 1997 and 1998 fiscal year was 934 and 1177, respectively. The total number of elements in 1997 and 1998 fiscal year amounted 2461 and 3166, respectively. A very serious problem is the decrease in the number of supporting staff due to retirement.

1.1.2 1st Laboratory

Toshiya Hirata (Head), Takayoshi Kimoto, Masahiko Shimoda

1.1.2.1 Research work

The 1st laboratory is concerned with the three topics described below.

- (1) Some materials, like perovskite compounds and high T_c superconductors, have been studied by Fourier-transform infrared and Raman spectroscopy, paying attention to the behavior of optic phonons to understand the structural changes and physical properties of materials.
- (2) Surface structures and electronic properties of semiconductors, high T_c superconductors and quasicrystals are under investigation by X-ray photoelectron diffraction (XPD), which provides useful information to address the subject issue; XPD has been applied to investigate the sulfur-terminated GaAs (001)-(2x6) surface in detail.
- (3) With the development of a high-speed CCD camera and related techniques, an attempt has been made to improve the resolution of imaging in TEM at low temperatures. The structural evolution of high T_c superconductors at low temperatures was significantly elucidated by the improved technique; based on the intensities in electron diffraction, a method to determine long-range order parameters and/or lattice distortions was successfully constructed.

<Research themes>

- (1) Infrared and Raman Spectroscopic Study of Metallic Oxides (General Research: April 1995 to March 1998, Toshiya Hirata)

- (2) Composition, Temperature and Pressure-Induced Structural Changes and Physical Properties of Materials (General Research: April 1998 to March 2001, Toshiya Hirata)
- (3) Two Dimensional Photoelectron Spectroscopic Studies on Surface Structures and Properties (General Research: April 1995 to March 1998, Masahiko Shimoda)
- (5) Angle-Resolved Photoelectron Spectroscopy Study on Surface Structure and Electron Properties (General Research: April 1998 to March 2001, Masahiko Shimoda)
- (6) Determination of Order Parameters in Alloys from Electron Diffraction Intensities Using CCD Camera System and it's Application to Examination of Ordering Process (General Research: April 1993 to March 1995, Takayoshi Kimoto)
- (7) In-Situ TEM Observation and Structure Analysis of High T_c Superconductors for Fusion Reactor at Low Temperature (Nuclear Energy Research: April 1994 to March 1999, Takayoshi Kimoto)
- (8) Coating of Photocathode and Fundamental Investigation of Photoelectron Emission from Laser Illuminated Photocathode for TEM (General Research: April 1999 to March 2002, Takayoshi Kimoto)

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- (3) T. Hirata: Pressure, Temperature and Concentration Dependences of Phonon Frequency with Variable Grüneisen Parameter: Fits to the Raman-Active E_g Mode in TiO_2 and $\text{Ti}_{1-x}\text{Zr}_x\text{O}_2$ ($x \leq 0.1$), *Phys Stat. Sol. (b)*, **209**, 17(1997).
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- (5) T. Hirata: Oxygen Concentration Dependence of Raman Active Phonons with Variable Grüneisen Parameter in $\text{YBa}_2\text{Cu}_3\text{O}_x$, *Physica B*, **263-264**, 822(1999).
- (6) T. Hirata: Orientation Dependence of Infrared Spectra on Thermal Oxidation and Subsequent Etching of Single Crystal Si, *Sol. Stat. Commun.*, **111**, 421(1999).
- (7) M. Shimoda, S. Tsukamoto, N. Koguchi: Photoelectron and Auger Electron Diffraction Studies of a Sulfur-Terminated GaAs (001)-(2x6) Surface, *Surface*

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- (10) T. Kimoto, W. Sun: Improvement of Low-Temperature TEM Imaging of (Bi,Pb)₂St₂Ca₂Cu₃O₁₀ with High-Speed CCD Camera, 6th Asian-Pacific Conference on Electron Microscopy, Hong Kong, July 1-5, 1996.
- (11) T. Kimoto, T. Takeda: Long-Range Order Parameter near Grain Boundary in Cu₃Au and B-Doped Ni₃Al Determined from Electron Diffraction Intensities Detected by CCD Camera, 6th Asia-Pacific Conference on Electron Microscopy, Hong Kong, July 1-5, 1996.
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Oral Presentation: 3

- (1) International Conference: 3
- (2) Domestic Conference: 0

Patent Application

- (1) Laser-Irradiation Electron Gun: T. Kimoto, The Netherlands, 13 July 1998, No10002246.

1.1.3 2nd Laboratory

Akiyuki Matsushita(Head), Jinhua Ye, Takashi Naka, Jie Tang, Hideki Abe, Susumu Ishida (domestic research fellow), Koichi Maruyama (domestic research fellow), Mitsuru Uehara (guest researcher), Yu Yamada (guest researcher)

1.1.3.1 Research work

We have been pursuing several research projects in high pressure physics, centering around the physical properties of magnetic intermetallic compounds. Much of our

effort centers on an attempt to find novel phenomena which are produced with various kinds of electronic interactions acting between electrons in condensed matters. We are aggressively pursuing interdisciplinary research work by collaborating with researchers in different fields of study, such as synthetic chemistry, organic chemistry and so forth.

One of the current research interests focuses on the superconductivity of $\text{Pr}_1\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ (Pr123). This compound has been considered to be a non-superconductor for about 10 years. We found that the pressure coefficient of T_c , dT_c/dP is extremely large for the superconducting Pr123[2]. This is in contrast to the previous results for (Y,Pr)123, in which large negative values of dT_c/dP have been reported. It is surprising that a compound with the same chemical composition shows an opposite pressure dependence. At present we are investigating the crystal structure of the superconducting Pr123 in detail.

In the research on 3d-intermetallic compounds we recently found some interesting phenomena that magnetic or other physical properties can be controlled with heat treatment. One concerns the ferromagnetism in Zr-Fe-Co-Si compounds. This compound is non-magnetic under normal conditions, but becomes ferromagnetic by cooling under a weak magnetic field. We are applying for a patent. Another concerns the physical properties of a 3d intermetallic compound, Fe_2VAl . This material exhibits interesting properties which resemble those of heavy fermion materials, such as an enhancement of specific heat at low temperatures, semiconducting temperature dependence of electrical resistivity, and so forth. Recently we found that these properties are extremely sensitive to heat treatment. For example, a quenched sample exhibits a large enhancement of specific heat and does not exhibit magnetic ordering, while the enhancement of specific heat is suppressed and a ferromagnetic transition is observed for the samples which are annealed at low temperatures. These differences of physical properties are closely related to the mixture among the Fe and V atoms.

Our program also encompasses developing high pressure techniques. In this past few years our efforts in this area have been put into high-pressure X-ray diffraction techniques. We have constructed a diamond-anvil X-ray diffraction system equipped with a monochromator. With this system we can obtain excellent X-ray diffraction data under high pressures which are comparable to that obtained using synchrotron radiation facilities. Now we are constructing a high-pressure and low-temperature X-ray diffraction system. Many interesting phenomena under high pressures are observed at low temperatures. This X-ray diffraction system would be useful to elucidate the origins of novel phenomena found at low temperatures and high

pressures.

<Research themes>

- (1) Control of Electron State by Using High Pressure (General Research: April 1996 to March 1999, Akiyuki Matsushita)
- (2) Angle Dispersive Type Crystal Analysis under Low Temperature and High Pressure (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 1999, Jie Tang)
- (3) Optimization of Conducting Transition Temperature of High Temperature Oxide Super Conductor (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 1999, Jinhua Ye)
- (4) Development Program of New High Pressure Techniques and Investigation of New Magnetic Compounds and their Physical Properties (General Research: April 1999 to March 2002, Akiyuki Matsushita)

1.1.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) J. Ye, T. Shishido, T. Sasaki, T. Takahashi, K. Obara, R. Note, T. Matsumoto, T. Fukuda: Synthesis and Characterization of New Quaternary Borocarbides RRh_2B_2C (R=rare earth), J. Solid State Chem. 133, 77(1997).
- (2) J.Tang, H.Kitazawa, A.Matsushita, T.Matsumoto: High-pressure Study of Antiferromagnetic Order in $CePd_2(Al,Ga)_3$, Physica B 230-232, 208(1997).
- (3) J.Tang, T.Matsumoto, T.Kosaka, T.Matsumura, N.Mori, T.Suzuki: Structural and Electrical Properties of TmTe under High Pressure, Advances in High Pressure Research in Condensed Matter, 419(1997).
- (4) J. Tang, Y. Okada, Y. Yamada, S. Horii, A. Matsushita, T. Kosaka, T. Matsumoto: Pressure Effect on T_c in $RBa_2Cu_4O_8$ (R=Sm, Er, Dy) Superconductors, Physica C 282-287, 1443(1997).
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- (16) S. Ishida, M. Suzuki, N. Mori, S. Todo, N. Shiratori: Ultrasonic Study of the High Temperature Phase of Fe_3O_4 , *J. Phys. Soc. Jpn.*, **67**, 3125(1998).
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- (46) S. Ishida, T. Naka, A. Matsushita, J. Ye, T. Shishido, T. Fukuda: The Physical Properties of the New Quaternary Borocarbides $\text{RRh}_2\text{B}_2\text{C}$ ($\text{R}=\text{Gd, Sm and Nd}$), *Physica B*, in press.

Patent application

- (1) Magnetic-Field-Induced Ferromagnetic Silicide, H. Abe, M. Uehara, A. Matsushita, 1999, H11-110885.

Number of oral presentations: 34

- (1) International conference : 4
- (2) Domestic conference : 30

1.1.4 3rd Laboratory

Kazuhiro Hono (Head), Mitsuhiro Murayama, De Hi Ping, Yagiao Wu (Graduate student), Akihisa Inoue (Guest Researcher)

1.1.4.1 Research work

The main goal of this research group is to obtain better understanding of the underlying mechanisms of mechanical and magnetic properties of metallic materials by characterizing the microstructures with the atomic scale. Atom probe field ion microscopy (APFIM) is the main technique employed in this group, which is capable of analyzing local chemical compositions with an atomic resolution. The laboratory is equipped with an energy compensated time-of-flight atom probe (conventional AP) and a three dimensional atom probe (3DAP), both of which are originally designed and built in the laboratory. In addition, an energy compensated three-dimensional atom probe (EC-3DAP) is now under construction. By making use of a three dimensional atom probe (3DAP), it is possible to map out elemental distributions in alloys with a near atomic resolution. For complementary microstructural characterization, conventional transmission electron microscopy (TEM) and high-resolution electron microscopy (HREM) techniques are also employed. The research subjects conducted in this group includes nanocrystalline magnetic materials, magnetic thin films, amorphous and nanocomposite alloys, microstructures and phase transformations in steels and aluminum alloys and other industrial metallic materials.

<Research themes>

- (1) Atom Probe Microanalysis of Advanced Metallic Materials (Special Research: April 1996 to March 2001, K. Hono)
- (2) The Nanocrystalline and Supercooled Liquid States of Alloys (NEDO International Joint Research Grant: April 1996 to March 1999, K. Hono)

- (3) Phase Transformations under Unusual Environment and of New Materials (Grant-in-Aid for Scientific Research on Priority Areas, The Ministry of Education, Science, Sports and Culture, #287 "The elucidation of microscopic mechanisms of phase transformations: April 1997 to March 2000, K. Hono)
- (4) Development of Microscale Measurement Technique (Grant-in-Aid for Scientific Research in Priority Area on "Nanoscale Magnetism and Transport", No. 281, Ministry of Education, Science, Sports and Culture: April 1997 to March 2000, K. Hono)
- (5) Fundamental Research on the Mechanism of the Formation of Fine Structure in the Next Generation Steels (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 1999, K. Hono)

1.1.4.2 Research Products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) H. G. Read, H. Murakami, K. Hono: Decomposition Precedes Al Partitioning in MA956, an ODS Ferritic Stainless Steel, *Scripta Mater.* **36**, 355 (1997).
- (2) S. P. Ringer, K. Hono, T. Sakurai, I. J. Polmear: Cluster Hardening in an Aged Al-Cu-Mg Alloy: *Scripta Mater.*, **36**, 517 (1997).
- (3) A. Almazouzi, H. Numakura, M. Koiwa, K. Hono, T. Sakurai: Site Occupation Preference of Fe in Ni₃Al: an Atom Probe Study, *Intermetallics*, **5**, 37 (1997).
- (4) H. G. Read, K. Hono, A. P. Tsai, A. Inoue: Preliminary Atom Probe Studies of PdNi(Cu)P Supercooled Liquids, *Mater. Sci. Eng. A*, **A226/228**, 453 (1997).
- (5) K. Hono, Y. Zhang, A. Inoue, T. Sakurai, APFIM Studies on Nanocrystallization of Amorphous Alloys, *Mater. Sci. Eng. A*, **A226/228**, 498 (1997).
- (6) Y. Tanaka, N. Kimura, K. Hono, K. Yasuda, T. Sakurai, Microstructures and Magnetic Properties of Fe-Pt Permanent Magnet, *J. Mag. Mag. Mater.* **170**, 289 (1997).
- (7) K. Hono, T. Sakurai: Recent Atom Probe Studies at IMR - a Comprehensive Review, *Sci. Rep. RITU*, **A44**, 223 (1997).
- (8) S. P. Ringer, C. R. Hutchinson, K. Hono, I. J. Polmear, T. Sakurai: Clustering and Precipitation Processes in Microalloyed Aluminium Alloys, *Sci. Rep. RITU*, **A44**, 241 (1997).
- (9) K. Hono, T. Hashizume, H. Shinohara, T. Sakurai: Field Ion Microscopy of C₆₀ Molecules, *Sci. Rep. RITU*, **A44**, 258 (1997).
- (10) H. G. Read, W. T. Reynolds Jr., K. Hono, T. Tarui: APFIM and TEM Studies of

- Drawn Pearlitic Wire, *Scripta Mater.* **37**, 1221 (1997).
- (11) M. Ohnuma, E. Abe, K. Hono, H. Onodera, M. Mitani, H. Fujimori: Microstructure of Co-Al-O Granular Alloy Thin Films, *J. Appl. Phys.*, **82**, 5646 (1997).
 - (12) M. Murayama, K. Hono: Subnanoscale Microstructure Characterization by Atom Probe FIM: *J. Jpn. Soc. Heat Treatment*, **38**, 20 (1998). (in Japanese)
 - (13) M. Murayama, K. Hono, M. Saga, M. Kikuchi: Pre-precipitation Solute Clustering in Al-Mg-Si Alloys, *Mater. Sci. Eng. A*, **A250**, 127 (1998).
 - (14) K. Hono, Y. Zhang, T. Sakurai, A. Inoue: Microstructure of a Rapidly Solidified Al-4V-2Fe Ultrahigh Strength Aluminum Alloy, *Mater. Sci. Eng. A*, **A250**, 152 (1998).
 - (15) M. H. Hong, K. Hono, M. Watanabe: Microstructure of FePt/Pt Magnetic Thin Films with High Perpendicular Coercivity, *J. Appl. Phys.* **84**, 4403 (1998).
 - (16) K. Hono, M. Murayama: Nanoscale microstructural Analyses by Atom Probe Field Ion Microscopy, *High Temperature Materials and Processes*, **17**, 69 (1998).
 - (17) D. H. Ping, K. Hono, S. Hirosawa: Partitioning of Ga and Co in a Fe₃B/Nd₂Fe₁₄B Nanocomposite Magnet, *J. Appl. Phys.* **83**, 7769 (1998).
 - (18) M. Murayama, K. Hono: Three Dimensional Atom Probe Analysis of Pre-precipitate Clustering in Al-Cu-Mg-Ag Alloys, *Scripta Mater.*, **38**, 1315 (1998).
 - (19) M. H. Hong, W. T. Reynolds, K. Hono: Atom Probe and Transmission Electron Microscopy Investigations of Heavily Drawn Pearlitic Steel Wire, *Met. Mater. Trans. A*, **30A**, 717 (1999).
 - (20) L. Reich, M. Murayama, K. Hono: 3DAP Study of the Effect of Mg and Ag Additions on Precipitation in Al-Cu(-Li) Alloys, *Proc. Inter. Conf. Aluminum Alloys 6 (ICAA-6)*, Toyohashi, Japan, July 5 - 10, 1998, 645.
 - (21) M. Murayama, K. Hono: Atom Probe Characterization of Preprecipitate Clusters and Precipitates in Al-Mg-Si(-Cu) Alloys, *Proc. Inter. Conf. Aluminum Alloys 6 (ICAA-6)*, Toyohashi, Japan, July 5 - 10, 1998, 837.
 - (22) S. Hirosawa, H. Kanekiyo, D. H. Ping, K. Hono: Magnetic Properties and Microstructure of Thick Fe₃B/Nd₂Fe₁₄B Nanocomposite Permanent Magnets Produced by Low-speed Melt Spinning Technique, *Proc. 14th Inter. Workshop on Rare Earth Magnets and Their Applications*, Werkstoff-Informationsgesellschaft mbH, Hamburg, 1998, pp. 215.
 - (23) M. Murayama, Y. Katayama, K. Hono: Microstructural Evolution in PH 17-4 Stainless Steel after Long Term Aging at 400°C, *Met. Mater. Trans. A*, **30A**, 345 (1999).
 - (24) L. Reich, M. Murayama, K. Hono: Evolution of Ω Phase in an Al-Cu-Mg-Ag Alloy - a Three-dimensional Atom Probe Study, *Acta Mater.* **46**, 6053 (1998).

- (25) K. Hono, M. Ohnuma, D. H. Ping, H. Onodera: Cu Clustering and Si Partitioning in the Early Crystallization Stage of an $\text{Fe}_{73.5}\text{Si}_{13.5}\text{B}_9\text{Nb}_3\text{Cu}_1$ Amorphous Alloy, *Acta Mater.* **47**, 997 (1999).
- (26) M. Ohnuma, K. Hono, H. Onodera, J. S. Pedersen, S. Mitani, H. Fujimori: Distribution of Co Particles in Co-Al-O Granular Thin Films, *Mat. Sci. Forum*, **307**, 171 (1999).
- (27) D. H. Ping, K. Hono, A. Inoue, Oxygen Distribution in $\text{Zr}_{65}\text{Cu}_{15}\text{Al}_{10}\text{Pd}_{10}$ Nanocrystalline Alloys, *Mat. Sci. Forum*, **307**, 31 (1999).
- (28) K. Hono, D. H. Ping: APFIM Studies of Nanocomposite Soft and Hard Magnetic Materials *Mat. Sci. Forum*, **307**, 69 (1999).
- (29) M. Ohnuma, K. Hono, H. Onodera, J. S. Pedersen, S. Linderoth: Cu Clustering Stage before the Crystallization in Fe-Si-B-Nb-Cu Alloys, *Nanostructured Materials*, in press.
- (30) M. Ohnuma, K. Hono, H. Onodera, S. Mitani, J. G. Ha, H. Fujimori: Microstructure Change in $\text{Co}_{46}\text{Al}_{19}\text{O}_{35}$ Granular Thin Films by Annealing, *Nanostructured Materials*, in press.
- (31) M. Murayama, K. Hono: Pre-precipitate Clusters and Precipitation Processes in Al-Mg-Si alloys, *Acta Mater.* **47**, 1359 (1999).
- (32) D. H. Ping, K. Hono, H. Kanekiyo, S. Hirosawa: Mechanism of Grain Size Refinement of $\text{Fe}_3\text{B}/\text{Nd}_2\text{Fe}_{14}\text{B}$ Nanocomposite Permanent Magnet by Cu Addition, *J. Appl. Phys.* **85**, 2448 (1999).
- (33) D. H. Ping, K. Hono, H. Kanekiyo, S. Hirosawa: Effect of Cu Addition on the Microstructure and Magnetic Properties of an $\text{Fe}_3\text{B}/\text{Nd}_2\text{Fe}_{14}\text{B}$ Nanocomposite Magnet, *J. Mag. Soc. Japan*, **23**, 1101 (1999).
- (34) D. H. Ping, K. Hono, A. Inoue: Microstructural Characterization of a Rapidly Solidified Ultrahigh Strength $\text{Al}_{94.5}\text{Cr}_3\text{Co}_{1.5}\text{Ce}_1$ Alloy, *Metall. Mater. Trans. A*, in press.
- (35) S. P. Ringer, K. Hono: Microstructural Evolution and Age hardening in Aluminum Alloys: Atom Probe Field Ion Microscopy and Transmission Electron Microscopy Studies, *Mater. Characterization*, in press.
- (36) K. Hono, D. H. Ping: Atom Probe Studies of Nanocrystallization of Amorphous Alloys *Mater. Characterization*, in press.
- (37) M. W. Chen, A. Inoue, T. Sakurai, D. H. Ping, K. Hono: Impurity Oxygen Redistribution in a Nanocrystallized $\text{Zr}_{65}\text{Cu}_{15}\text{Al}_{10}\text{Pd}_{10}$ Metallic Glass, *Appl. Phys. Lett.* **74**, 812 (1999).
- (38) D. H. Ping, K. Hono, A. Inoue: Oxygen Distribution in Zr-based Metallic Glasses

- MRS Proceedings, 554 (1999), in press.
- (39) L. Reich, S. P. Ringer, K. Hono: Origin of the Initial Rapid Age-hardening in an Al-1.7Mg-1.1Cu Alloy, *Phil. Mag. Lett.* (1999), in press.
 - (40) Y. Q. Wu, D. H. Ping, K. Hono, A. Inoue: Atom Probe Characterization of an α -Fe/Nd₂Fe₁₄B Nanocomposite Magnet with a Remaining Amorphous Phase, *IEEE Mag. Mag.* (1999), in press.
 - (41) D. H. Ping, K. Hono, H. Kanekiyo, S. Hirosawa: The Microalloying Effect of Cu and Nb to the Microstructure and Magnetic Properties of Fe₃B/Nd₂Fe₁₄B Nanocomposite Magnet, *IEEE Mag. Mag.*, (1999), in press.
 - (42) K. Hono, M. Murayama, L. Reich: Clustering and Segregation of Mg and Ag Atoms during the Precipitation Processes in Al(-Li)-Cu-Mg-Ag alloys, *Proc. Inter. Conf. Solid -> Solid Phase Transformations '99*, May 24 -28, 1999, Kyoto., in press.
 - (43) K. Hono, D. H. Ping: Atom Probe Studies of Nanocrystallization of Amorphous Alloys, *Proc. Inter. Conf. Solid -> Solid Phase Transformations '99*, May 24 -28, 1999, Kyoto, in press.
 - (44) M. Murayama, K. Hono: The Effect of Aging on the Clustering and Precipitation Process in Al-Mg-Si alloys, *Proc. Inter. Conf. Solid → Solid Phase Transformations '99*, May 24 -28, 1999, Kyoto, in press.
 - (45) K. Hono, D. H. Ping, S. Hirosawa: Effect of Cu on Microstructural Evolution of Nanocrystalline Soft and Hard Magnetic Materials, *MRS Proceedings*, in press.
 - (46) K. Hono, M. Murayama: 3DAP Studies of Solute Clustering and Precipitation in Aluminum alloys, *Materia Japan*, 38 (1999), in press. (in Japanese)
 - (47) K. Hono: Atom Probe Field Ion Microscopy, *Ferum* 4, 474 (1999). (in Japanese)
 - (48) K. Hono: Atom Probe Microanalysis and Nanoscale Microstructures in Metallic Materials (Overview), *Acta Mater.* in press.
 - (49) S. K. Maloney, K. Hono, I. J. Polmear, S. P. Ringer: The Chemistry of Precipitates in an Aged Al-2.1Zn-1.7Mg at.% Alloy *Scripta Mater.* in press.
 - (50) M. Murayama, Z. Horita, K. Hono: Microstructural Evolution in an Al-Cu Alloy Deformed by Equal Channel Angular Pressing, *Mater. Trans. JIM*, in press.
 - (51) M. Murayama, K. Hono, H. Hirukawa, T. Ohnuma, S. Matsuoka: The Combined Effect of Molybdenum and Nitrogen on the Fatigued Microstructure of 316 type Austenitic Stainless Steel, *Scripta Mater.* (1999) in press.
 - (52) D. H. Ping, K. Hono, H. Kanekiyo, S. Hirosawa: Microstructural Evolution of Fe₃B/Nd₂Fe₁₄B Nanocomposite Magnets Microalloyed with Cu and Nb, *Acta Mater.* submitted.
 - (53) M. Ohnuma, K. Hono, H. Onodera, S. Ohnuma, H. Fujimori, J. S. Pedersen:

Microstructures and Magnetic Properties of Co-Al-O Granular Thin Films, J. Appl. Phys. submitted.

Patent Application:

- (1) Alloys for Nanocomposite Rare Earth Magnets and its Processing Method:
K. Hono, D.H. Ping, T. Hirose, H. Kanekiyo, K. Shigemoto, September 18, 1998, H10-264104
- (2) Fatigue Resistant Steel: T. Ohmura, H. Hirukawa, S. Matsuoka, M. Murayama, K. Hono, K. Tsuzaki, February 27, 1999, H-096766

Oral Presentation: 47

- (1) International Conference : 31(1997: 13, 1998: 18),
- (2) Domestic Conference: 16 (1997: 6, 1998: 10)

Awards:

- 1997.1 Tsukuba Research Promotion Award (Ibaraki Prefecture), K. Hono
- 1997.3 Distinguished Achievement Award (Japan Institute for Metals), K. Hono
- 1997.3 Metallography Award (Japan Institute of Metals), M. H. Hong and K. Hono
- 1997.3 Metallography Award (Japan Institute of Metals), M. Murayama and K. Hono

1.1.5 4th Laboratory

Muneyuki Amano (Head), Morimasa Saito, Masatoshi Fukamachi, Nobuhiro Ishikawa, Hiroaki Isago, Yutaka Kagaya, Md. Hasan Zahir (STA fellow), Shinichiro Nakajima (Guest researcher)

1.1.5.1 Research work

In this laboratory 4 kinds of research works have been carried out.

- (1) The pair-correlation-analysis method has been applied for improving the resolution of X-ray images which are obtained by electron probe and have a resolution of less than several μm . The resolution of more than 1 μm was accomplished.
- (2) A numerical simulation method has been applied to improve the accuracy of trace element analyses by using EDS and WDS. The resolution of more than 1 mass% was accomplished.

- (3) Efforts to make metal-phthalocyanine complexes with unusual electronic structures are being made with an intention to apply them for molecular devices. Their electronic structures have been investigated mainly based on spectroscopy, electrochemistry, and spectroelectrochemistry. Some complexes, particularly antimony complexes, have been found to show intriguing spectroscopic and electrochemical properties, indicating that they have quite unusual electronic structures.
- (4) Mirror-finishing surfaces for metals could be obtained by using glow discharge plasma with Kr/Ne mixture.

<Research themes>

- (1) Improvement of Resolution in EPMA Xray Image (General Research: April 1995 to March 1998, Masatoshi Fukamachi)
- (2) Evaluation of Structures with Trace Analysis (General Research: April 1998 to April 2000, Masatoshi Fukamachi)
- (3) Investigation on Synthesis and Characterization of Metal Complexes with High Electron-Spin Multiplicity (General Research: April 1997 to March 2000, Hiroaki Isago)
- (4) Investigation on the Degenerate Electronic Ground State and Electronic Transitions of Metal Complexes with High Symmetry (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 1999, Hiroaki Isago)
- (5) Study on Flat and Mirror-Finishing by Glow Discharge Plasma Using Rare Gases (General Research: April 1995 to March 1998, Morimasa Saito)

1.1.5.2 Research Products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) N. Kobayashi, H. Miwa, H. Isago, T. Tomura: An Adjacent Dibenzo-tetraazaporphyrin: A Structural Intermediate between Tetraazaporphyrin and Phthalocyanine, *Inorg. Chem.*, **38**, 479(1999)
- (2) H. Isago, C. C. Leznoff, M. F. Ryan, R. A. Metcalfe, R. Davids, A. B. P. Lever: Aggregation Effects on Electrochemical and Spectroelectrochemical Properties of [2,3,9,10,16,17,23,24-Octa(3,3-dimethyl-1-butynyl) phthalocyaninato] cobalt (II) Complex, *Bull. Chem. Soc. Jpn.*, **71**, 1039(1998)
- (3) H. Isago, D. Terekhov, C. C. Leznoff: Synthesis and NMR Studies of a Single Isomer of an Alkynyl Substituted Binuclear Phthalocyanine, *J. Porphyrins Phthalocyanines*, **1**, 135(1998)

- (4) Y. Kagaya, H. Isago: Synthesis of Dichloro(phthalocyaninato)antimony(V) Perchlorate, Tetrafluoroborate, and Hexafluorophosphate and Electrochemical Reinvestigation on the New Complex Salts, *Bull. Chem. Soc. Jpn.*, **70**, 2179 (1997)
- (5) Y. Kagaya, H. Isago: Rapid Reactions of Phthalocyanines with Tellurium Tetrachloride in Non-Aqueous Solutions, *J. Porphyrins Phthalocyanines*, in press.
- (6) C. C. Leznoff, Z. Li, H. Isago, D. S. Terekhov: The Syntheses of Octaalkynyl-phthalocyanines, *J. Porphyrins Phthalocyanines*, submitted.
- (7) M. Saito: The Relationship between Relative Sensitivity Factors and Ionization Potential in DC Glow Discharge Mass Spectrometry Using Ar/0.2%vol.%H₂ Mixture, *Anal. Chim. Acta*, **355**, 129(1997)
- (8) M. Saito: Effect of Ar/H₂ and Kr/H₂ Mixtures as Discharge Gas on the Ion Intensity in DC Glow Discharge Mass Spectrometry, *Fresenius' J. Anal. Chem.*, **357**, 18(1997)
- (9) M. Saito: Matrix Effects and the Relationship between the Relative Sensitivity Factors and the Ionization Potential in Glow Discharge Mass Spectrometry Using Kr/0.2%H₂ Mixture, *BUNSEKI KAGAKU*, **48**, 78(1999)

Oral Presentation: 22

- (1) International Conference: 5
- (2) Domestic Conference: 17

Patent Application

- (1) Phthalocyanine Radical Anion: H. Isago, Y. Kagaya, Md. H. Zahir, September 25, 1998, 10-271627, Patent No. 2949230
- (2) A Manufacturing Process for Phthalocyanine Radical Anion: H. Isago, Y. Kagaya, Md. H. Zahir, September 25, 1998, 10-272257, Patent No. 2958461

1.1.6 5th Laboratory

Muneyuki Amano (Head), Katuyuki Takahashi, Yoshisuke Nakamura, Takeshi Kobayashi, Takayuki Yoshioka, Katura Yamada, Masayoshi Kiyokawa, Kazuhiro Honma, Kazuyuki Ogawa, Shinji Ito, Takashi Kimura, Koichi Sato, Kunikazu Ide, Shinichi Hasegawa, Hitoshi Yamaguchi, Ryousuke Hasegawa (guest researcher), Hitoshi Suzuki (technician), Kenji Matsuda (technician), Isao Hamano (special technical staff), Toru Awane (special technical staff)

1.1.6.1 Research work

Systematic study on the improvement of various kinds of analytical technologies has been carried out to promote the R & D of new metallic materials by using advanced facilities for physical and chemical analyses. The aim of this research is to increase the adaptable range of samples and elements, and also to improve the detection limits and precision in instrumental analytical methods. For direct analyses, glow discharge mass spectrometry (GD-MS) and graphite furnace atomic absorption spectrometry (GF-AAS) are used. For separation analyses, inductively coupled plasma-mass spectrometry (ICP-MS), X-ray fluorescence analyzer (XRFA) and inductively coupled plasma-atomic emission spectrometry (ICP-AES) are used. For microarea analyses, electron-probe microanalyzer (EPMA), scanning electron microscope (SEM), transmission electron microscope (TEM) and X-ray diffractometer (XRD) are used. The support work for physical and chemical analyses has been also undertaken in the 5th Laboratory.

<Research themes>

- (1) Development of Techniques for High Precision Analyses of Elements in Metallic Materials (General Research: April 1997 to March 2000, Takeshi Kobayashi)
- (2) Development of High Techniques for Sample Preparation and Analysis by Means of ICP-MS (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 2000, Koichi Sato)

1.1.6.2 Research Products, including papers submitted (April 1997 to March 1999)

- (1) S. Itoh, R.Hasegawa: Measurement of Isotope Ratios of Boron and Silicon by Glow Discharge Mass Spectrometry Using a Small Quantity of Sample, J. Japan Inst. Metals **62**,289(1998) (in Japanese)
- (2) S.Itoh, R.Hasegawa: Studies of Ion Formation in GDMS Ion Sources, J. Japan Inst. Metals **63**,783(1999) (in Japanese)
- (3) S.Itoh, H.Yamaguchi, T.Yoshioka, T.Kimura, T.Kobayashi, Optimization of Measuring Condition for the Determination of Carbon and Nitrogen in Steel by Glow Discharge Mass Spectrometry, Tetsu to Hagane **85**(1999),in press (in Japanese)
- (4) S. Hasegawa, T. Kobayashi, R. Hasegawa: Determination of Trace Aluminum in Iron and Steel Samples by Graphite Furnace Atomic Absorption Spectrometry, J. Japan Inst.Metals **62**,1163(1998) (in Japanese)

- (5) S.Hasegawa, T.Kobayashi, K.Sato, S.Igarashi, K.Naitoh: Determination of Trace Elements in High Purity Iron by Chromazurol B Separation ICP-MS, J. Japan Inst. Metals, submitted (in Japanese)
- (6) T. Kobayashi, S. Hasegawa, T. Yosioka, R. Hasegawa: Determination of Trace Elements in Acidic Sample Solution of Iron and Steels by GF-AAS, CAMP-ISIJ, **11**, 638(1998)
- (7) T.Kobayashi, S.Hasegawa, T.Yoshioka: Determination of Trace Elements in Acidic Solution of Iron and Steels by GF-AAS, Tetsu-to-Hagane **85**,40(1999) (in Japanese)
- (8) H. Yamaguchi, S. Itoh, S. Igarashi, K. Naitoh, R. Hasegawa: Trace Analysis of High Purity Copper by Total Reflection X-ray Fluorescence Spectrometry, Fresenius J. Anal. Chem. **362**,395(1998)
- (9) H.Yamaguchi, S.Itoh, S.Igarashi, K.Naitoh, R.Hasegawa: TXRF Analysis of Solution Samples Using Polyester Film as a Disposable Sample-Carrier Cover, Anal. Sci. **5**,909(1998)
- (10) H.Yamaguchi, S.Itoh, S.Igarashi, K.Naitoh, R.Hasegawa: An Improvement of Microdroplet-X-ray Fluorescence Analysis, Bunseki Kagaku(1999),in press (in Japanese)
- (11) K.Yamada, R.Hasegawa, O.Kujirai: Determination of Seventeen Impurities in High Purity Titanium and Titanium Disilicide by ICP-AES, Mater. Trans. JIM **39**, 663(1998)
- (12) Y.Nakamura, K.Takahashi, O.Kujirai, H.Okochi: Evalution of Electrothermal Vaporization, Emission Intensity-Time-Wavelength Measurement and Time Resolution Combined with an Axially Viewed Horizontal Inductively Coupled Plasma Using an Echelle Spectrometer With Wavelength Modulation, J.Anal. At.Spectrom. **12**,349(1997)
- (13) Y.Nakamura, K.Ide, R.Hasegawa: Use of an Ulutrasonic Nebulizer and Long Torch for Improving the Sensitivity in an Axially-viewed Horizontal ICP-AES, Bunseki Kagaku **48**,339(1999) (in Japanese)
- (14) T.Kimura, H.Doi, K.Hashimoto, E.Abe, Y.Isoda: Phase Equilibria in the TiAl-rich Portion of Ti-Al-Sb System at 1373 and 1573 K, J.Japan Inst. Metals **61**, 385(1997) (in Japanese)
- (15) T.Kimura, Y.Yamamoto, K.Hasimoto, H.Moriya: Residual Strain in Powders of L12 (AlMn)₃Ti Titanium Trialuminide with Additions of Zr, Ga, Ag or V, J.Japan Inst.Metals **63**,174(1999) (in Japanese)
- (16) K.Ogawa, T.Kikuchi, S.Kajiwarra: HREM Observations of Continuously Changing Intermediate Structures between f.c.c. and b.c.c. at the Austenite-Martensite

Interface, J. PHYS.IV FRANCE7,C5-119 (1997)

- (17) K.Ogawa, T.Kikuchi, S.Kajiware, T.Matsunaga, S.Miyazaki: Coherent Subnanometric Plate Precipitates Formed During Crystallization of As-Sputtered Ti-Ni films, J. PHYS.IV FRANCE 7, C5-221(1997)
- (18) K. Sato, K. Ide, M. Kohri, R. Hasegawa : Method of Sample Dissolution by Microwave-Digestion, *The 1997 Fiscal Year Research Accomplishment Report*, June,1998 (in Japanese)
- (19) K. Ide, M. Kohri, K. Sato, Y. Inoue, H. Okochi : Solid Phase Extraction of Mono- and Di-substituted Organotin Compounds in Seawater. Inter. Trace Analy. Symp. '98, 93 (1998)
- (20) K. Ide, M. Kohri, K. Sato, Y. Inoue, H. Okochi : Solid Phase Extraction of Mono- and Di-substituted Organotin Compounds in Seawater, *Bunseki Kagaku* 48, 245 (1999) (in Japanese)

Oral Presentation: 51

- (1) International conference: 3
- (2) Domestic conference: 48

Patent

- (1) Analytical Apparatus of Ultra Low Contents of Oxygen and Hydrogen: H. Okochi, T. Yoshioka, Y. Morimoto, Japan, 25 April, 1990, 2-111264, 16 April,1999, patent No.2914389
- (2) A Formed Filter Paper Medium for Microdroplet Analysis by X-ray Fluorescence Spectrometry: H. Yamaguchi, S. Itoh, S. Igarashi, K. Naitou, R. Hasegawa, Japan, 20 November,1998, 10- 331467
- (3) Ti-Ni ShapeMemory Alloy and its Production Method: S. Kajiware, T. Kikuchi, K. Ogawa, S. Miyazaki, T. Matsunaga, Japan, 8-066820, 19 March,1999, patent No.2899682.

1.1.7 Researchers in the Materials Physics Division

Age and research fields of researchers in the Materials Physics Division. (March 31, 1999)

Age of Researchers	Number	Metal-lurgy	Solid-state Physics	Crystallog-raphy	Physical Analysis	Chemical Analysis
60 ~	6	0	1	0	1	4
50 ~ 60	13	1	1	0	4	7
40 ~ 49	6	0	2	0	0	4
30 ~ 39	12	8	2	1	1	0
22 ~ 29	4	1	3	0	0	0
Summation	41	10	9	1	6	15

1.1.8 Research Budgets in the Materials Physics Division

Research budgets in the Materials Physics Division.(yen)

Kinds of budgets	1997	1998
Special Research	22,848,000	27,170,000
Research on Structural Materials for 21 st Century	3,000,000	3,000,000
General Research	27,084,000	27,972,000
Special Coordination Funds for Promoting Science and Technology	17,250,000	28,015,000
Supplementary Budgets	0	41,470,000
New Energy and Industrial Technology Developing Organization(NEDO)	3,000,000	3,000,000
Summation	73,182,000	130,627,000

1.2 Physical Properties Division

Giyuu Kido (Director)

1.2.1 Research work

Electronic properties such as the electrical conductivity, magnetic susceptibility, superconductivity, etc. are the most important characteristics in materials. Especially, investigation of these properties in new materials is an important basis of materials science. Noble phenomena and valuable properties are expected in the materials of a strongly correlated electron system, low-dimensional system and nano-structured materials. Various compounds containing rare-earth metals have been synthesized at high temperatures using special techniques. At the High Magnetic Field Research Station, world-largest class magnet systems are open to various users. Systems to measure transport properties, optical spectra, magnetization, and NMR have been developed for the hybrid magnet, Bitter magnet, pulsed magnets and superconducting magnets. Low dimensional organic conductors, superconducting materials, heavy fermion systems have been investigated.

Collaborative studies have been carried out between the 1st and 2nd laboratories of the Physical Properties Division as well as with the Materials Physics Division, High Resolution Beam Research Station, Extreme High Vacuum Research Station, other research organizations and universities.

The members of the Physical Properties Division, as at March 31, 1999, consisted of 13 researchers, 3 STA fellows, 5 visiting researchers, 4 joint-doctoral course students, 2 graduate students, 1 secretary, 2 special technical staffs, 8 COE fellows, 3 technicians and 12 guest researchers.

The output of the research activities has increased since the start of the 5th long-term plan. The number of publications, including papers submitted, amount to 102, patents submitted are 6 between April 1, 1997 to March 31, 1999. The details are shown in the research activities of each Laboratory.

1.2.2 1st Laboratory

Giyuu Kido (Head, 1997 FY.), Hideaki Kitazawa (Head, 1998 FY.), Tadashi Takamasu, Mitsutake Oshikiri, Kanji Takehana, Seiichi Kato, Hiroyuki Suzuki, Yasutaka Imanaka

1.2.2.1 Research work

- (1) Various crystals have been synthesized with tetra-arc furnace, tungsten mesh heater and image furnaces. Anomalous magnetization processes were found in ternary compounds RT_mB_n ($m=1,2$, $n=1,2,3$) system, RNi_5 , etc. Single crystals of diluted magnetic semiconductors were grown by using the Bridgeman technique with sealed tungsten crucibles.
- (2) Magneto-optical apparatuses have been constructed for high field magnets in the High Magnetic Field Research Station. Field dependence of the phonon spectra in the spin-Peierls compounds $CuGeO_3$ and the cyclotron resonance of II-VI quantum well have been studied in the far-infrared range. In the visible range, magneto-photoluminescence spectra have been investigated for 2D electrons of hetero-structures and quantum dots.
- (3) A magnetometer has been developed with pulsed high magnetic field to establish the evaluation technique of permanent magnets.
- (4) Coil winding machines were developed to make pulsed field magnets with strong Cu-Ag wire.

<Research themes>

- (1) Spectroscopic Study of Low Dimensional System in High Magnetic Fields
(General Research: April 1997 to March 1998, Giyuu Kido)
- (2) Study on the Unusual Metallic State and Electronic Structure of Magnetic Materials
(General Research: April 1998 to March 2001, Hideaki Kitazawa)
- (3) Development of Measuring Techniques for Quantum Effects in High Magnetic Fields and Research in Strongly Correlated Electron System
(Special Coordination Funds for Promoting Science and Technology: April 1995 to March 2000, Giyuu Kido)
- (4) Magneto-optical Study on Semiconductors in High Magnetic Fields
(Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1998, Tadashi Takamasu)

1. 2. 2. 2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

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 - (15) H. Abe, H. Kitazawa, H. Suzuki, G. Kido, T. Matsumoto: Magnetic Properties of CeRh_2Si_2 and CePd_2Si_2 Single Crystals, *J. Mag. Mag. Mat.*, **177-181**, 479(1998)

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- (17) H. Kito, S. Ikeda, S. Takekawa, H.Abe, H.Kitazawa: Identification of the New Intermetallic Compounds $Y_3Ni_4B_4C_3$ and $Y_1Ni_1B_1C_1$, Related Homologous Series $(LnC)_m(B-Ni_2-B)_n$, *Physica C*, **291**, 332(1997)
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- (44) T.Tsutaoka, Y.Nakamori, T.Tokunaga, M.Kosaka, M.Uwatoko, H.Suzuki, H.Kitazawa, Y.Kido, H.Kadomatsu, T.Itoh: High Field Magnetization of R₇Rh₃ (R=Tb, Dy, Ho, Er), 4th Inter. Symp. on Advanced Physical Field, Tsukuba March 9-12, 167(1999)
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- (59) S.Kato, G.Kido: Measuring Method for Magnetic Properties of Permanent Magnets Using Pulsed Magnetic Fields: to be published in J. Magnetism Society of Japan.
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(note: H. Abe and J. Tang are researchers of Materials Physics Division)

• **Oral Presentation** :69

- (1) International Conference: 24
- (2) Domestic Conference: 45

• **Patent Application** :

- (1) Wire Taping Machine: G.Kido, K.Sakamoto, No-09-304283, September 11, 1997.
- (2) Growth Method of Bulk Single Crystal: G.Kido, H.Suzuki, No.-10-329406, September 19, 1998.
- (3) Drum Controlling Coil Winding Machine: G.Kido, K.Sakamoto, No.-11-060830, March 8, 1999.

- (4) Linear Controlling Coil Winding Machine: G.Kido, K.Sakamoto, No.-11-060831, March 8, 1999
- (5) Sulfide Spinel Super Conducting Material: H.Suzuki, H.Kitazawa, T.Matamoto, No.-11-102202, April 9, 1999.
- (6) Pulsed Field Magnetometer: S.Kato, G.Kido, No.-11-104780, April 13, 1999.

1.2.3 2nd Laboratory

Haruyoshi Aoki (Head, 1997 FY.), Giyu Kido (Head, 1998 FY.), Tadashi Shimizu, Sinya Uji, Taichi Terashima, Atuo.

1.2.3.1 Research work

- (1) Low dimensional electron systems and highly correlated electrons have been investigated under high magnetic fields up to 20 T and low temperatures down to 25 mK. We focused on the high field state of organic conductors and the metamagnetic transition in highly correlated electron systems.
- (2) Development of the nuclear double resonance spectrometer was carried out, with a wide frequency range, from 10 to 200 MHz. It was found that the dimension of the Cu spin system plays an important role in the formation of spin gap state Cu oxide superconductors.

<Research themes>

- (1) Study on Strongly Correlated Electron System under Multi-extreme Conditions.
(R&D on New Superconducting Materials: April 1997 to March 1998, Haruyoshi Aoki, April 1998 to March 1999, Giyu Kido)
- (2) Development of Measuring Techniques for Quantum Effects in High Magnetic Fields and Research in Strongly Correlated Electron System
(Special Coordination Funds for Promoting Science and Technology: April 1995 to March 1998, Haruyoshi Aoki, April 1998 to March 2000, Giyu Kido)

1.2.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

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- (4) M.Takashita, H.Aoki, T.Matsumoto, C.J.Haworth, T.Terashima, A.Uesawa, T.Suzuki: Highly Anisotropic Pressure Dependence of the Fermi Surface of CeSb, *Phys. Rev.Let.*, **78**, 1948(1997)
- (5) H.Ikezawa, H.Aoki, M.Takashita, C.J.Haworth, S.Uji, T. Terashima, K.Maezawa, R.Settai, Y.Onuki: Fermi Surface Properties of Ferromagnetic CeRu_2Ge_2 , *Physica B*, **237-238**, 210(1997)
- (6) G.Bottger, P.Fischer, A.Donni, P.Berastegui, Y.Aoki, H.Sato, F.Fauth: Long-range Magnetic Order of the Er ions in $\text{Er}_2\text{Ba}_4\text{Cu}_7\text{O}_{14.92}$, *Phys. Rev. B*, **55**, R12005(1997)
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- (11) T.Terashima, C.Haworth, M.Takashita, H.Aoki, N.Sato, T.Komatsubara: Heavy Fermions Survive the Metamagnetic Transition in UPd_2Al_3 , *Phys.Rev.B.*, **55**, R13369(1997)
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- (18) H.Aoki, M.Takashita, C.J.Haworth, T.Matsumoto, T.Terashima, S.Uji, C.Terakura, T.Miura, A.Uesawa, T.Suzuki: dHvA Effect of CeSb under Pressure in the Antiferromagnetic Phase, *J. Mag. Mag. Mater.*, **177-181**, 371(1998)
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- (34) T.Shimizu, T.Matsumoto, A.Goto, K.Yoshimura, K.Kosuge: Spin Susceptibility of the One Dimensional Antiferromagnet CuO , 4th Inter. Symp. on Advanced Physical Field, Tsukuba March 9-12, 285(1999)
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- (37) A.Goto, T.Shimizu, M.Kato, K.Yoshimura, K.Kosuge: Antiferromagnetic Ordering in $\text{PrBa}_2\text{Cu}_3\text{O}_6$ Studied by NMR, 4th Inter. Symp. on Advanced Physical Field, Tsukuba March 9-12, 271(1999)
- (38) T.Terashima, C.Terakura, S.Uji, H.Aoki, J.S.Qualls, D.Hall, J.S.Brooks, T.Fukase:

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- (39) T.Miura, H.Aoki, M.Takashita, C.Terakura, S.Uji, G.Kido: dHvA Effect Study of CeBi in the Ferrimagnetic Phase, 4th Inter. Symp. on Advanced Physical Field, Tsukuba March 9-12, 165(1999)
- (40) T.Ebihara, K.Koizumi, S.Uji, C.Terakura, T.Terashima, H.Suzuki, H.Kitazawa, G.Kido: de Haas-van Alphen Effect in a Field Induced Ferromagnetic State of $CePb_3$, 4th Inter. Symp. on Advanced Physical Field, Tsukuba March 9-12, 161(1999)
- (41) N.Nakayama, N.Kimura, H.Aoki, S.Uji, T.Komatushara: Fermi Surfaces of $La_3Pd_{20}Ge_6$, $Ce_3Pd_{20}Ge_6$, 4th Inter. Symp. on Advanced Physical Field, Tsukuba March 9-12, 157(1999)
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- (43) G.Kido, T.Takamasu, K.Takehana, T.Imanaka, M.Oshikiri: Magneto-optical Study on Low Dimensional Materials in High Magnetic Fields, 4th Inter. Symp. on Advanced Physical Field, Tsukuba March 9-12, 55(1999)
- (44) C.Terakura, T.Miura, T.Terashima, H.Suzuki, S.Uji, H.Aoki, A.Ramakrishnan, B.Becker, A.A.Menovsky, G.J.Nieuwenhuys, J.Mydosh: Metamagnetic Transition and dHvA Effect of $U_2Rh_3Si_5$, *Physica B*, **259-261**, 1087(1999)
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- (46) A. Goto, T. Shimizu: Magnetic Scaling in the Underdoped Superconductor $Hg_{0.8}Re_{0.2}Ba_{0.2}Ca_2Cu_3O_8$ Studied by ^{63}Cu NMR, to be published in *Phys. Rev. B*.
- (47) A. Goto, T. Shimizu: NMR Studies of Low Energy Magnetic Excitations in High-Tc Cuprates, to be published in *Butsuri*. (in Japanese)

Oral Presentation: 61

- (1) International Conference: 26
- (2) Domestic Conference: 35

Award :

- (1) JPSJ Research Article Prize: G. Kido, March 30, 1999

- (2) The Young Researcher Award: The Japan institute of Metals,
T.Terashima, September 24, 1997

1.2.4 Researchers in the Physical Properties Division

Age and research fields of researchers in the Physical Properties Division.

(March 31, 1999)

Age of Researchers	Number of researchers	Solid-state Physics
60 ~	0	0
50 ~ 60	1	1
40 ~ 49	2	2
30 ~ 39	10	10
22 ~ 29	0	0
Summation	13	13

1.2.8 Research Budgets in the Physical Properties Division

Research budgets in the Physical Properties Division. (yen)

Kinds of budgets	1997	1998
General Research	8,316,000	9,500,000
R&D on new Superconducting Materials	22,913,000	25,338,000
Special Coordination Funds for Promoting Science and Technology	78,625,000	68,951,000
Supplementary Budget	12,000,000	13,000,000
Summation	121,854,000	116,789,000

1.3 Computational Materials Science Division

Takehiko Matsumoto (Director)

1.3.1 Research work

In the field of computational science, it is remarkable that computer performance is greatly enhanced and the algorithm for analyses is progressing significantly. With the technological and scientific advances, we aim to predict materials properties, and develop new materials. In this case, it is essential that the phenomena in materials are theoretically understood in terms of the motion and structures of electrons and atoms. Therefore, we require advance in the methodological and analytical techniques that are fitted to the hierarchy of materials, for example, from a microscopic property, due to electrons, to a macroscopic strength in structural materials.

For these targets, we currently investigate the following subjects. (1) First-principles simulations for structures and reaction processes on materials surfaces. (2) Research of magnetic properties, materials design and structural predictions by large scale simulations based on theoretical analyses. (3) Evaluation of materials strength by molecular-dynamics simulations and finite element method. (4) Construction of databases for promoting materials development. Consequently, we have achieved outstanding results.

The members of the Computational Materials Science Division, as at March 31, 1999, consisted of 21 researchers, 2 postdoctoral (domestic) research fellows, 2 graduate student and 2 secretaries. The productivity of the research activities has been increased since the start of the 5th Long-term Plan. During April 1, 1977 to March 31, 1999, the number of publications, including submitted papers, is about 86, and 9 plenary and invited talks were given at international conferences. The details are shown in the research activities of each Laboratory.

1.3.2 1st Laboratory

Masatoshi Nihei (Head), Yuji Asada, Masao Sakamoto, Etsuo Nakata

1.3.2.1 Research work

- (1) The mesoscopic simulation method, using the continuum mechanics, was developed to analyze the microscopic deformation of polycrystalline materials. This research work was carried out in collaboration with the Strength and Life Evaluation Research Station.

- (2) The prototype of a new unification system, DIMNET, has been developed to utilize a networking environment and to treat many materials scientific databases of various different types which are located on different sites bound with a network system such as by INTERNET.
- (3) A numerical database, SUPERCON, for superconducting materials has been constructed and is updated every month. A Tc-prediction system using the neural network method has been developed using a data set from the SUPERCON as the training data.

<Research themes>

- (1) Modeling and Simulation for the Prediction of Material Strength
(Special Coordination Funds for Promoting Science and Technology-Virtual Experiments for Material Design Project-: April 1995 to March 1998, Masatoshi Nihei)
- (2) Development of Unification System for Distributed Materials Scientific Data utilizing Networking Environment
(Computational Materials Research: April 1995 to March 2000, Masatoshi Nihei)
- (3) Development of Knowledge Database for High-Tc Superconducting Materials
(Multi-core project: April 1995 to March 2000, Yuji Asada)

1.3.2.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) J.Onodera, J.Onoue, M.Nihei: A Fatigue Design Procedure for Screw Threads Using Predicted S-N Curves, Trans. JSME, 63-605A, 207(1997)
- (2) M.Sakamoto, M.Nihei: Local Stress-Strain Behavior of Polycrystalline Materials, J. of SMSJ (Zairyou), 48-1, 44(1999)
- (3) M.Nihei: Development of International Standards – Activities of ISO/TC 164 (Mechanical Testing of Metals) -, J. of JSME, 102-966, 305(1999)
- (4) M.Sakamoto, M.Nihei: Construction of Material Strength Database for Springs, Trans. of JSSR, 44, 31(1999)
- (5) Y.Asada, E.Nakada, S.Matsumoto, H.Uesaka: Prediction of Tc for Yba₂Cu₃O_z Doped with Ca Using Neural Network, J.Superconductivity 10, 23(1997)

Oral Presentation: 6

(1) International Conference: 0

(2) Domestic Conference: 6

1.3.3 2nd Laboratory

Takahisa Ohno (Head), Taizo Sasaki, Jun Nara, Tsuyoshi Miyazaki, Yoshitaka Takeyama, Shin'ichi Higai (domestic research fellow), Takahide Ezaki (joint-doctoral course student)

1.3.3.1 Research work

The thermal reaction processes on various solid surface systems, such as semiconductor surfaces and metal surfaces, have been theoretically investigated by using first-principles total-energy calculation techniques within density functional theory. The structural stability and optimum atomic arrangement of semiconductor surfaces such as GaAs have been theoretically determined. First-principles simulations for the adsorption of atoms and molecules on semiconductor and metal surfaces, for example, Si on H-terminated Si(001), Cl₂ on GaAs(001), O₂ on Al(111), and Ti on Si(001), have been performed, which clarify the microscopic mechanism of epitaxial growth, etching, oxidation, and interface formation. Atomic and electronic structures of misfit dislocations in semiconductor hetero-interfaces such as InAs/GaAs(110) have been theoretically studied. Methodological and numerical techniques have been developed for first-principles total-energy calculation.

<Research themes>

- (1) Study on the First-principles Simulation for Surface Reactions (Computational Materials Research: April 1994 to March 2000, Takahisa Ohno)
- (2) Virtual Experiments for Material Design Project I "Theoretical Study on the atomic motions on surfaces" (Special Coordination Funds for Promoting Science and Technology: April 1995 to March 1998, Taizo Sasaki)
- (3) Virtual Experiments for Material Design Project II "Theoretical Study on the structural control for metal-semiconductor" (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 2000, Taizo Sasaki)
- (4) First-Principles Study on Dislocations in Solid (Joint Research Promotion System on Computational Science and Technology: April 1998 to March 2001, Takahisa

Ohno)

1.3.3.2 Research products, including paper submitted (April 1, 1997 to March 31, 1999)

- (1) J. Nara, T. Sasaki, T. Ohno: Theoretical Investigation on Delta Doping of Se Atoms in GaAs, *Appl. Phys. Lett.* **70**, 3534(1997)
- (2) J. Nara, T. Sasaki, T. Ohno: Adsorption and Diffusion of Si Atoms on the H-Terminated Si(001) Surface: Si Migration Assisted by H Mobility, *Phys. Rev. Lett.* **79**, 4421(1997)
- (3) A. Taguchi, T. Ohno: Erbium in GaAs: Coupling with Native Defects, *Phys. Rev. B.* **56**, 9477(1997)
- (4) S. Tsukamoto, T. Ohno, N. Koguchi: Scanning Tunneling Microscopy and First-Principles Investigation on GaAs(001)(2x6)-S Surface Formed by Molecular Beam Epitaxy, *J. Crystal Growth* **175**, 1303(1997)
- (5) T. Miyazaki, K. Terakura: (DI-DCNQI)2Cu as a Unique Member of DCNQI-Cu Family: A Theoretical Study of High-Pressure Phases, *Phys. Rev. B* **56**, R477 (1997)
- (6) T. Ohno, T. Sasaki, A. Taguchi: First-Principles Calculations of Diffusion of Chlorine Atoms in GaAs, *Mater. Res. Society Symp. Proc.* **442**, *Defects in Electronic Materials II*, p.529 (1997)
- (7) T. Ohno: Theory of Surface Electronic States, *Electronic States of Surfaces and Interfaces*, Surface Science Series Vol. 4, ed. by A. Koma (Maruzen, Tokyo 1997)
- (8) J. Nara, T. Sasaki, T. Ohno: First-Principles Calculations on Diffusion of Si Adatoms on H/Si(001)-(2x1) Surfaces, *Appl. Surf. Sci.* **130-132**, 254 (1998)
- (9) T. Sasaki, Theoretical Study on the Pressure-Induced Instability of the B1 structure of Ionic Materials, *The Review of High Pressure Science and Technology*, vol.7, 169(1998)
- (10) B.D. Yu, Y. Miyamoto, O. Sugino, T. Sasaki, T. Ohno: Favorable Formation of the C49-TiSi₂ Phase on Si(001) Determined by First-Principles Calculations, *Appl. Phys. Lett.* **72**, 1176(1998)
- (11) B.D. Yu, Y. Miyamoto, O. Sugino, T. Sasaki, T. Ohno: Unusual Ti-Adsorption and Activation of Si-Ejection on Si(001), *Phys. Rev. B.* **58**, 3549(1998)
- (12) Z. Fang, K. Terakura, H. Sawada, T. Miyazaki, I. Solovyev: Inverse versus Normal NiAs Structures as High Pressure Phases of FeO and MnO, *Phys. Rev. Lett.* **81**, 1027(1998)
- (13) T. Ogitsu, T. M. Briere, K. Kusakabe, S. Tsuneyuki, Y. Tateyama: First-

- Principles Study of the Ortho-KC60 Polymer, Phys. Rev. B. **58**, 13925(1998)
- (14) S. Tsuneyuki, Y. Tateyama: Theoretical Design of BCN Heterodiamond, The Review of High Pressure Science and Technology **9**, 268(1998)
 - (15) T. Ohno, T. Sasaki, A. Taguchi: Ab Initio Investigations on Diffusion of Halogen Atoms in GaAs, Materials Science Forum **258-263**, 1821(1998)
 - (16) T. Ohno: Breaking of Backbonds of GaAs(001) Surfaces by Adsorption of Chlorine Molecules, Proc. 24th Inter. Conf. on Physics of Semiconductors (World Scientific Publishing, 1998)
 - (17) J. Nara, T. Ohno: Exotic Effects of Hydrogen Termination During Silicon Epitaxial Growth, Proc. 24th Inter. Conf. on Physics of Semiconductors (World Scientific Publishing, 1998)
 - (18) T. Ohno, J. Nara, T. Sasaki: Diffusion of Si Adatoms on H-Terminated Si(001) Surfaces, Mater. Res. Society Symp.Proc. **492**, *Microscopic Simulation of Interfacial Phenomina in Solids and Liquids*, p.275 (1998)
 - (19) T. Ohno: Behavior of Fluorine Atoms in Compound Semiconductors, Oyobuturi Vol.67, No.2, 155 (1998)
 - (20) J. Nara, T. Sasaki, T. Ohno: Theory of Adsorption and Diffusion of Si Adatoms on H/Si(100) Stepped Surfaces, J. Crystal Growth **201/202**, 77(1999)
 - (21) T. Ezaki, T. Ohno: Theoretical Investigations of Adsorption of Fluorine Atoms on the Si(001) Surface, to be submitted to Surf. Sci. (1999)
 - (22) T. Sasaki, T. Ohno: Adsorption of the Oxygen to the Al(111) Surface, Computational Mater. Sci. **14**, 8(1999)
 - (23) T. Sasaki, T. Ohno: Dissociation Process of O₂ on the Al(111) Surface, Surf. Sci. (in press)
 - (24) T. Miyazaki, T. Ohno: First-Principles Study of the Electronic Structure of the Organic Solids (CH₃)₄N[M(dmit)₂]₂ (M=Ni and Pd): Role of Dimerization and the Stability of the Formation of a Dimer, Phys. Rev. B. **59**, R5269(1999)
 - (25) N. Katoh, T. Miyazaki, T. Ohno: First-Principles Study on the Insulating State of Alpha'-NaV₂O₅, Phys. Rev. B. **59**, R12723(1999)
 - (26) Q.K. Xue, Q.Z. Xue, R.Z. Bakhtizin, Y. Hasegawa, I.S.T. Tsong, T. Sakurai, T. Ohno: Structures of GaN(0001) 2x2, 4x4 and 5x5 Surface Reconstructions, Phys. Rev. Lett. **82**, 3074(1999)
 - (27) Y. Tateyama, S. Tsuneyuki: Material Design of BCN Compounds via First-Principles Calculations, NEW DIAMOND, No. 53, 6(1999)
 - (28) B. D. Yu, Y. Miyamoto, O. Sugino, A. Sakai, T. Sasaki, T. Ohno: Dimer Reconstruction at Metal-Silicide/Silicon Interfaces: A First-Principles Study,

- Mater. Res. Society Symp. Proc., *Advanced Interconnects and Contacts*, (1999) (in press)
- (29) T. Ohno: Surfaces as Quantum Mechanical Materials, *Handbook of Quantum Mechanics* (Asakura, Tokyo 1999)

Oral Presentation: 58

- (1) International Conference: 24
- (2) Domestic Conference: 34

1.3.4. 3rd Laboratory

Hidehiro Onodera (Head), Masato Shimono, Masato Ohnuma, Taichi Abe,
Masahiko Katagiri, Tu Geng (domestic research fellow)

1.3.4.1. Research work

Atomic configurations, such as long range ordering and short range ordering, not only play a significant role on relative phase equilibrium but also affect the physical properties of alloys. For establishing materials design technologies, based on predictions of atomic configuration, the cluster variation method and the central atoms model have been applied to thermodynamic analyses on atomic configurations in intermetallic compounds and solid solution alloys, respectively. For nanocrystalline and amorphous alloys, microstructural analyses have been performed by small-angle X-ray experiments and the molecular dynamics simulation to make clear the formation process of these microstructures on the atomic level. The molecular dynamics technique has also been applied to make clear the microscopic mechanism of the martensitic transformation from a metastable fcc phase into a stable bcc phase in Fe-Ni alloys and the hydrogen-induced amorphization in C15 Laves phases.

<Research themes>

- (1) Thermodynamic Analysis of Formation Processes of Metastable and Stable Phases
(General Research: April 1997 to March 2000, Hidehiro Onodera)
- (2) Improvement of the Long Term Creep Strength of Ferritic Steels by the Minimum Alloying (Special Coordination Funds for Promoting Science and Technology: April 1996 to March 1998, Hidehiro Onodera)
- (3) Virtual Experiments for Material Design Project (Special Coordination Funds for

Promoting Science and Technology: April 1995 to March 2000, Hidehiro Onodera)

- (4) Molecular Dynamics Study on Superplastic Deformation and Embrittlement due to Grain Boundary Segregation (Joint Research Promotion System on Computational Science and Technology: April 1997 to March 2000, Hidehiro Onodera)

1.3.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) M. Ohnuma, J. Suzuki, S. Funahashi, H. Onodera, Y. Hamaguchi: Small-Angle neutron scattering study on amorphous Fe-Cu-Nb-Si-B alloys, Mater. Sci. & Eng. A, Rapidly Quenched & Metastable, Materials supplement, 183(1997)
- (2) M. Ohnuma, K. Hono, E. Abe, S. Mitani, H. Onodera, H. Fujimori: Microstructure of Co-Al-O granular alloy thin films, J. Appl. Phys, 82, 5646(1997)
- (3) H. Onodera, T. Abe, M. Shimono, M. Ohnuma: Design of Alloys Based on the Prediction of Atomic Configuration, Proc. Int. Symp. on Processing, Designing and Properties of Advanced Materials (ISAEM-'97), Toyohashi, Japan, 131(1997)
- (4) T. Abe, H. Onodera, K. Kimura, K. Halada, K. Ijima: Improvement of the ecobalance of ferritic steels by the minimum alloying, Proc. 3rd Inter. Conf. on ECOMATERIALS, 47(1997)
- (5) H. Onodera, T. Abe: Alloy Design Based on the Prediction of Atomic Configuration, Australasia-Pacific Forum on Intelligent Processing and Manufacturing of Materials, Australia, 1171(1997)
- (6) M. Ohnuma, S. Linderoth, N. Pryds, M. Eldrup, A.S. Pedersen: On the Effect of Al on the Formation of Amorphous Mg-Al-Cu-Y Alloys, MRS proceedings series, 554(1998)
- (7) M. Shimono, H. Onodera: Molecular Dynamics Study on Liquid-to-Amorphous Transitions in Ti-Al Alloys, Mater. Trans. JIM, 39, 147(1998)
- (8) T. Suzuki, M. Shimono: Simulation of Martensitic Transformation in Fe-Ni Alloys, Proc. 3rd Pacific Rim Inter. Conf. on Advanced Materials and Processing (PRICM 3), Hawaii, USA, 1193(1998)
- (9) H. Onodera, M. Yamazaki: Computational Materials Science, *Materials Science and Engineering*, edited by H. Hojo, published by Shokabo, 199(1998)
- (10) H. Onodera: Design of Titanium Alloys and Intermetallic Compounds, Metals and Technology, 68, 444(1998)
- (11) T. Abe, H. Onodera: Effect of Solute Elements on the Creep Properties of Ferritic Steels, Proc. 3rd Inter. Workshop on Super Steels, 136(1998)
- (12) K. Hono, D.H. Ping, M. Ohnuma, H. Onodera: Cu Clustering and Si Partitioning

- in the Early Crystallization Stage of an FeCuNbSiB Amorphous Alloy, *Acta Mater.*, **47**, 997(1999)
- (13) M. Ohnuma, K. Hono, H. Onodera, J.S. Pedersen, S. Mitani, H. Fujimori: Distribution of Co Particles in Co-Al-O Granular Thin Films, *Mater. Sci. Forum*, **307**, 171(1999)
- (14) T.Suzuki, M.Shimono, S.Takeno: Vortex on the Surface of a Very Small Crystal during Martensitic Transformation, *Phys. Rev. Lett.*, **82**, 1474(1999)
- (15) Taichi Abe, Hidehiro Onodera, Kazuhiro Kimura, Hideaki Kushima: Effect of M-C(M=Mo, Mn and Cr) Atomic Pairs on Creep Properties of Fe-M-C Ternary Alloys, *J.Japan Inst. Metals*, **63**, 717(1999)
- (16) M. Ohnuma, K. Hono, H. Onodera, S. Mitani, J.G. Ha, H. Fujimori: Microstructure Change in $\text{Co}_{46}\text{Al}_{19}\text{O}_{53}$ Granular Thin Films by Annealing, *Nano Stuc. Mater.*, in press
- (17) M. Ohnuma, K. Hono, H. Onodera, J.S. Pedersen, S. Linderorth: Cu Clustering Stage before the Crystallization in an Fe-Si-B-Nb-Cu Amorphous Alloy, *Nano.Struc. Mater.* in press
- (18) M. Ohnuma, K. Hono, H. Onodera, S. Ohnuma, H. Fujimori, J.S. Pedersen: Microstructure and Magnetic Properties of Co-Al-O Granular Thin Films, *J. Appl. Phys.*, submitted
- (19) M.Ohnuma, N. H. Pryds, S. Linderorth, M. Eldrup, A. S. Pedersen, J.S. Pedersen: Bulk Amorphous $(\text{Mg}_{0.98}\text{Al}_{0.02})_{60}\text{Cu}_{30}\text{Y}_{10}$ Alloy, *Scripta Mater.* in press
- (20) M.Shimono, H.Onodera: Order-disorder Transition in HCP Binary Alloys -Next-to-nearest-neighbor Interactions-, *Phys. Rev. B*, submitted
- (21) M.Shimono, H.Onodera, T.Suzuki: FCC-BCC Phase Transition in Iron under a Periodic Boundary Condition, *Mater. Trans. JIM*, in press
- (22) M.Shimono, H.Onodera, T.Suzuki: Atomistic Simulation of the Martensitic Transformation with a Periodic Boundary Condition, *Proc. Inter. Conf. on Solid-Solid Phase Transformations (PTM '99)*, Kyoto, Japan, (1999), in press
- (23) M. Katagiri, H. Onodera : A Molecular Dynamics Study of Hydrogen Induced Amorphization -Softening Effect by Incorporation of Hydrogen-, *Mater. Trans., JIM*, in press
- (24) M. Katagiri, H. Onodera: A Molecular Dynamics Study of Hydrogen Induced Amorphization – Role of Atomic Relaxation -, *Journal of Computer-Aided Materials Design*, in press
- (25) M. Katagiri, H. Onodera : A Molecular-Dynamics Simulation of Hydrogen-Induced Amorphization, *Trans. MRS-J*, in press

- (26) M. Katagiri, D. L. Patrick, R. M. Lynden-Bell: Molecular Dynamics Simulation of Atomic Force Microscopy - imaging Single-atom Vacancies on Ag(001) and Pt(001) -, Surface Science, in press
- (27) T.Suzuki, M.Shimono: Evolution of Lattice Vibration through Vortex into Change of Crystal Structure - a Path in Martensitic Transformation -, Proc. Inter. Symp. and Exhi. on Shape Memory Materials '99 (SMM '99), Kanazawa, Japan, (1999), in press
- (28) T.Suzuki, M.Shimono: Role of Lattice Defects in Martensitic Transformation, Proc. Int. Conf. on Solid-Solid Phase Transformations (PTM'99), Kyoto, Japan, (1999), in press
- (29) Taichi Abe, Hidehiro Onodera: Effect of M-C(M=Mo, Mn, and Cr) Atomic Pairs on Creep Properties of Ferrite Alloys in Fe-M-C Ternary System, CFEMS-8, in press
- (30) H.Onodera: Design of Titanium Alloys, *Intermetallic Compounds and Heat Resistant Ferritic Steels, Computational Materials Design*, Springer-Verlag, in press

Oral presentations: 63

- (1) International Conference: 22,
- (2) Domestic Conference: 41

Awards

- (1) Young Researcher Award: IUMRS-ICA-97, 1997, Masato Ohnuma
- (2) Commendation by the Minister of State for Science and Technology: 1998, Hidehiro Onodera

1.3.5 4th Laboratory

Xiao Hu (Head), Yoshihiko Nonomura

1.3.5.1 Research work

- (1) The phase transitions and phase diagrams of high-T_c superconducting materials in an external magnetic field have been clarified by means of large scale computer simulations.
- (2) The mechanism of high-T_c superconductivity has been studied by focusing on the

- competition and unification of anti-ferromagnetism and d-wave superconductivity.
- (3) Micromagnetic theory has been formulated for nano-scale magnetic phenomena, such as spin reorientation transitions in ultrathin magnetic films and multilayers.

<Research themes>

- (1) Theoretical and Computational Research on the Nano-scale Magnetic Materials and Phenomena (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1998, Xiao Hu)
- (2) Theoretical Study on New Phenomena in Ultrathin Magnetic Films and Multilayer Systems (General Research: April 1997 to March 2000, Xiao Hu)
- (3) Computer Simulation on Vortex States and Phase Transition in High Tc Superconductors (Computational Materials Research: April 1999 to March 2004, Xiao Hu)

1.3.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) X. Hu, S. Miyashita, M. Tachiki: δ -function Peak in the Specific Heat of High-Tc Superconductors: Monte Carlo Simulation, Phys. Rev. Lett. **79**, 3498(1997)
- (2) X. Hu, S. Miyashita, M. Tachiki: Simulation for the First-order Vortex-lattice Melting Transition in High-Tc Superconductors, Physica C, **282-287**, 2057(1997)
- (3) X. Hu: Magnetization Reversal and Coercive Force in Ultrathin Films with Perpendicular Surface Anisotropy: Micromagnetic Theory, Phys. Rev. B **53**, 8382(1997)
- (4) M. Tachiki, X. Hu: Mystery of Vortex in Superconductors, Mathematical Science, **412**, 34 (1997)
- (5) X. Hu: Micromagnetic Theory of Ultrathin Magnetic Films, J. Mag. Soc. Japan, **21**, 1281(1997)
- (6) X. Hu, M. Tachiki: Structure and Phase Transition of Josephson Vortices in Anisotropic High-Tc Superconductors, Phys. Rev. Lett. **80**, 4044(1998)
- (7) X. Hu, S. Miyashita, M. Tachiki: Monte Carlo Simulation on the Phase Transition in the Flux States of High-Tc Superconductors: B || C Axis, Phys. Rev. B **58**, 3438(1998)
- (8) X. Hu, Y. Kawazoe: Micromagnetic Study on Ultrathin Magnetic Films, Comput. Mater. Sci. **10**, 198(1998)
- (9) M. Tachiki, S. E. Shafranjuk, X. Hu: Vortex Lattice Structure and Josephson

- Plasma in High-Tc Superconductors, Chinese J. Phys. **36**, 171(1998) = invited
- (10) T. Yorozu, X. Hu: Magnetization of a Trilayer Structure with Alternate In-Plane and Out-of-Plane Anisotropies, J. Appl. Phys. **84**, 3278(1998)
 - (11) Y. Nonomura: New Quantum Monte Carlo Study of Quantum Critical Phenomena with Trotter-Number-Dependent Finite-Size Scaling and Non-equilibrium Relaxation, J. Phys. A: Math. and Gen. **31**, 7939(1998)
 - (12) X. Hu: Spin Reorientation in Ultrathin Magnetic Films with Normal Surface Anisotropy, Condensed Matter News, **7**, 18(1998)
 - (13) X. Hu, T. Koyama, M. Tachiki: Phase Diagram of a Superconducting and Antiferromagnetic System with SO(5) Symmetry, Phys. Rev. Lett. **82**, 2568(1999)
 - (14) X. Hu, M. Tachiki: Monte Carlo Simulation on the First-order Flux Lattice Melting in High-Tc Superconductors, *Advances in Superconductivity XI*, ed. by N. Koshizuka and S. Tajima (Springer-Verlag, Tokyo 1999), 579
 - (15) T. Yorozu, X. Hu: Magnetization Hystereses in Trilayer Systems with an In-plane Component Defect, J. Appl. Phys. in press
 - (16) Y. Nonomura, X. Hu, M. Tachiki: Flux-line Entanglement as the Mechanism of Melting Transition in High-Temperature Superconductors in a Magnetic Field, Phys. Rev. B **59**, R11657(1999)
 - (17) N. Ito, Y. Ozeki, Y. Nonomura: Non-equilibrium Relaxation Study on Equilibrium State, Butsuri **54**, 336(1999) (in Japanese)

Oral Presentation: 39

- (1) International Conference: 21
- (2) Domestic Conference: 18

1.3.6 6th Laboratory

Katsuyuki Kusunoki (Head), Norio Akaiwa

1.3.6.1 Research work

- (1) The dynamical processes of deformation and phase transition of crystalline materials have been studied using molecular-dynamics simulations.
- (2) Microstructural evolution in elastically stressed solids, spinodal decomposition, and particle coarsening under microgravity have been studied using the sharp and diffuse interface models.

<Research themes>

- (1) A Computer-Simulation Study of the Plastic Deformation of Crystals (General Research: April 1997 to March 2000, Katsuyuki Kusunoki)
- (2) Numerical Simulation of Phase Separation (General Research: April 1998 to March 2000, Norio Akaiwa)

1.3.6.2 Research Products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) K. Kusunoki: Melting-temperature Depression at Crystal Interfaces with Large Lattice Mismatch, submitted to Phys. Rev. Lett.
- (2) N. Akaiwa, P. W. Voorhees: Numerical Simulation of Microstructural Evolution in Elastically Stressed Solids Using Interfacial Model, Proc. Conf. on Computational Engineering and Science, 3,989(1998)
- (3) N. Akaiwa, P. W. Voorhees: Large Scale Numerical Simulation of Particle Coarsening in Elastically Stressed Solids, Materials for Advanced Power Engineering, 5, 1421(1998)
- (3) N. Akaiwa, P. W. Voorhees: Large Scale Numerical Simulation of Microstructural Evolution in Elastically Stressed Solids, Mater. Sci. and Engi. A, in press.
- (4) J. Alkemper, V. Snyder, N. Akaiwa, P. W. Voorhees: Dynamics of Late-Stage Phase Separation: A Test of Theory, Phys. Rev. Lett. 82,2725(1999)

Oral Presentation: 16

- (1) International Conference: 6
- (2) Domestic Conference: 10

1.3.7 Researchers in the Computational Materials Science Division

Age and research fields of researchers in the Computational Materials Science Divisions (March 31,1999)

Age of Researchers	Number of Researchers	Physics	Metallurgy	Chemistry	Mechanics
61 ~	0	0	0	0	0
50 ~ 60	6	2	3	0	1
40 ~ 49	5	3	1	0	1
30 ~ 39	8	4	3	1	0
22 ~ 29	2	2	0	0	0
Summation	21	10	7	1	2

1.3.8 Research Budgets in the Computational Materials Science Division

Research budgets in the Computational Materials Science Division

Kinds of budgets	1997 (Yen)	1998 (Yen)
General Research	14,994,000	15,350,000
Multi-core project	20,432,000	20,060,000
Special Coordination funds for Promoting Science and Technology	40,453,000	13,020,000
Computational Materials Research	13,211,000	13,136,000
Joint Research Promotion System on Computational Science and Technology	14,998,000	22,943,000
Supplementary Budgets	0	3,500,000
Summation	104,088,000	88,009,000

1.4 Mechanical Properties Division

Hirosada Irie (Director)

1.4.1 Research work

Basic research on the mechanical properties of metals under various environments, and development of heat-resistant intermetallic compounds and metallic oxides or metallic nitrides with high ductility have been carried out according to the 5th Long-term Plan. Examples of the achievements are:

- (1) The substructure change under fatigue loads and its effect on fatigue properties in titanium alloys have been basically investigated.
- (2) A fine grain aluminum with high ductility at high temperatures has been developed and the dynamic behaviors of the grain boundary of aluminum and zirconium oxides have been experimentally and theoretically investigated.
- (3) Dynamic mechanical properties of structural materials at temperatures from room temperature to ultra low temperature and their testing methods have been studied and an international standardization VAMAS project has been conducted.
- (4) Single crystal Ni₃Al intermetallics with high ductility have been developed and the deformation properties of single- and poly-crystal Ni₃Al have been basically investigated.
- (5) Creep properties of TiAl intermetallics at high temperatures, for gas nuclear power plants, and austenitic and ferritic steels for BWR, have been investigated from the metallurgical point of view, and creep data have been stored.

The members of Mechanical Properties Division, as at March 31, 1999, consisted of 21 researchers, 4 graduate students, 4 guest researchers and 1 secretary.

The productivity of the research activities has been increased since the start of the 5th Long-term Plan. The number of publications, including submitted papers, were about 85 between April 1, 1997 and March 31, 1999. The details are shown in the research activities of each laboratory.

1.4.2 1st Laboratory

(3.1 Biomaterial Research Team)

1.4.3 2nd Laboratory

Toshio Kainuma (Head), Ryuichi Hamano, Fumio Morito, Shozou Ikeda (guest researcher)

1.4.3.1 Research work

- (1) The fatigue behavior and the dislocation substructure during fatigue of titanium alloys such as Ti, Ti-3Al-8V-6Cr-4Mo-4Zr and Ti-6Al-4V were studied to understand the changes in deformation resistance and the strain localization. The effects of maximum stress and R value ($R=-1$ or 0.1) on the fatigue properties of titanium and its alloys were examined for both rolled and annealed states. In addition, the change in the substructure and dislocation configuration under fatigue stress were examined with optical and transmission electron microscopes. The results of fatigue tests showed that softening or hardening process by cyclic stress has a close relationship with substructure change, except for Ti-6Al-4V alloy.
- (2) In order to investigate the influence of environment on the fatigue properties of iron and aluminum alloys, fatigue experiments of these alloys have been carried out under argon gas and corrosive environments. Using cyclic loaded specimens under various environments, crack formation, the deformation behavior of structure and fractured surface have been investigated from macroscopic and microscopic points of view. These results can be utilized for the improvement of anti-corrosion properties by controlling material structures.

<Research themes>

- (1) Relationships between Fatigue Behavior and TEM Structure of Titanium Alloys
(General Research: April 1997 to March 1999, Toshio Kainuma)
- (2) Dependence of Fatigue Property on Environment
(General Research: April 1997 to March 2000, Ryuichi Hamano)

1.4.3.2 Research Products, including papers submitted(April 1,1997 to March 31,1999)

- (1) F.Morito: Structures and Properties of Molybdenum-Rhenium Alloys, *Rhenium and Rhenium Alloys*, B. D. Bryskin, ed., TMS, (1997), 559-568.
- (2) F.Morito: Weldability and Fracture of Mo-50%Re Welds, Proc. 14th Inter. Plansee Seminar, H. Kneringer, P. Rdhammer, P. Wilhartitz, eds., Plansee AG, 1(1997), 1037-1049.
- (3) A.V.Krajnikov, F. Morito, V. N. Slyunyaev: Impurity-induced Embrittlement of Heat-

affected Zone in Welded Mo-based Alloys, *Inter. J. of Refractory Metals and Hard Materials*, 15,325(1997).

- (4) F. Morito, J. Takahashi, S. Muneki, T. Kainuma: Fatigue Behavior and Microstructure of Pure Titanium, *Non-Aerospace Applications of Titanium*, F. H. Froes, P. G. Allen and M. Niinomi, eds., TMS p.p. 29-36(1998).
- (5) V. P. Chakin, F. Morito, V. A. Kazakov, Y. D. Goncharenko, Z. E. Ostrovsky, Radiation Embrittlement of Mo-Re Welds under Low Temperature Irradiation in the SM Reactor, *J. Nuc. Mater.*, 258-263,883(1998).

Oral presentation: 9

- (1) International conference : 3
- (2) Domestic conference: 6

1.4.4 3rd Laboratory

Keiijiro Hiraga (Head), Keishi Nakano, Byung-Nam Kim, Koji Morita, Hideo Yoshinaga (guest researcher)

1.4.4.1 Research work

Basic aspects of high temperature deformation and failure in fine-grained oxide ceramics have been investigated, placing special attention on such dynamic microstructural changes as grain growth, precipitation of intergranular phases, and intergranular cavitation. The following issues are within the scope of this investigation: ① theoretical modeling of static and dynamic grain growth, ② examination of the microstructural aspects of deformation and failure in superplastic zirconia in relation with intergranular segregation and/or precipitation of amorphous phases, ③ quantitative analysis of cavity nucleation and growth behavior in alumina- and zirconia-base materials with and without second phases, ④ analysis of tensile failure in superplastic oxides in relation with intergranular cavitation, microcracking and crack extension behavior, and ⑤ development of new superplastic oxides and related powder processing. The last one has been performed in cooperation with this 3rd Laboratory of the Chemical Processing Division and has achieved a success in firstly attaining substantial superplasticity in alumina-base materials.

<Research themes>

- (1) Research on Fundamental Science of Frontier Ceramics (I)
(Special Coordination Funds for Promoting Science and Technology: April 1995 to March 1998, Keiji Hiraoka)
- (2) Research on Fundamental Science of Frontier Ceramics (II)
(Special Coordination Funds for Promoting Science and Technology: April 1998 to March 2000, Keiji Hiraoka)
- (3) Dynamic Microstructural Change and Mechanical Properties in Ceramics at High Temperatures (General Research: April 1998 to March 2001, Keiji Hiraoka)

1.4.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) T. Uchikoshi, Y. Sakka, K. Ozawa, K. Hiraoka: Preparation and Sintering of Silica-doped Zirconia by Colloidal Processing, *Mater. Res. Soc. Symp. Proc.*, **457**, 33(1997).
- (2) K. Hiraoka, K. Nakano: Cavitation Damage Mechanisms in a Superplastic Zirconia (3Y-TZP), *Mater. Sci. Forum.*, **243-245**, 387(1997).
- (3) K. Hiraoka, H. Yasuda, Y. Sakka: The Tensile Creep Behavior of Superplastic Tetragonal Zirconia Doped with Small Amounts of SiO_2 , *Mater. Sci. Eng.*, **A234**, 1026(1997).
- (4) H. Yasuda, K. Hiraoka: Cavity Damage Accumulation and Fracture in SiO_2 -Doped Zirconia during Superplastic Deformation, *Mater. Sci. Eng.* **A234**, 343(1997).
- (5) T. S. Suzuki, Y. Sakka, K. Hiraoka: Colloidal Processing for Fine Particles of Al_2O_3 -15vol% ZrO_2 System, *J. Jpn. Soc. Powder and Powder Metall.*, **44**, 356 (1997). (in Japanese)
- (6) K. Nakano, T. S. Suzuki, K. Hiraoka, Y. Sakka: Superplastic Tensile Ductility Enhanced by Grain Size Refinement in a Zirconia-Dispersed Alumina, *Scripta Mater.* **38**, 33(1998).
- (7) T. S. Suzuki, Y. Sakka, K. Nakano, K. Hiraoka: The Effect of Ultrasonication on the Colloidal Dispersion of Al_2O_3 and ZrO_2 Powders by Adsorption of Polyelectrolyte, submitted to *J. Am. Ceram. Soc.*
- (8) T. Uchikoshi, Y. Sakka, K. Ozawa, K. Hiraoka: Preparation of Fine-Grained Monoclinic Zirconia Ceramics by Colloidal Processing, *J. Mater. Res.*, **13**, 840(1998).
- (9) T. S. Suzuki, Y. Sakka, K. Nakano, K. Hiraoka: Effect of Ultrasonication on Colloidal Dispersion of Al_2O_3 and ZrO_2 powders in pH Controlled Suspensions, *Mater. Trans. JIM*, **39**, 689 (1998).
- (10) T. Uchikoshi, K. Ozawa, Y. Sakka, K. Hiraoka: Fabrication of Nanostructured

Monoclinic Zirconia Ceramics by Colloidal Processing, Mat. Res. Soc. Symp. Proc., 520, 103(1998).

- (11) Y. Sakka, T. Uchikoshi, K. Ozawa, K. Hiraga: Preparation of Fine-Grained Zirconia Systems by Colloidal Processing, Ceramic Trans., 83, 233(1998).
- (12) Y. Sakka, T. S. Suzuki, K. Nakano, K. Hiraga: Microstructural Control and Superplastic Property of Zirconia Dispersed Alumina Ceramics, J. Jpn. Soc. Powder and Powder Metall. 45, 1186(1998). (in Japanese)
- (13) T. Uchikoshi, Y. Sakka, K. Ozawa, K. Hiraga: Pressure Filtration and Sintering of Fine Zirconia Powder, J. Euro. Ceram. Soc., 18, 669(1998).
- (14) K. Ozawa, Y. Sakka, T. Uchikoshi, K. Hiraga: Preparation of ZrO_2 Powders Doped with SiO_2 by Hydrolysis of Tetraethoxysilane, J. Ceram. Soc. Jpn., 106, 234(1998). (in Japanese)
- (15) Y. Sakka, T. Uchikoshi, K. Ozawa, T. S. Suzuki, K. Hiraga: Preparation of Fine-Grained Zirconia and Alumina Ceramics Systems Through Colloidal Processing, Adv. Sci. Technol., 14, 593(1999).
- (16) Y. Sakka, T. Uchikoshi, K. Ozawa, K. Hiraga: Preparation and Some Properties of SiO_2 -Doped ZrO_2 , Adv. Sci. Technol., 14, 81(1999).
- (17) S. Akutsu, Y. Sakka, T. S. Suzuki, K. Hiraga, S. Itou: Colloidal Processing and Some Properties of Alumina Dispersed Tetragonal Zirconia, Trans. MRS Japan, in press.
- (18) T. S. Suzuki, Y. Sakka, K. Nakano, K. Hiraga: Colloidal Processing and Superplasticity of Monoclinic-, Tetragonal-, and Cubic-Zirconia Dispersed Aluminas, Trans. MRS Japan, in press.
- (19) T. S. Suzuki, Y. Sakka, K. Nakano, K. Hiraga: Superplastic Tensile Ductility in a Zirconia-Dispersed Alumina Produced by Colloidal Processing, Mater. Sci. Forum. 304-306, 489(1999).
- (20) K. Hiraga, K. Nakano, T. S. Suzuki, Y. Sakka: Cavitation Damage During High Temperature Tensile Deformation in Fine-Grained Alumina Doped with Magnesia or Zirconia, Scripta Mater, 39, 1273(1998).
- (21) B-N. Kim, K. Hiraga, Y. Sakka, B-W. Ahn: Analysis of Dynamic Grain Growth Behavior during Superplastic Deformation Using a Grain Boundary Diffusion Mechanism, J. Jpn. Inst. Metals, 63, 589(1999).(in Japanese)
- (22) K. Hiraga, K. Nakano, T. S. Suzuki, Y. Sakka: Cavity Damage Accumulation in a Zirconia-Dispersed Alumina, Mater. Sci. Forum, 304-306, 431(1999).
- (23) K. Morita, Y. Sakka, K. Hiraga: Crack-Like Cavitation in a 0.3 wt% SiO_2 -Doped Tetragonal Zirconia, Mater. Sci. Forum, 304-306, 663(1999).
- (24) K. Morita, K. Hiraga, Y. Sakka: Creep Cavitation Enhanced by Glass Pocket

Formation in a Tetragonal Zirconia Doped with 0.3 wt% Pure Silica, Mat. Res. Soc. Symp. Proc., 539, 125(1999).

- (25) T. Uchikoshi, Y. Sakka, K. Hiraga: Effect of Silica Doping on the Electrical Conductivity of 3 mol% Yttria-stabilized Tetragonal Zirconia Prepared by Colloidal Processing, J. Electroceram. in press.
- (26) K. Hiraga, T. S. Suzuki, Y. Sakka, K. Nakano: Superplasticity in Zirconia-Dispersed Alumina Prepared by Colloidal Processing, Proc. 8th Int. Conf. on Mech. Behaviour of Mater, Victoria, 981(1999).
- (27) B-N. Kim, K. Hiraga, Y. Sakka: Dynamic Grain Growth Enhanced by Diffusional Mechanism during Superplastic Deformation of Ceramics, *Recrystallization and Related Phenomena*, ed. by T.Sakai and H.G.Suzuki, Jpn. Inst. Metals, 507(1999). (in Japanese)
- (28) B-N. Kim, K. Hiraga, Y. Sakka, B-W. Ahn: A Grain Boundary Diffusion Model of Dynamic Grain Growth during Superplastic Deformation, Acta Mater. in press.

Oral Presentation: 44

- (1) International Conference: 16
- (2) Domestic Conference: 28

Award:

Award for Progress in Powder Metallurgy: Japan Society of Powder and Powder Metallurgy, 1998, Keijiro Hiraga, Tohru S. Suzuki, Yoshio Sakka

1.4.5 4th Laboratory

Toshio Ogata(Head), Tetsumi Yuri

1.4.5.1 Research work

- (1) Evaluation techniques for the effects of temperature and gas environments on the deformation and fracture behavior of materials, from room temperature to cryogenic temperatures have been developed. The effects of the solidification structure, the amount of delta-ferrite, and a hydrogen-gas environment on the mechanical properties of austenitic stainless steels and aluminum alloy at low temperatures have been studied and valuable data of high-cycle and low-cycle fatigue properties of those materials at 4K have been obtained.

(2) Constructing the intellectual infrastructure of the evaluation techniques of tensile properties and fracture toughness properties of cryogenic structural materials in cryogenic temperatures and in high-magnetic field has been promoted with international collaboration and the strong relation to the VAMAS(Versailles Project on Advanced Materials and Standards), and suitable testing conditions for the tests in cryogenic temperatures and in high-magnetic field have been proposed. NRIM has been leading this international project on cryogenic structural materials and more than eleven research institutes from seven countries have participated in the international round robin tests of the project.

<Research themes>

- (1) Effect of Cryogenic Temperature and Gas-environment on Deformation and Fracture Behavior (General Research: April 1996 to March 1999, Toshio Ogata)
- (2) Constructing Intellectual Infrastructure through International Pre-standardization for Promoting Practical Use of Advanced Materials - Characterization and Evaluation of Cryogenic Structural Materials (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 2000, Toshio Ogata)

1.4.5.2 Research Products, including papers submitted (April 1, 1997 to March, 1999)

- (1) T. Yuri, T. Ogata, M. Saito, Y. Hirayama: High Cycle Fatigue Properties of Austenitic Stainless Steel Welds at Cryogenic Temperatures, Tetsu to Hagane, 84, 888(1998) (in Japanese)
- (2) H. Nakagawa, M. Yabumoto, M. Saito, S. Okaguchi, H. Fujii, Y. Wada, T. Iida, T. Ogata: Effects of Delta-ferrite Phase on the Mechanical Properties of the Weld Metals of Austenitic Stainless Steels at Low Temperatures, Proc. World Hydrogen Energy Conf. XII, 3, 1853(1998)
- (3) H. Fujii, M. Yabumoto, T. Ogata, M. Hayashi, M. Saito, S. Okaguchi, Y. Wada, T. Iida, H. Nakagawa: Evaluation of Mechanical Properties of Austenitic Stainless Steels and Aluminum Alloy in Liquid Hydrogen, Proc. World Hydrogen Energy Conf. XII, 3, 1892(1998)
- (4) T. Ogata, D. Evans, A. Nyilas: VAMAS Round Robin Tests on Composite Material and Solder at Liquid Helium Temperature, Advances in Cryogenic Engineering Materials, 42, 269(1998)

Oral presentation: 14

(1) International conference: 3

(2) Domestic conference: 11

1.4.6 5th Laboratory

Toshiyuki Hirano (Head), Aiko Aoki, Motoharu Imai, Toshio Mawari, Masahiko Demura, Naoya Ohmura (guest researcher)

1.4.6.1 Research work

(1) Mechanical and electrical properties of intermetallic compounds, aluminides and silicides are focused to study. ① The room-temperature brittleness of polycrystalline Ni_3Al has been significantly improved by directional solidification. ② The stoichiometry effect of Ni_3Al on plastic deformation has been found using high-quality single crystals. ③ Pressure-induced phase transitions in BaSi_2 have been measured by in-situ x-ray diffraction.

(2) Certain species of microorganism accelerate the aqueous oxidation of metals and inorganic compounds. *Thiobacillus ferrooxidans*, was applied in this study. A significant catalytic influence of the microorganism on the oxidation process included the secondarily solid phase on the surface. ① The role of the crystallographic orientation of the sulfide on the reaction mechanism of the bacterial oxidation were clarified. ② An AFM observation in tapping mode at the pyrite surface was carried out. The untreated (100) surface was distinguished by a regular array of round humps at AFM, however, the round humps disappeared and etching pits were produced by the oxidation after 30 hrs oxidation. The results obtained here will receive wide application to solving the environmental problems caused by microorganism; bacterial corrosion of metals or acidic drainage of closed mines.

<Research themes>

(1) Fabrication of Ni_3Al -Base Intermetallic Compounds by Unidirectional Solidification

(General Research: April 1997 to March 2000, Toshiyuki Hirano)

(2) Fundamental Studies of the Microbial Reaction with Inorganic Compounds

(General Research: April 1996 to March 1999, Aiko Aoki)

1.4.6.2 Research Products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) M.Demura, T.Hirano: Stress Response by the Strain-Rate Change in Binary, Stoichiometric Ni_3Al Single Crystal, *Phil. Mag. Lett.* **75**, 143 (1997)
- (2) E.P.Geroge, M.Imai, T.Hirano: Effect of Alloy Stoichiometry on Grain Boundary Chemistry and Fracture Behavior of Directionally Solidified Ni_3Al , *Intermetallics*, **5**, 425 (1997)
- (3) T.Hirano, T.Mawari, M.Demura, Y.Isoda: Effect of Directional Growth-Rate on the Mechanical Properties of Ni_3Al , *Mater. Sci. Eng. A* **239-240**, 324 (1997)
- (4) D.Golberg, M.Demura, T.Hirano: Compressive Flow Stress of a Binary Stoichiometric Ni_3Al Single Crystal, *Scripta Mater.*, **37**, 1777(1997)
- (5) D.Golberg, M.Demura, T.Hirano: High Temperature Yield Stress of Binary Stoichiometric and Al-rich Ni_3Al Single Crystals, 2nd Inter. Symp. on Structural Intermetallics, Champion, Pennsylvania, Sep.21-25, 1997
- (6) M.Imai, T.Hirano, T.Kikegawa, O.Shimomura: In-situ Measurements of Orthorhombic-to-Trigonal Transition in BaSi_2 under High-Pressure and High-Temperature Conditions, *Phys. Rev. B*, **55**, 132 (1997)
- (7) M.Imai, T.Hirano, T.Kikegawa, O.Shimomura: Phase Transitions of BaSi_2 at High Pressures and High Temperatures, *Phys. Rev. B*, **58**, 11922 (1998)
- (8) D.Golberg, M.Demura, T.Hirano: Single Crystal Growth and Characterization of Binary Stoichiometric and Al-rich Ni_3Al , *J. Cryst. Growth*, **186**, 624(1998)
- (9) D.Golberg, M.Demura, T.Hirano: Effect of Al-rich Off-Stoichiometry on the Yield Stress of Binary Ni_3Al Single Crystals, *Acta Mater.*, **46**, 2695 (1998)
- (10) T.Hirano, D.Golberg, M.Demura: Effect of Deviation from Stoichiometry on the Mechanical Properties of Single-Crystal Ni_3Al , 2nd Inter. Symp. on Interstitial and Substitutional Solute Effects in Intermetallics, Rosemont, Illinois, October 11-15, 1998
- (11) T.Hirano, M.Demura, E.P.George, O.Umezawa: Recrystallization in Binary Stoichiometric Ni_3Al , Grain Growth in Polycrystalline Materials III, Pitttuburgh, PA, June 14-19, 1998
- (12) D.Golberg, M.Demura, T.Hirano: Structure and Yield Strength of Directionally Solidified Ni_3Al Intermetallic Premelted with MoSi_2 , *Intermetallics*, **7**, 109 (1999)
- (13) T.Hirano, M.Demura, E.P.George, O.Umezawa: Fabrication of Large-Grained Binary Stoichiometric Ni_3Al , *Scripta Mater.*, **40**, 63 (1999)
- (14) T.Hirano, M.Demura, D.Golberg: Compliance to Schmid Law in the Stress Anomaly Regime of Binary Stoichiometric Ni_3Al , *Acta Mater.*, in press
- (15) A.Aoki: Characterization of the Secondary Mineral Particles during the Biooxidation of Pyrrhotite, *Proc. IBS'97* (1997) PM31

- (16) A. Aoki: Acid Bacterial Leaching of Pyrite Single Crystal, Proc. IBS'99 (1999) vol.A, p35
- (17) A. Aoki: Treatment of Inorganic Metal Sulfide by Microorganism, Natural Resources Technology 44,32(1997) (in Japanese)
- (18) A. Aoki: Design of Bacteria Monitoring Apparatus by Using a Widely Used Biological Microscopy and Analysis of the Pictures Obtained, Res. Rep. for the Grand of the Japan Mining Promotion Society (1997) 94 (in Japanese)

• **Oral Presentation: 22**

- (1) International Conference: 4
- (2) Domestic Conference: 18

1.4.7 6th laboratory

Hirosada Irie (Head), Tatsuhiko Tanabe (Head of 1st Laboratory.), Isao Mutoh, Masakazu Fujitsuka, Masayoshi Yamazaki, Hiromichi Hongo, Takashi Watanabe, Munenori Nakazawa (guest researcher)

1.4.7.2 Research work

- (1) Properties of TiAl intermetallics for gas nuclear power plants and W-Re alloys for the first wall of fusion reactors have been investigated. ① The effect of microstructures on the creep rupture properties of TiAl at ultra high temperatures of up to 1373K has been investigated. Larger grain size leads to a better creep rupture life and strength. The presence of α_2 phase at grain boundaries causes also better creep strength at higher temperatures and better rupture ductility at lower temperatures. ② The effect of Re content on the thermal and mechanical properties of W was investigated. Thermal conductivity decreased with increase of Re content. Pure metal(W) and alloys showed different temperature dependence of diffusivity from each other. Further, the effect of neutron irradiation on the thermal conductivity of W-Re alloys was studied and the diffusivity of W and W-5%Re decreased at a neutron fluence larger than 10^{20} n/cm², while those of 10%Re and 25%Re increased. Room temperature ductility and strength of W increased with an increase in Re content.
- (2) The creep properties of welded joints in austenitic and ferritic steels for Fast Breeder Reactor have been investigated and the life prediction technique using these data is

under development. ①The relation between creep behavior and microstructural evolution was investigated in SUS304 steel plates. During the creep rupture test, for more than 10^5 h the creep rate-time curve showed a complicated behavior depending on precipitation of carbides and/or the σ phase. ② The creep rupture test results on the welded joints of 2.25Cr-1Mo steel multi-layer weldment showed that the fracture location shifted from the base metal to the weld metal as the stress decreased. ③ Creep damage in 316FR steel was inspected ultrasonically. The possibility of assessment of creep damage by ultrasonic inspection in austenitic stainless steel was indicated. ④ The creep rupture curve and rupture life of welded joints of SUS 304 plates can be successfully simulated with use of a simple analysis model, developed at the NRIM, considering residual stress.

<Research themes>

- (1) Improvement of High Temperature Properties in Materials for High/Ultra-High Temperature Use

(General Research: April 1996 to March 1999, Tatsuhiko Tanabe)

- (2) Remaining Life Prediction for Weldments in FBR Based on Creep Damage Evaluation

(Nuclear Energy Research: April 1996 to March 2001, Tatsuhiko Tanabe)

1.4.7.3 Research Products, including papers submitted (April 1,1997 to March 31,1999)

- (1) I. Mutoh, Y. Kawano, T. Tanabe, M. Nakamura: Effect of Microstructure on the Creep Rupture Properties of Ti-49at.%Al at High Temperatures, *Report of the 123 Committee on Heat-Resisting Materials and Alloys*, Japan Soc. for the Promotion of Science., **38**, 139(1997). (in Japanese)
- (2) I. Mutoh, Y. Kawano, T. Tanabe, M. Nakamura: Effect of Microstructure on the Creep Rupture Properties of Ti-49at.%Al, *ICM&M'97 Inter. Conf. on Materials and Mechanics'97*, p.335,(1997).
- (3) T. Tanabe, Y. Kurata, I. Mutoh, H. Tsuji, K. Hiraga, M. Shindo: Creep Damage in Welded Joints of Ni-Base Heat-Resistant Alloy Hastelloy XR, *J. Mater Sci. and Eng*, **A234-236**, 1087(1997).
- (4) M. Fujitsuka, I. Mutoh, T. Tanabe, T. Shikama: High Heat Load Test of Tungsten and Tungsten-Rhenium Alloys, *Proc.1997 Fall Meeting of the Atomic Energy Soc. of Japan*, vol.1,p.144(1997). (in Japanese)

- (5) H. Tsuji, T. Tanabe, Y. Nakasone, T. Nakajima: Creep Behavior of Hastelloy XR under Varying temperature/Load, *ibid.*, vol.3,p.920(1997).(in Japanese)
- (6) I.Mutoh,Y. Kawano, T. Tanabe, M. Nakamura: Effect of Microstructure on the Creep Rupture Properties of Ti-51at.%Al, Abstract of 122nd Meeting of Japan Institute of Metals,p271(1998).(in Japanese)
- (7) M. Nakamura, M. Nobuki, T. Tanabe, T. Kumagai, I. Mutoh, E. Abe: Microstructural Control and High Temperature Properties of TiAl base Alloys, *Inter. metallics*, 6, 637(1998).
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- (9) T. Kumagai, S. Utsunomiya, I. Mutoh, T. Tanabe, M. Nakamura: Observation of Creep Deformation Structure in Ti-51at.%Al, Abstract of 123rd Meeting of JIM,p352(1998).(in Japanese)
- (10) M. Fujitsuka, I. Mutoh, T. Tanabe, T. Shikama: Effect of Re Addition on Thermal and Mechanical Properties of W, *Advances in Science and Technology Vol.24, Innovative Materials in Advanced Energy Technologies*, ed. P.Vincenzini, Techna, Faenza, p339(1999).
- (11) M. Song, K. Furuya, T. Tanabe, T. Noda: High-Resolution Electron Microscopy of γ -TiAl Irradiated with 15KeV Helium Ions at Room Temperature, *J.Nucl.Mater.*, 271-272, 200(1999).
- (12) M. Yamazaki, Y. Yamauchi: Construction and Utilization of the Network System at NRIM, *Surface Technology*, 48, 623(1997). (in Japanese)
- (13) K. Nakacho, M. Yamazaki: Estimation of Creep Life of Thick Welded Joints Using a Simple Model, *Proc. Welding Structure Sympo'97*,p.93(1997). (in Japanese)
- (14) M. Yamazaki, H. Hongo, T. Watanabe, J. Kinugawa, T. Tanabe, Y. Monma: Local Variability of Creep Properties for 304 Stainless Steel Welds, *Proc. 35th Symp. on Strength of Materials at High Temperatures*, p.169(1997).
- (15) H. Hongo, M. Yamazaki, T. Watanabe, J. Kinugawa, T. Tanabe, Y. Monma: Creep Behavior of Low Carbon-Medium Nitrogen Type 316 Weld Metals, *ibid.*, p.164.
- (16) M. Yamazaki, H. Hongo, T. Watanabe, K. Kimura, T. Tanabe, H. Irie, T. Matsuo: Correlation between Creep Rate-Time Curve and Microstructural Parameter in austenitic Heat Resisting Steel, Report of the 123 Committee on Heat Resisting Materials and Alloys, Japan Soc. for the Promotion of Science. vol.39, p.111(1998), *(CAMPS-ISIJ,vol.11, p.1169(1998))* (in Japanese)
- (17) M. Yamazaki, H. Hongo, T. Watanabe, K. Kimura, T. Tanabe, H. Irie, T. Matsuo:

- Temperature Dependence of Creep Rate-Time Curve in SUS304 Ruptured at approximately 105 h, Report of the 123 Committee on Heat-Resisting Materials and Alloys, Japan Soc. for the Promotion of Science.vol.39, p.183(1998).(in Japanese)
- (18) M. Yamazaki, H. Hongo, T. Watanabe, K. Kimura, T. Tanabe, H. Irie, T. Matsuo: Effect of Temperature on Creep Deformation in SUS304, Report of the 123 Committee on Heat-Resisting Materials and Alloys, Japan Soc. for the Promotion of Science, vol.40(1999), p.25.(CAMPS-ISIJ,vol.11(1998),p.1170.) (in Japanese)
- (19) M. Yamazaki, H. Hongo, T. Watanabe, K. Kimura, T. Tanabe, H. Irie, T. Matsuo: Effect of Carbon and Nitrogen on Creep Rate-Time Curves in SUS304, Report of the 123 Committee on Heat-Resisting Materials and Alloys, Japan Soc. for the Promotion of Science,vol.40, p.127(1999). (in Japanese)
- (20) M. Yamazaki, H. Hongo, T. Watanabe, H. Irie: Creep Properties of Welded Joint(I), J. Japan Welding Soc., 67,475(1998).(in Japanese)
- (21) T. Watanabe, M. Yamazaki, H. Hongo, J. Kinugawa: Creep Properties of Welded Joint(II), J. Japan Welding Society, 67,531(1998). (in Japanese)
- (22) H. Hongo, M. Yamazaki, T. Watanabe, J. Kinugawa: Creep Properties of Welded Joint(III), J. Japan Welding Soc., 67,1(1998). (in Japanese)
- (23) K. Kubo, T. Watanabe, M. Tabuchi, J. Kinugawa, F. Abe: Creep Rupture Properties of High Chromium Steel with Simulated HAZ Heat-resisting, CAMPS-ISIJ, 11,1251(1998). (in Japanese)
- (24) T. Watanabe, M. Yamazaki, J. Kinugawa, T. Tanabe, N. Onogi: Hardness Distribution Change due to Aging in Fine-Grained Region of Heat-Affected Zone in 2.25Cr-1Mo Weld, CAMPS-ISIJ, 12, 436(1999). (in Japanese)
- (25) T. Watanabe, M. Yamazaki, H. Hongo, J. Kinugawa, T. Tanabe, Y. Monma: Long-Term Creep-Rupture Properties and Microstructure of Weld Metal on 2.25Cr-1Mo Steel Thick Plate, J. Society Mater. Sci. Japan, 48, 122(1999). (in Japanese)
- (26) H. Hongo, M. Yamazaki, T. Watanabe, J. Kinugawa, T. Tanabe, Y. Monma: Creep Deformation Behavior of Weld metal and Heat Affected Zone on 316FR Steel Thick Plate Welded Joint, J. Society Mater. Sci. Japan, 48, 116(1999). (in Japanese)
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- (28) T. Watanabe, M. Yamazaki, H. Hongo, T. Tanabe: Long-Term Creep Rupture Properties and Microstructural Change in Welds of 2.25Cr-1Mo Thick Plates, Progress in Mechanical Behavior of Materials and Processing, ICM8, 2, 659(1999).
- (29) T. Watanabe, M. Yamazaki, H. Hongo, T. Tanabe: Creep Behavior of Weld Metals for

Low Carbon-Medium Nitrogen Type 316 Steel, Progress in Mechanical Behavior of Materials and Processing, ICM8, 2, 629(1999).

- (30) M. Yamazaki, H. Hongo, T. Watanabe, T. Tanabe: Influence of Welding Performance on Creep Rupture Properties of Welds in 304 Stainless Steel Thick Plates, Progress in Mechanical Behavior of Materials and Processing, ICM8, 2, 641(1999).

Oral presentation : 30

(1) International conference : 2

(2) Domestic conference: 28

1.4.7 Researchers in Mechanical Properties Division

Age and research fields of researchers in Mechanical Properties Division (March31,1999)

Age of Researchers	Number of Researchers	Metallurgy	Solid state Physics	Chemistry	Others
60 ~	2	2			
50 ~ 59	11	6		1	4
40 ~ 49	8	3		2	3
30 ~ 39	2	1	1		
22 ~ 29	2	2			
Summation	25	14	1	3	7

1.4.8 Research Budget in Mechanical Properties Division

Research budgets in Mechanical Properties Division (yen)

kinds of budgets	1997	1998
Nuclear Energy Research	8,101,000	11,597,000
General Research	21,120,000	21,876,000
Special Coordination Funds for Promoting Science and Technology	27,945,000	32,534,000
Summation	57,166,000	66,007,000

1.5 Materials Processing Division

Hirowo G. Suzuki (Director)

1.5.1 Research work

The processing techniques for the fabrication of materials are very important to realize advanced materials. The developments of the processing techniques to control the microstructures, the physical properties as well as the functions of materials are indispensable and attractive research targets. The main targets of this Division are as follows.

- (1) To make clear the processing mechanisms, such as solid, liquid and vapor phase processing, solidification, powder and joining processes.
- (2) To develop new computer aided processing techniques to fabricate advanced materials requiring a lower processing energy, in view of ecological needs.
- (3) To clarify the deformation and fracture mechanisms of the advanced materials related to the research work in this Division, and to obtain fundamental physical data in order to analyze or separate the elements of advanced materials.

1.5.2 1st Laboratory

Tadayuki Fujii (Head), Kenjiro Goto, Koji Maiwa, Kinichi Honda,
Kazushige Kamihira

1.5.2.1 Research work

- (1) A new technique for preparation of the oriented single crystals of refractory metals through solid state processes has been developed. Consequently, molybdenum and tungsten single crystals with a desired crystallographic orientation could be easily produced.
- (2) Selenium (VI) removal from wastewater has been studied by using a fluidized bed process.
- (3) Crystal growth process in a $\text{Sr}(\text{NO}_3)_2\text{-H}_2\text{O}$ binary peritectic system have been studied by using an optical microscope. It was found that both high- $(\text{Sr}(\text{NO}_3)_2)$ and low- $(\text{Sr}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O})$ temperature phases can grow simultaneously in a liquid with a composition near the peritectic point when the liquid is cooled below T_p .

<Research themes>

- (1) Control of the Oriented Single Crystals of Refractory Metals through Solid State

Low Carbon-Medium Nitrogen Type 316 Steel, Progress in Mechanical Behavior of Materials and Processing, ICM8, 2, 629(1999).

- (30) M. Yamazaki, H. Hongo, T. Watanabe, T. Tanabe: Influence of Welding Performance on Creep Rupture Properties of Welds in 304 Stainless Steel Thick Plates, Progress in Mechanical Behavior of Materials and Processing, ICM8, 2, 641(1999).

Oral presentation : 30

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- (3) Crystal growth process in a $\text{Sr}(\text{NO}_3)_2\text{-H}_2\text{O}$ binary peritectic system have been studied by using an optical microscope. It was found that both high- $(\text{Sr}(\text{NO}_3)_2)$ and low- $(\text{Sr}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O})$ temperature phases can grow simultaneously in a liquid with a composition near the peritectic point when the liquid is cooled below T_p .

<Research themes>

- (1) Control of the Oriented Single Crystals of Refractory Metals through Solid State

- Process (General Research: April 1997 to March 2000, Tadayuki Fujii)
- (2) Selenium (VI) Removal from Wastewater by a Fluidized Bed Process (General Research: April 1997 to March 2000, Kenjiro Goto)
- (3) Crystal growth and dissolution in Peritectic System (General Research: April 1998 to March 2001, Koji Maiwa)

1.5.2.2 Research products, including papers submitted(April 1, 1997 to March 31, 1999)

- (1) K. Maiwa, M. Plomp, W.J.P van Enckevort, P.Bennema: AFM Observation of Barium Nitrate {111} and {100} Face: Spiral Growth and Two-Dimensional Nucleation Growth, *J. Crystal Growth* No.186, 214 (1998).
- (2) K. Kamihira, K. Honda, T. Fujii: Preparation of Molybdenum Single Crystal with a Desired Orientation by Means of Zone Annealing, *J. Jpn. Soc. Powder and Powder Metallurgy*, 44, 770(1997). (in Japanese)
- (3) K. Honda, K. Kamihira, T. Fujii : Crystallographic Orientation Relationship between the Primary and Secondary Recrystallized Grains in Multi-Layer Molybdenum Crystals, *J. Jpn. Soc. Powder and Powder Metallurgy*, 45 ,801(1998).
- (4) M. Shimizu, K. Itoh, T. Fujii, T. Igarashi, K. Okamoto: Solar Thermal Thruster Made of Single Crystal molybdenum, *Acta Astronautica*, 41 ,23(1998).
- (5) M. Shimizu, K. Itoh, T. Fujii, T. Igarashi, K. Okamoto: Single Crystal Molybdenum Solar Thermal Thruster for Microsatellites, *Proc. 49th Inter. Astronautical Congress*, Sep. 28-Oct. 2 (1998), Melbourne, Australia.
- (6) M. Plomp, K. Maiwa, W.J.P. Enckevort : Atomic Force Microscopy Observations of Hollow Cores on the {111} and {100} Faces of Barium Nitrate, *J. Crystal Growth* No.198/199 ,246(1999).
- (7) K. Tsukamoto, E.Yokoyama, K.Maruyama, K. Maiwa, K.Shimizu, R.F.Sekerka, S.Morita, S. Yoda: Transient Crystal Rate in Microgravity, *J. Jpn. Soc. of Microgravity* 15, 2(1998).
- (8) X. L. Liu, K. Maiwa, T. Tsukamoto: Heterogeneous Two-dimensional Nucleation and Growth Kinetics, *J. Chem. Phys.* 106,1870(1997).

Oral presentation: 4

- (1) International Conference: 0
- (2) Domestic Conference: 4

Patent Application :

- (1) Oriented Molybdenum or Tungsten Single Crystal and Manufacturing Method

thereof: T. Fujii, K. Honda , July 8 , 1997, USA Patent Application No. 08/890,005.

Registered Patent:

- (1) Single Crystal of Dispersion Strengthened Molybdenum and Manufacturing Method thereof: T. Fujii, K. Honda, Oct.3, 1997, Japan Patent No.2702669.
- (2) Single Crystal of Precipitation Strengthened Molybdenum and Manufacturing Method thereof: T. Fujii, K. Honda, July 8, 1997, Japan Patent No. 2535774.
- (3) Oriented Molybdenum or Tungsten Single Crystal and Process for Production thereof: T. Fujii, K. Honda, March 30, 1999, Japan Patent No.2920202.

1.5.3 2nd Laboratory

Chitoshi Masuda (Head), Youichi Ogawa, Osamu Kujirai, Eiju Takakura, Yoshihisa Tanaka, Yufu Liu, Shigeki Sakai (visiting researcher), Shoujin Sun (STA fellow), Evelyne Peron Gonia (STA fellow)

1.5.3.1 Research work

- (1) New high strength and high electrical conductive Cu-Cr in situ composites were developed and the deformation and fracture mechanisms were examined for those composites.
- (2) Interfacial damage mechanisms of titanium alloy matrix composites for a high specific strength and high temperature resistance were examined and modeled. The interfacial sliding mechanism was very important to prolong the fatigue life at high temperatures.
- (3) The mechanical properties for metal matrix composites, such as the tensile properties for SiCw/A2009 MMC at room and high temperatures, were evaluated and the testing method was proposed to ISO standard. In the near future, fatigue properties will be evaluated for SiCw/A2009MMC and titanium alloy matrix composite.
- (4) Interfacial damage during the cyclic loading condition for titanium alloy matrix composites is very important for their practical application. The effect of the interfacial damage on crack propagation at high temperatures are examined and the damage process is calculated in order to improve the fatigue properties.
- (5) High-lying energy levels of light and middle rare-earth atoms have been studied by using the laser resonance photoionization method and the characteristics of surface

ionization have also been studied.

<Research themes>

- (1) High Strength and High Electrical Conductive Cu Base in-situ Composites (General research: April 1998 to March 2001, Hirowo G. Suzuki, Eiju Takakura(1999-)).
- (2) Effect of Interfacial Damage on Fatigue Fracture Mechanism for Titanium Alloy Matrix Composite at High Temperature (General research: April 1996 to March 1999, Chitoshi Masuda).
- (3) Evaluation of Mechanical Properties for Metal Matrix Composites. (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 2000, Chitoshi Masuda)
- (4) Effect of Interfacial Damage on Fatigue Crack Propagation Mechanism for Titanium Alloy Matrix Composites (General Research; April 1999 to March 2002, Chitoshi Masuda)
- (5) Study on the Efficiency of Resonance Photoionization Process (General Research: April 1995 to March 1998, Youichi Ogawa)
- (6) Application of Ionization Separation Technique to Gaseous Process (General Research: April 1998 to March 2001, Youichi Ogawa)

1.5.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) Y. F. Liu, C. Masuda, Y. Tanaka: Analysis of Cylindrical Model with Circumferential Crack of Composite, JSME, 63, 1886(1997) (in Japanese)
- (2) J. L. Bobet, C. Masuda: X-ray Diffraction Analysis of Thermal Residual Stresses in SiC/Ti-15-3 Composite, Eur. J. Solid Inorg. Chem., 34, 1093(1997).
- (3) K. Adachi, S. Tsubokawa, T. Takeuchi, H. G. Suzuki: Plastic Deformation of Cr Phase in Cu-Cr Composite during Cold Rolling, J. Japan Inst. Metals, 61, 391(1997). (in Japanese)
- (4) K. Adachi, S. Tsubokawa, T. Takeuchi, H. G. Suzuki: Strengthening Mechanism of Cold-Drawn Wire of In-situ Cu-Cr Composite, J. Japan Inst. Metals, 61, 397(1997). (in Japanese)
- (5) K. Mihara, T. Takeuchi, H. G. Suzuki: Effect of Carbon Addition on Solidification Structure and Strength of Cu-Cr in situ Composite, J. Japan Inst. Metals, 61, 1044(1997). (in Japanese)
- (6) Y. Jin, K. Adachi, T. Takeuchi, H. G. Suzuki: Correlation between the Electrical Conductivity and Aging Treatment for a Cu-15wt%Cr Alloy Composite

- Formed in situ, *Mater. Lett.*, **32**, 307(1997).
- (7) J. L. Bobet, C. Masuda: Estimation of Residual Stresses in SiC/Ti-15-3 Composites and their Relaxation during a Fatigue Test, *J. Mat. Sci.*, **32**, 6357(1997).
 - (8) K. Mihara, T. Takeuchi, H. G. Suzuki: Effect of Zr on Aging Characteristics and Strength of Cu-Cr In-situ Composite, *J. Japan Inst. Metals*, **62**, 238(1998).
 - (9) K. Mihara, T. Takeuchi, H. G. Suzuki: Effect of Ti on Aging Characteristics and Strength of Cu-Cr In-situ Composite , *J. Japan Inst. Metals*, **62**, 599(1998). (in Japanese)
 - (10) Y. F. Liu, Y. Tanaka, C. Masuda: Analysis of Fiber-Matrix Cylindrical Model with a Circumferential Crack, *Int. J. Fracture*, **88**, 87(1998).
 - (11) Y. Jin, K. Adachi, T. Takeuchi, H. G. Suzuki: Aging Characteristics of Cu- Cr In-situ Composite, *J. Mater. Sci.*, **33**, 1333(1998).
 - (12) Y. Jin, K. Adachi, T. Takeuchi, H. G. Suzuki: Correlation between the Cold-Working and Aging Treatments in a Cu-15wt%Cr in situ Composite, *Metall. Trans.*, **29A**, 2195(1998).
 - (13) Y. F. Liu, Y. Tanaka, C. Masuda: Debonding Mechanisms in the Presence of the Interphase in Composite, *Acta Mater.*, **46**, 5237(1998).
 - (14) Y. F. Liu, C. Masuda , R. Yuuki: Parameters on the Fracture Behavior of Fiber-Reinforced Ceramics, *Mechanics of Materials*, **29**, 111(1998).
 - (15) Y. F. Liu, Y. Tanaka, C. Masuda: An Efficient BEM to Calculate Weight Functions and its Application to Bridging Analysis in an Orthotropic Medium, *Computational Mechanics*, **22**, 418(1998).
 - (16) K. Mihara, T. Takeuchi, H. G. Suzuki: Effect of Carbon Addition on Solidification Structure and Strength of Cu-Cr In-situ Composite, *Mater. Trans, JIM*, **39**, 1093(1998).
 - (17) S. Q. Guo, Y. Kagawa, Y. Tanaka, C. Masuda: Microstructure and Role of Outermost Coating for Tensile Strength of SiC Fiber, *Acta Mater.* **46**, 5941(1998).
 - (18) S. Q. Guo, Y. Kagawa, H. Saito, C. Masuda: Microstructural Characterization of Interface in SiC Fiber-Reinforced Ti-15-3 Matrix Composite, *Mat. Sci. Eng.*, **A246**, 25(1998).
 - (19) O. Kujirai, Y. Ogawa: Observation of Even-Parity Autoionization States of Lutetium Atom by Optogalvanic Spectroscopy, *J. Phys Soc. Japan*, **67**, 1056(1998)
 - (20) Y. Tanaka, Y. F. Liu, C. Masuda: Observation of Fatigue Damage Process in

- SiC/Ti-15-3 Composite at High Temperature, *Met. Trans.*, 30A, 221(1999).
- (21) D. L. Zhang, K. Mihara, E. Takakura, H. G. Suzuki: Effect of the Amount of Cold Working and Aging on the Ductility of a Cu-15%Cr-0.2%Ti in situ Composite, *Mater. Sci. & Engineering A*, A266,99(1999).
 - (22) H. G. Suzuki, E. Takakura, D. Eylon: Hot Strength and Hot Ductility of Titanium Alloys - a Challenge for Continuous Casting Process, *Mater. Sci. & Engineering A*, A263,230(1999).
 - (23) Y. Kagawa, C. Masuda: Numerical Analysis of the Interface Problem in Continuous Fiber Ceramic Composites, *Computational Materials Design*, Edited by T. Saito, Springer, 280 (1999).
 - (24) Yufu Liu, Y. Kagawa, R. Yuuki: Analysis of Initial Debonding Propagation in Layered Materials, *J. Mat. Sci., Letter*, 18, 131(1999).
 - (25) Y. Ogawa, O. Kujirai: Study of Even-Parity Autoionization States of Lutetium Atom by Laser Resonance Photoionization Spectroscopy, *J. Phys. Japan*, 68, 428(1999)
 - (26) Y. F. Liu, Y. Tanaka, C. Masuda: In-situ Detection of Fiber Break and Analysis of its Effect on Stress Transfer during Tensile Test of a Metal Matrix Composites, *Composites: Manufacturing and Applied Science* (in press).
 - (27) Y. F. Liu, Y. Kagawa: Release Rate for Interface Debond Crack in Ceramic-Matrix Composites, *Composite Science and Technology*(submitted).

Oral Presentation: 27

- (1) International Conference: 8
- (2) Domestic Conference : 19

Patent :

- (1) Manufacturing of High Strength and High Electrical Conductivity Cu-base Alloy: K. Adachi, T. Takeuchi, T. Mitsui, H. G. Suzuki: Japan Patent No. 2869859.
- (2) Manufacturing of High Strength and High Electrical Conductivity High Cr-added Cu Alloy: K. Mihara, T. Takeuchi, N. Sakuma, H. G. Suzuki, S. Miyauchi: Japan Patent No. H10-140267 .
- (3) Manufacturing of High Strength and High Electrical Conductivity High Cr-added Cu Alloy: K. Mihara, T. Takeuchi, N. Sakuma, H. G. Suzuki, S. Miyauchi: Japan Patent No. 2895796

Award:

1998.3. Mishima Award, Japan Iron & Steel Institute, Hirowo G. Suzuki

1.5.4 3rd Laboratory

Yoshio Sakka (Head), Satoru Ohno, Kiyoshi Ozawa, Tetsuo Uchikoshi, Hideo Okuyama, Tohru S. Suzuki, Mika Eguchi (domestic research fellow), Satoshi Akutsu (joint-graduate course student), Kenichi Sodeyama (visiting researcher)

1.5.4.1 Research work

- (1) Fine-grained materials are prepared through colloidal processing, such as slip casting, pressure filtration, tape casting, etc. Excellent mechanical and electric properties are obtained. Especially on superplasticity, in collaboration with the Mechanical Property Division, approximately 550% tensile elongation for alumina-based ceramics, and 1000% tensile elongation for zirconia-based ceramics, without a glass-phase, were established.
- (2) Proton conductive antimonite fine powders and films were prepared by soft chemical processing, such as ion-exchanged reaction, sol-gel processing and autoclave reaction. High proton conductive antimonite-based film at room temperature was prepared and the conduction mechanism was examined.
- (3) Nanoparticles were prepared by DC plasma or RF plasma methods. Dumbbell-type metal (Fe,Co,Ni)-TiN nanocomposite particles were prepared showing unique catalytic properties. Titanium oxide fine powders prepared by RF plasma CVD showed excellent photocatalytic activity for the decomposition of water.
- (4) Research for advanced materials processing is conducted using external fields to create non-linear amplification effects. High microstructure control using extremely high-magnetic fields or electric fields was examined.

<Research themes>

- (1) Characterization of Photocatalytic Properties of Ultrafine Particles Synthesized by Plasma Processing (General Research: April 1996 to March 1999, Satoru Ohno)
- (2) Synthesis and Utilization of Mesoporous Materials (General Research: April 1996 to March 1999, Yoshio Sakka)
- (3) Material Processing Using External Fields to Create Non-linear Amplification Effects (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 2001, Yoshio Sakka)

1.5.4.2 Research Products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) T.S.Suzuki, Y. Sakka, K. Hiraga: Colloidal Processing for Fine Particles of Al_2O_3 -15vol% ZrO_2 Systems, J. Japan Soc. Powder Powder Metall., **44**, 356(1997). (in Japanese)
- (2) K. Ozawa, Y. Sakka, M. Amano: Preparation and Electrical Conductivity Measurement of LiSbO_3 thin Films, Mater. Res. Soc. Symp. Proc., **453**, 617 (1997).
- (3) Y. Sakka, K. Sodeyama, T. Uchikoshi, K. Ozawa, M. Amano: Characterization of Proton Conducting Antimonic Acids with Amorphous, Cubic and Monoclinic Structures, Mater. Res. Soc. Symp. Proc., **453**, 629(1997).
- (4) T. Uchikoshi, Y. Sakka, K. Ozawa, K. Hiraga: Preparation and Sintering of Silica-Doped Zirconia by Colloidal Processing, Mater. Res. Soc. Symp. Proc., **457** 33(1997).
- (5) Y. Sakka, H. Okuyama, T. Uchikoshi, S. Ohno: Morphology and Hydrogen Desorption Characteristics of Ni-TiN Nanocomposite Particle Prepared by RF Plasma, Nanostruct. Mater. **8**, 465 (1997).
- (6) K. Sodeyama, Y. Sakka, Y. Kamino, K. Hamaishi: Processing and Some Properties of Shirasu/Alumina Light Weight Composites, J. Ceram. Soc. Japan, **106**, 815(1997). (in Japanese)
- (7) K. Sodeyama, Y. Sakka, Y. Kamino, H. Seki, K. Nishimoto, M. Yazaki: The Manufacturing Process of the Fine Shirasuballoons Using a Fluidized Bed Furnace, J. Soc. Powder Tech. Japan, **34**, 697 (1997).
- (8) K. Ozawa, Y. Sakka, T. Uchikoshi, K. Hiraga: Preparation of ZrO_2 Powders Doped with SiO_2 by Hydrolysis of Tetraethoxysilane, J. Ceram. Soc. Japan, **106**, 234 (1998).
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- (10) Y. Sakka, T. Uchikoshi, K. Ozawa, K. Morita, H. Yasuda, K. Hiraga: Preparation and Some Properties of SiO_2 -doped Tetragonal Zirconia, Proc. US-JAPAN Workshop on Electrically Active Ceramic Interfaces, 97 (1998).
- (11) K. Ozawa, Y. Sakka, M. Amano: Preparation and Electrical Properties of Three Types of Antimonic Acid Films, J. Mater. Res., **13**, 830 (1998).
- (12) T. Uchikoshi, Y. Sakka, K. Ozawa, K. Hiraga: Processing of Fine-Grained Monoclinic Zirconia Ceramics by Colloidal Processing, J. Mater. Res., **13**, 840 (1998).
- (13) T. Uchikoshi, Y. Sakka, K. Ozawa, K. Hiraga: Pressure Filtration and Sintering of

- Fine Zirconia Powder, J. Europ. Ceram. Soc., **18**, 669 (1998).
- (14) T. S. Suzuki, Y. Sakka, K. Nakano, K. Hiraga: The Effect of Ultrasonication on the Colloidal Dispersion of Al_2O_3 and ZrO_2 in pH Controlled Suspension, Mater. Trans., JIM, **39**, 689 (1998).
 - (15) T. Uchikoshi, Y. Sakka, K. Ozawa, K. Hiraga: Fabrication of Nanostructured Monoclinic Zirconia Ceramics by Colloidal Processing, Mater. Res. Soc. Symp. Proc., **502**, 103 (1998).
 - (16) Y. Sakka, T. Uchikoshi, K. Ozawa, T. S. Suzuki, K. Hiraga: Preparation of Fine-Grained Zirconia Systems by Colloidal Processing., Ceram. Trans., **83**, 233 (1998).
 - (17) Y. Sakka, K. Sodeyama: Preparation of Shirasu/Alumina Lightweight Composites through a Colloidal Processing, Ceram. Trans., **83**, 283 (1998).
 - (18) Y. Sakka, T.S. Suzuki, K. Nakano, K. Hiraga: Microstructure Control and Superplastic Property of Zirconia Dispersed Alumina Ceramics, J. Japan Soc. Powder Powder Met., **45**, 1186(1998). (in Japanese)
 - (19) T. Ishigaki, T. Sato, J. Tanaka, Y. Sakka, Y. Moriyoshi: Application of Titanium Carbide Powders Treated in RF Induction Plasma Al_2O_3 -TiC Composite Ceramics, J. Japan Inst. Metal., **63**, 82(1999). (in Japanese)
 - (20) T.S. Suzuki, Y. Sakka, K. Nakano, K. Hiraga: Superplastic Tensile Ductility in a Zirconia-Dispersed Alumina Produced by Colloidal Processing, Mater. Sci. Forum, **304-306**, 489(1999).
 - (21) M. Eguchi, K. Ozawa, Y. Sakka, T. Kishi: Electrochemical Property of $(\text{H}_3\text{N}(\text{CH}_2)_3\text{NH}_3)[\text{V}_4\text{O}_{10}]$ as a Lithium Insertion Host, J. Japan Soc. Powder Powder Met. **46**, 305 (1999).
 - (22) K. Ozawa, Y. Sakka, M. Amano: Preparation and Electrical Conductivity of Bismuth-doped Antimonic Acids, Mater. Res. Soc. Symp. Proc., (1999) in press.
 - (23) K. Sodeyama, Y. Sakka, Y. Kamino, H. Seki: Preparation of Fine Expanded Perlite, J. Mater. Sci., **34**, 2461 (1999).
 - (24) Y. Sakka, T. Uchikoshi, K. Ozawa, K. Hiraga: Preparation and Some Properties of SiO_2 -doped ZrO_2 , Adv. Sci. Tech., **14B**, 81 (1999).
 - (25) Y. Sakka, T. Uchikoshi, K. Ozawa, T.S. Suzuki, K. Hiraga: Preparation of Fine-Grained zirconia and Alumina Ceramics Systems through Colloidal Processing, Adv. Sci. Tech., **14B**, 593 (1999).
 - (26) Y. Sakka, K. Hiraga: Preparation Methods and Superplastic Properties of Fine-Grained Zirconia and Alumina Based Ceramics, J. Chem. Soc. Japan, **1999**, 493 (1999). (in Japanese)
 - (27) T.S. Suzuki, Y. Sakka, K. Nakano, K. Hiraga: Colloidal Processing and

Superplasticity of Monoclinic-, Tetragonal-, and Cubic-Zirconia Dispersed Aluminas, Trans. Mater. Res. Soc. Japan, (1999) in press.

- (28) S. Akutsu, Y. Sakka, T.S. Suzuki, K. Hiraga, S. Itou: Colloidal Processing and Some Properties of Alumina Dispersed Tetragonal Zirconia, Trans. Mater. Res. Soc. Japan, (1999) in press.
- (29) T. Uchikoshi, Y. Sakka, K. Hiraga: Effect of Silica Doping on the Electrical Conductivity of 3 mol% Yttria-Stabilized Tetragonal Zirconia Prepared by Colloidal Processing, Journal of Electroceramics, in press.
- (30) T. S. Suzuki, Y. Sakka, K. Nakano, K. Hiraga: The Effect of Ultrasonication on the Colloidal Dispersion of Al_2O_3 and ZrO_2 Powders by Adsorption of Polyelectrolyte, submitted to J. Am. Ceram. Soc.
- (31) T.S. Suzuki, Y. Sakka, K. Nakano, K. Hiraga: Fabrication of Superplastic Zirconia-Dispersed Alumina Through Colloidal Processing, submitted to Mater. Trans. JIM.
- (32) H. Okuyama, K. Honma, S. Ohno: Photocatalytic Activity of Ultrafine Particles Synthesized by an RF Plasma CVD, J. Japan Inst. Met., **63**, 74 (1999). (in Japanese)

Oral Presentation: 49

- (1) International Conference: 15
- (2) Domestic Conference: 34

Patent Application:

- (1) Al_2O_3 Superplastic Ceramics and Making Process thereof: Y. Sakka, T.S. Suzuki, K. Nakano, K. Hiraga, August 25, 1997, Japanese Patent Application, H9-227965.
- (2) High-Proton-Conductive Antimonic Acid Film and Method of Manufacturing Same: K. Ozawa, Y. Sakka, T. Uchikoshi, and M. Amano, Sept. 26, 1997, US Patent Application, 08/938,521
- (3) Monoclinic Dense Zirconia and Making Process thereof: T. Uchikoshi, K. Ozawa, Y. Sakka, K. Hiraga, Feb. 27, 1998, Japanese Patent Application, H10-48433.
- (4) Al_2O_3 Superplastic Ceramics and Making Process thereof: Y. Sakka, T.S. Suzuki, K. Nakano, K. Hiraga, March 25, 1998, US Patent S.N., 09/047, 213.
- (5) Bismuth-Doped Antimonic Acid: K. Ozawa, Y. Sakka, T. Uchikoshi, M. Amano, July 2, 1999, Japanese Patent 2945975 (Application H10-278691).

Award:

- (1) Award for Improvement of Basic Research on Powder and Powder Metallurgy, Japan

Society of Powder and Powder Metallurgy (May 1998), Y. Sakka, T.S. Suzuki,
K. Hiraga

(2) Excellent Poster Award, Mater. Res. Soc. Japan (Dec. 1998), T. S. Suzuki

1.5.5 4th Laboratory

Tasuku Dendo (Head), Tohru Shiota, Shigeo Yamamoto, Takayoshi Kasugai,
Kazuo Ei

1.5.5.1 Research Work

- (1) Plastic deformation processes under semi-molten state, in which solid and liquid phases coexist, have been attempted so as to explore new controlling methods of microstructure and/or texture in the formed materials.
- (2) The joinability of the diffusion bonding between stainless steel and metals of group IV~VI has been studied in connection with the periodic table. In this study, the formation of metallic compounds and the diffusing process in the bonding zone of these metals have been investigated.
- (3) The effects of segregation at the grain boundary and micro structures on the corrosion resistance of stainless steel have been studied in the welded zone by high heat input TIG arc welding, and in the rapid solidification layer by a high power CO₂ laser.
- (4) Machinability of Ti oxide dispersed steels has been studied from a metallurgical perspective in order to develop recyclable free cutting steels in stead of lead containing steels.

<Research themes>

- (1) Micro Structure Control with Plastic Deformation under Mushy State
(General Research: April 1996 to March 1999, Tasuku Dendo)
- (2) Diffusion Bonding of Stainless Steel and Metals of Group IV~VI
(General Research: April 1996 to March 1999, Takayoshi Kasugai)
- (3) Effect of Aging Degradation on Localized Corrosion of Structural Materials for Light Water Reactors
(Nuclear Energy Research: April 1996 to March 2000, Yasuyuki Katada)
- (4) Manufacturing Process of Recyclable Pb-free Cutting Steel
(Pollution Prevention Research: April 1996 to March 2001, Akira Sato)

1.5.5.2 Research Products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) T. Shirota, T.Dendo, K. Hashimoto, H. Dohi, T. Kimura, M. Nakamura: Synthesis of Ti-Al Intermetallic Compounds by Pressure Infiltration, J. Japan Society for Technology of Plasticity 38,75(1997) (in Japanese)
- (2) T. Dendo, T. Shirota, B. Han, T. Kimura: An Attempt for Fabrication of Clad Parts through Semi-Molten Processing, 5th Inter. Conf. on Semi-Solid Processing of Alloys and Composites, Golden Colorado, June 23-25, 1998
- (3) K. Ei, H. Irie, T. Kasugai: Corrosion Property for Solid State Bonded Joint of SUS304L Stainless Steel and Zr, Tetsu-to Hagane, 85,405(1999) (in Japanese)

• Oral Presentation: 12

- (1) International Conference: 1
- (2) Domestic Conference: 11

Patent Application :

- (1) Process for Fabricating Partially Composite Metal thereof: T. Dendo, T. Shirota, B. Han, February 25, 1998, No.44020(Heisei 10)
- (2) Cladding Process of Metal thereof: T. Dendo, T. Shirota, B. Han, February 25, 1998, No.44021(Heisei 10)
- (3) Pressuring Method of Molten or Semi-molten Metal thereof: T. Dendo, T. Shirota, B. Han, February 25, 1998, No.44022(Heisei 10)

1.5.5.3 Researchers in the Processing Materials Division

Age and research fields of researchers in the Processing Materials Division

Age of Researchers	Number of Researchers	Crystal Growth	Powder Metallurgy	Casting	Composite Materials
61 ~	0	0	0	0	0
51 ~ 60	15	4	2	6	4
41 ~ 50	4	0	2	0	1
30 ~ 40	15	1	3	0	3
Summation	26	5	7	6	8

1.5.5.4 Research Budgets in the Materials Processing Division

Research Budgets in the Materials Processing Division (yen)

Kinds of budgets	1997	1998
General Research	16,104,000	15,750,000
Special Coordination Funds for Promoting Science and Technology	0	26,165,000
New Energy and Industrial Technology Developing Organization (NEDO)	0	20,380,000
Summation	16,104,000	62,295,000

Chapter 2 Research for Materials Development

5 Research Groups, numbered from 1st to 5th, are conducting R&D on the specified materials following the 5th Long-term Plan of the NRIM. They are the 1st Research Group (Superconducting Materials), the 2nd Research Group (Advance Nuclear Materials), the 3rd Research Group (Advanced High Temperature Materials), the 4th Research Group (Energy Conversion Materials) and the 5th Research Group (Intelligent Materials). All of the research subjects of the Groups are expected to bring a new breakthrough in the field of materials science and to become the forefront of the next generation of technology. Each Group is organized to perform intensive and effective work under the leadership of a Supervising Researcher during the research term. Each Group will be reviewed after the beginning of its own 5-year project, depending on the results of the research and the social demands at that time.

2.1 1st Research Group (Superconducting Materials)

Kazumasa Togano (Supervising Researcher)

2.1.1 Research work

The application of superconducting materials will have an impact on the economy, environmental control, transportation, electric power technology, medicine, communications and other areas of fundamental research. The importance of R & D of the new superconducting materials, such as oxide high temperature superconductors, is stressed in the 5th Long-term Plan of the NRIM.

The target of the 1st Research Group is to establish the basic technologies for the application of high temperature superconductors (HTSC). The Group is developing various processes to synthesize high quality single crystals, wires and films of HTSC. The relation between the structure and electromagnetic properties of HTSC is also studied to understand the vortex state and pinning mechanism of HTSC. The information obtained here is fed back to the processes to optimize their parameters. The Group consists of 5 Subgroups and the following subjects are being studied.

(1) Understanding Electromagnetic Phenomena

As fundamental research for the improvement of characteristics, clarification of electromagnetic phenomena, such as the behaviors of magnetic flux in HTSC, is being conducted by using high quality single crystals. The newly discovered Josephson Plasma phenomena are also being studied for future application to new-type electronic devices.

(2) Development of the Processes for HTSC Wires

The development of the wire making process of HTSC, such as Bi-Sr-Ca-Cu-O originally developed in the NRIM, and trial production of coils is being conducted. Comprehensive work on the microstructure and phase changes during processing are being carried out to achieve large improvements of critical current density.

(3) Structure Control by Thin Film Synthesis

The structure control and growth mechanism of HTSC thin films are being studied by using various deposition techniques, such as alternative sputtering technique. The technology of atomic layer control established in this work will be applied to the fabrication of high quality devices.

(4) Microstructure Control by Vapor Deposition Techniques

Studies on the texture control of YBaCuO film on metallic substrates are being conducted to overcome the weak link problem of this material. The Group has succeeded to develop its own technique of modified bias sputtering (MBS) which enables the in-plane alignments of the YSZ buffer layer and the YBCO film.

The members of the 1st Research Group, as at March 31, 1999, consisted of 17 researchers, 2 STA fellows, 1 domestic research fellow, 3 joint-doctoral course students, 1 secretary and 7 guest researchers.

The number of publications, including submitted papers, are about 97, patents submitted are 5, between April 1, 1977 to March 31, 1999. The details are shown in the research activities of each Subgroup.

Most of the funding for the R & D of HTSC in the 1st Research Group is provided from the Multi-core Research Project on Superconducting Materials which was set up by the Science and Technology Agency in 1988.

2.1.2 1st Subgroup

Kazuto Hirata (Subgroup Leader), Hiroyuki Takeya, Takashi Mochiku

2.1.2.1 Research work

- (1) To understand the mechanism of superconductivity in high T_c superconductors (HTSCs) and to make clear the superconducting states of HTSCs in an electromagnetic field, a phenomenological approach has been made to establish a theory based on the experimental results.
- (2) Pinning mechanism of vortices in HTSCs has been studied on high-quality single

crystal of HTSCs to improve the critical current at high temperatures and high magnetic fields.

- (3) The intrinsic Josephson effect has been studied on single crystalline HTSCs to find a new function for fabricating micro devices.
- (4) To improve critical currents in HTSCs, computer simulation study has been made to calculate the physical parameters by incorporating hypothetical and artificial pinning centers.
- (5) Co-existence of magnetism and superconductivity has been studied on single crystalline $\text{ReNi}_2\text{B}_2\text{C}$ (Re = rare earth elements).
- (6) Growth of single crystalline HTSCs has been studied, and the growth under microgravity assumed.

<Research themes>

- (1) Understanding of the Mechanism of Superconductivity in HTSCs.
(Multi-core project: April 1995 to March 2002, Kazuto Hirata)
- (2) Study on the Pinning Mechanism of Vortices in HTSCs.
(Multi-core project: April 1995 to March 2002, Kazuto Hirata)
- (3) Fabrication of Intrinsic Josephson Junctions and Evaluation of their Physical Properties
(Multi-core project: April 1995 to March 2002, Kazuto Hirata)
- (4) Computer Simulation Study on the Mechanism of HTSCs.
(Joint Research Promotion System on Computational Science and Technology: April 1997 to March 1999, Kazuto Hirata)
- (5) Coexistence of Magnetism and Superconductivity in Single Crystal of Boro-carbide System
(April 1997 to March 1998, Hiroyuki Takeya)
- (6) Single Crystal Growth of HTSCs under Microgravity
(Japan Space Forum, April 1997 to March 2000, Kazuto Hirata)

2.1.2.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) T. Mochiku: Preparation of Single Crystal, *Bismuth-Based High-Temperature Superconductors*, eds. H. Maeda and K. Togano (Marcel Dekker, New York, 1997), 227(1997).
- (2) Y. Matsuda, M.B. Gaifullin, K. Kumagai, M. Kosugi, K. Hirata: Interlayer Phase Coherence in the Vortex State of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ Proved by Josephson Plasma Resonance, *Phys. Rev. Letters* **78**, 1972(1997).
- (3) T. Mochiku, K. Hirata, K. Kadowaki: Crystallinity Improvement of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ Single Crystal by TSFZ Method, *Physica C* **282-287**, 475(1997).

- (4) Y. Matsuda, M.B. Gaifullin, K. Kumagai, K. Kadowaki, T. Mochiku, K. Hirata: Excitation of Josephson Plasma and Vortex Oscillation Modes in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ in Parallel Magnetic Fields, *Phys. Rev. B* **55**, R8685(1997).
- (5) W. Prusseit, M. Rapp, K. Hirata, T. Mochiku: Intrinsic Josephson Junctions under Microwave Irradiation, *Physica C* **293**, 25(1997).
- (6) Y. Matsuda, M.B. Gaifullin, M. Kosugi, K. Kumagai, K. Hirata: Excitation of Josephson Plasmon in the Vortex States of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$, *Physica C* **293**, 13(1997).
- (7) S.Takahashi, M.Tachiki, I.Ikeya, K.Kindo, T.Mochiku, K.Kadowaki: Josephson Plasma Excitation in High-Tc Superconductors with Finite Dimensions, *Physica C* **293**, 64(1997).
- (8) M.Kosugi, Y.Matsuda, M.B.Gaifullin, K.Kumagai, N.Chikumoto, J.Shimoyama, K.Kishio, K.Hirata, M.Konczykowski: Interlayer Josephson Coupling of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ with Columnar Defects Probed by Plasma Resonance, *Physica C* **293**, 208(1997).
- (9) M.Kosugi, Y.Matsuda, M.B.Gaifullin, K.Kumagai, N.Chikumoto, J.Shimoyama, K.Kishio, K.Hirata, M.Konczykowski: Influence of Columnar Defects on Interlayer Coherence in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ from Josephson Plasma Resonance, *Physica C* **282-287**, 2073(1997).
- (10) Y.Matsuda, M.Kosugi, M.B.Gaifullin, K.Kumagai, K.Hirata, N.Chikumoto, M.Konczykowski, S.Watauchi, J.Shimoyama, K.Kishio: Interlayer Phase Coherence in the Vortex States of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ Probed by Josephson Plasma Resonance, *Physica C* **282-287**, 391(1997).
- (11) M.B.Gaifullin, Y.Matsuda, K.Kumagai, M.Kosugi, K.Kadowaki, T.Mochiku, K.Hirata: Influence of Columnar Defects on Interlayer Coherence in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ from Josephson Plasma Resonance; *Physica C* **282-287**, 2221(1997).
- (12) M.Kosugi, Y.Matsuda, M.B.Gaifullin, L.N.Bulaevskii, N.Chikumoto, M.Konczykowski, J.Shimoyama, K.Kishio, K.Hirata, K.Kumagai: Coupling Transition of the Vortex Liquid in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ with Columnar Defects, *Phys. Rev. Letters* **79**, 3763(1997).
- (13) M.Kosugi, Y.Matsuda, M.B.Gaifullin, L.N.Bulaevskii, N.Chikumoto, M.Konczykowski, J.Shimoyama, K.Kishio, K.Hirata: Josephson Coupling in the Vortex-Liquid State of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ with Columnar Defects, *Phys. Rev. B* **59**, 8970(1999).
- (14) K.Hirata, H.Takeya, S.Miyamoto, T.Mochiku, K.Togano: Peak Effect and Vortex

- Line Lattice Melting in Single Crystalline $\text{YNi}_2\text{B}_2\text{C}$, Chinese J. of Physics (Taipei), **36**, 215(1998).
- (15) T. Mochiku, M. Nakahara, E. Abe, T. Kamiyama, H. Asano, K. Hirata, F. Izumi: Crystal Structure of $\text{Sr}_{1.9}\text{Nd}_{1.1}\text{Cu}_{2.1}\text{Nb}_{0.9}\text{O}_8$ and $\text{Sr}_2(\text{Nd}_{0.75}\text{Ce}_{0.25})_2\text{Cu}_2\text{NbO}_{10}$: $\text{Ba}_2\text{YCu}_3\text{O}_{6+\delta}$ -Related Compounds with NbO_6 Octahedron, 10th Inter. Symp. on Superconductivity(ISS'97), Gifu, Oct. 27-30 (1997).
 - (16) K. Hirata, T. Mochiku, N. Nishida: Magnetization Measurements on $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ Single Crystals with Columnar Defects, 10th Inter. Symp. on Superconductivity(ISS'97), Gifu, Oct. 27-30 (1997).
 - (17) R. Sugano, T. Onogi, K. Hirata, M. Tachiki: Field-driven Coupling Transition in the Vortex State of Irradiated $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$, Computer Simulation Study, Phys. Rev. Letters **80**, 2925 (1998).
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 - (20) M. R. Norman, H. Ding, M. Randeria, J. C. Campuzano, T. Yokoya, T. Takeuchi, T. Takahashi, T. Mochiku, K. Kadowaki, P. Guptasarma, D. G. Hinks: Destruction of the Fermi Surface in Underdoped High-Tc Superconductors, Nature **392**, 157 (1998).
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 - (22) S. Kaneko, N. Nishida, T. Mochiku, K. Kadowaki, Scanning Tunneling Spectroscopy of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$, Physica C **298**, 105 (1998).
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- (32) H. Takeya, M. Kuznietz: Formation and Characterization of $(\text{Pr,Dy})\text{Ni}_2\text{B}_2\text{C}$ Samples, *Physica B* **259**, 596 (1999).
- (33) H. Takeya, M. Kuznietz: Magnetic Properties of the $(\text{Pr,Dy})\text{Ni}_2\text{B}_2\text{C}$ Solid Solutions, *J. Magn. Magn. Mater.* (1999) in press.

• **Oral Presentation:** 48

- (1) International Conference: 18
 (2) Domestic Conference: 30

2.1.3 2nd Subgroup

Hiroaki Kumakura (Subgroup Leader), Hitoshi Kitaguchi, Hiroki Fujii, Akiyoshi Matsumoto, Yeon Soo Sung (STA fellow)

2.1.3.1 Research work

- (1) $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ (Bi-2212) multilayer tape conductors were developed by applying a Pre-Annealing and Intermediate Rolling (PAIR process) before heat treatment. The PAIR processed tapes showed very excellent critical current densities (J_c) in a field of 10-30T at 4.2K. These high J_c values were attributed to the improved microstructure obtained by the PAIR process. Bi-2212 wire conductors having round cross sections were also fabricated by applying the ROtation Symmetric Arranged Tape-in-tube (ROSAT) method. The ROSAT wires show very small anisotropy with respect to field orientation, in spite of excellent J_c values at 4.2K and are promising as conductors of solenoid magnets.

- (2) Development of a prototype oxide superconducting magnet for magnetic separation system is now in progress. We fabricated Bi-2212 and $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ (Bi-2223) small magnets and tested them at various temperatures using a cryocooler. Both of the magnets showed excellent thermal and electromagnetic stabilities at temperatures above 20K, indicating that a bismuth oxide superconducting magnet is promising for magnetic separation systems.

<Research themes>

- (1) Development of High-Tc Oxide Superconducting Tapes and Wires having Excellent Performance (Multi-core Project: April 1995 to March 1999, Hiroaki Kumakura)
(2) Development of an Oxide Superconducting Magnet for Magnetic Separator (Multi-core Project: April 1995 to March 1999, Hiroaki Kumakura)

2.1.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) H.Kumakura, H.Kitaguchi, T.Kiyoshi, K.Inoue, K.Togano, M.Okada, K.Fukushima, K.Tanaka, K.Kato, J.Sato: Performance Tests of Bi-2212 Insert Magnets Fabricated by Ag Sheath Method and Dip-coating Method, IEEE Trans. Appl. Superconductivity 7, 646(1997).
(2) H.Fujii, H.Kumakura, H.Kitaguchi, K.Togano, W.Zhang, E.Hellstrom: The Effect of Oxygen Partial Pressure during Heat-treatment on the Microstructure of Dip-coated Bi-2212/Ag and Ag Alloy Tapes, IEEE Trans. Appl. Superconductivity 7, 1707(1997).
(3) H.Fujii, H.Kitaguchi, H.Kumakura, K.Togano: Effect of Oxygen Partial Pressure on Grain Boundaries in Bi-2212/Ag Tapes, Physica C282-287, 2567(1997).
(4) T.Hasegawa, Y.Hikichi, T.Koizumi, A.Imai, H.Kumakura, H.Kitaguchi, K.Togano: Fabrication and Properties of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ Multilayer Superconducting Tapes and Coils, , IEEE Trans. Appl. Superconductivity 7,1703(1997).
(5) M.Okada, K.Fukushima, K.Tanaka, T.Hirano, J.Sato, H.Kitaguchi, H.Kumakura, K. Togano, H.Wada: Superconducting Joints of Ag Sheathed $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ Tapes. J. Jpn. Institute of Metals 61,849(1997). (in Japanese)
(6) H.Fujii, H.Kumakura, K.Togano: Grain Boundary Structure of Bi-2212/Ag Tapes Prepared under Various Oxygen Atmospheres, J. Jpn. Institute of Metals 61, 856(1997). (in Japanese)
(7) T.Hasegawa, Y.Hikichi, T.Koizumi, A.Imai, H.Kumakura, H.Kitaguchi, K.Togano: Fabrication and Characterization of Ag-Mg Sheathed $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_y$ Multilayer Superconducting Tapes and Coils, Adv. Superconductivity IX, 871(1997).
(8) M.Okada, K.Tanaka, K.Fukushima, J.Sato, H.Kitaguchi, H.Kumakura, T.Kiyoshi, K. Inoue, K.Togano: Development of Bi-2212/Ag Superconducting Insert Magnets

- for High Magnetic Field Generation, Adv. Superconductivity IX, 977(1997).
- (9) T.Muroga, J.Sato, S.Kuma, H.Kitaguchi, H.Kumakura, K.Togano, M.Okada: Improvements in Coil Performance of Dip-coated Bi-2212 Tapes, Adv. Superconductivity IX, 981(1997).
 - (10) K.Fukushima, M.Okada, J.Sato, T.Kiyoshi, H.Kumakura, K.Togano, H.Wada: Persistent Mode Operation of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x/\text{Ag}$ Stacked Double Pancake Coils with Superconducting Joints, Jpn. J. Appl. Phys. 36, L1433(1997).
 - (11) H.Maeda, K.Kakimoto, M.Kikuchi, J.O.Willis, K.Watanabe, Y.Tanaka, H. Kumakura: Microstructure and Superconducting Properties of V Doped Bi-2223 Tapes, Appl. Superconductivity 5, 151(1997).
 - (12) H.Kitaguchi, H.Kumakura, K.Togano, M.Okada, K.Tanaka, K.Fukushima, K.Nomura, J.Sato: "Fabrication and Transport Properties of Bi-2212/Ag Multifilamentary Coils for High Field Generation (II); High Field Performance Test, 3rd European Conf. on Applied Superconductivity, The Netherlands June 30-July 3, 1997
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 - (14) H.Kitaguchi, H.Kumakura, K.Togano, M.Okada, K.Tanaka, J.Sato: High Field Performance Tests for Bi-2212 Magnets with 30mm Bore, Cryogenics 38, 181(1998).
 - (15) H.Miao, H.Kitaguchi, H.Kumakura, K.Togano: Fabrication of High Current Carrying Bi-2212/Ag Superconducting Tapes by the Pre-annealing and Cold Rolling Process, Cryogenics 38, 257(1998).
 - (16) H.Miao, H.Kitaguchi, H.Kumakura, K.Togano: Microstructure and Superconducting Properties of Pb-substituted $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x/\text{Ag}$ Dip Coated Tapes, Physica C298, 312(1998).
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 - (18) T.Hasegawa, Y.Hikichi, Y.Aoki, H.Kumakura, H.Kitaguchi, K.Togano: Effects of Microstructural Improvement on the Superconducting Properties of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ Multilayer Tapes, Adv. Superconductivity X, 759(1998).
 - (19) H.Miao, H.Kitaguchi, H.Kumakura, K.Togano, T.Hasegawa: Effects of PAIR(Pre-annealing and Intermediate Rolling) Process on Superconducting Properties of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x/\text{Ag}$ Multilayer Tapes, Physica C301, 116(1998).
 - (20) H.Miao, H.Kitaguchi, H.Kumakura, K.Togano: Effect of Starting Materials on Microstructure and Property of Bi-2212/Ag Tapes, Adv. Superconductivity X, 767(1998).

- (21) Y.Hishinuma, H.Kumakura, H.Kitaguchi, K.Togano: Effect of Cu Concentration on the Microstructure and Superconducting Properties of Bi-2212/Ag Tapes, *Adv. Superconductivity X*, 849(1998).
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- (23) M.Okada, K.Tanaka, S.Matsuda, K.Sato, J.Sato, H.Kitaguchi, T.Kiyoshi, H. Kumakura, K.Togano, H.Wada: Bi-2212/Ag Superconducting Magnet for High-field Applications, *Adv. Superconductivity X*, 1381(1998).
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- (25) H.Miao, H.Kitaguchi, H.Kumakura, K.Togano, T.Hasegawa, T.Koizumi Bi-2212/Ag Multilayer Tapes with J_c 500,000A/cm² at 4.2K and 10T by Using Pre-annealing and Intermediate Rolling Process, *Physica C* **303**, 81(1998).
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- (31) H.Fujii, H.Kumakura and K.Togano: Structure and Misorientation Angle Distributions of (00l) Twist Grain Boundaries in Bi-2212/Ag Composite Tapes Processed in Different Oxygen Partial Pressures, *J. Materials Res.* **14**,349(1998).
- (32) H.Kitaguchi, H.Miao, H.Kumakura, K.Togano, T. Hasegawa, K. Koizumi: Fabrication of Bi₂Sr₂CaCu₂O_x/Ag Tapes with $J_c(4.2K, 10T) > 300,000A/cm^2$ by Using PAIR Process, 7th Inter. Cryogenic Engineering Conf., Bouremouth, UK July 14-17, 1998
- (33) T.Muroga, J.Sato, H.Kitaguchi, H.Kumakura, K.Togano, M.Okada: Enhancement of Critical Current Density for Bi-2212/Ag Tape Conductors

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- (34) H.Kitaguchi, H.Miao, H.Kumakura, K.Togano, T.Hasegawa, T.Koizumi: Large Jc Enhancement of Bi-2212/Ag Tape by PAIR Process, Adv. Superconductivity XI, 811(1999).
 - (35) H.Maeda, W.P.Chen, K.Kakimoto, P.X.Zhang, K.Watanabe, M.Motokawa, H. Kitaguchi, H.Kumakura and K.Itoh: Microstructure and Properties of Bi-2212 Tapes and Bulks Grown in High Magnetic Field, Adv. Superconductivity XI, 823(1999).
 - (36) A.Motsumoto, H.Kumakura, K.Togano: Hg Doping Effects on Synthesis, Microstructure and Superconducting Properties of the Bi-2212 Phase, Physica C319,34(1999).
 - (37) K.Ohata, J.Sato, M.Okada, K.Tanaka, H. Kitaguchi, H. Kumakura, T. Kiyoshi, H.Wada, K.Togano: Development of a New Type Bi-2212 Multi-filamentary Wire, Adv. Superconductivity XI, 871(1999).
 - (38) H.Miao, H.Kitaguchi, H.Kumakura, K.Togano, T.Hasegawa, T. Koizumi: Effects of Starting Composition and Treating Atmosphere on Phase Formation, Microstructure and Jc of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x/\text{Ag}$ Multilayer Tapes, Adv. Superconductivity XI, 895(1999).
 - (39) H.Miao, H.Kitaguchi, H.Kumakura, K.Togano, T.Hasegawa, T. Koizumi: Low Temperature Annealing Effects on Superconducting Properties of Melt-solidified $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x/\text{Ag}$ Multilayer Tapes, Adv. Superconductivity XI, 899(1999).
 - (40) T.Koizumi, T.Hasegawa, H.Kitaguchi, H.Miao, H.Kumakura, K.Togano: Processing Related Issues on High Jc Bi-2212/Ag Multilayer Tapes Prepared by PAIR Process, Adv. Superconductivity XI, 919(1999).
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 - (43) K.Noto, Y.Kimura, A.Nagata, M.Iwakuma, H.Kumakura, S.Kosaka, T.Kaneko, N.Sadakata, A.Kimura, Y.Nagai: Results of RRT on Bi(2223)/Ag Tape Samples and Thermal Properties of High Tc Superconductors, Adv. Superconductivity XI, 1493(1999).
 - (44) H.Kumakura: "Fabrication of High-Tc Oxide Superconducting Tapes and Wires" in *High Temperature Superconductors and Exotic Superconductors*, ed. By S. Tanuma and Y. Iye, (Kyo-ritu Tokyo 1999)
 - (45) H.Kumakura, H.Kitaguchi, K.Togano, T.Muroga, J.Sato and M.Okada: The Influence of Ag Substrate on the Grain Alignment and Critical Current Density of Bi-2212 Tape Conductors, IEEE Trans. Appl. Superconductivity(accepted)

- (46) H.Kitaguchi, H.Kumakura, K.Togano, H.Miao, T.Hasegawa, T.Koizumi: Bi₂Sr₂CaCu₂Ox/Ag Multilayer Tapes with Jc(4.2K, 10T) of 500,000A/cm² by Using PAIR Process, IEEE Trans. Appl. Superconductivity(accepted)
- (47) T.Hishinuma, H.Kitaguchi, H.Kumakura, K.Itoh, K.Togano, H.Miao, B. Chenevier: Local Jc Distribution in Superconducting Oxide Layer of Bi-2212/Ag Tapes, IEEE Trans. Appl. Superconductivity(accepted).
- (48) H.Fujii, H.Kumakura, K.Togano: Properties of (Hg,X)-1223 Superconductors Synthesized under Ambient Conditions, IEEE Trans. Appl. Superconductivity (accepted)
- (49) M.Okada, K.Tanaka, T.Wakuda, K.Ohata, J.Sato, H.Kumakura, T.Kiyoshi, H. Kitaguchi, K.Togano, H.Wada: A New Symmetrical Arrangement of Tape-Shaped Multifilaments for Bi-2212/Ag Round-Shaped Wire, IEEE Trans. Appl. Superconductivity (accepted)
- (50) M.Okada, K.Tanaka, T.Wakuda, K.Ohata, J.Sato, H.Kumakura, T.Kiyoshi, H. Kitaguchi, K.Togano, H.Wada: Fabrication of Bi-2212/Ag Magnets for High Magnetic Field Applications, IEEE Trans. Appl. Superconductivity(accepted)

Oral presentations: 73

- (1) International Conference: 33
- (2) Domestic Conference: 40

Patent Applications :

- (1) Method of Bi-2212 Superconductor Fabrication: H. Kitaguchi, H. Miao, H. Kumakura, K. Togano, 1997
- (2) Fabrication Method of Oxide Superconducting Composites: K. Togano, H. Kumakura, H. Kitaguchi, T. Muroga, May 19, 1998, Application Number 10-136441
- (3) Method of Oxide Superconductor Fabrication: K. Togano, H. Kumakura, H. Kitaguchi, T. Muroga, May 19, 1998, Application Number 10-136442

Award:

Award of Superconductor Science and Technology: The Society of Non-Traditional Technology: April, 1998, H. Kumakura and K. Togano,.

2.1.4 3rd Subgroup

Takeshi Hatano (Subgroup Leader), Akira Ishii, Shunichi Arisawa, Yoshihiko Takano, Stephane Labat (STA-fellow), Keikichi Nakamura (guest researcher)

2.1.4.1 Research work

- (1) New synthesis route to grow $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ (2212) ribbon-shaped films has been developed. The ribbon-shaped films have been grown under ambient pressure from 2212 powder material on a silver substrate by the so called “partial melt and slow cooling” heat treatment procedure. The films have both better surface morphology and T_c s.
- (2) In order to keep a better surface morphology, thin films of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ (2212) were deposited by pulsed laser ablation onto $\text{MgO}(100)$ single crystal substrates at temperature about 50-100K lower than the decomposition point of the 2212 phase. The films were accurately post-annealed in the atmosphere. The T_c of the annealed films reached 75-80K retaining their the surface smoothness. We found that the film deposited at lower substrate temperature showed only the $2212[100] \parallel \text{MgO}[100]$ epitaxial relation. After annealing, the *in-plane* direction completely rotated by 45° , and only the $2212[110] \parallel \text{MgO}[100]$ epitaxial relation became observable.
- (3) Atomically layer-by-layer deposition: Superlattice films of bismuth-based oxide superconductors, for instance $\text{Bi}_2\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_{12}/\text{Bi}_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_8$ and $\text{Bi}_2\text{Sr}_2\text{Ca}_4\text{Cu}_5\text{O}_{14}/\text{Bi}_2\text{Sr}_2\text{CuO}_6$, have been sequentially synthesized. It was observed that the superconducting transition temperatures were enhanced by forming superlattice films of over doped ($\text{Bi}_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_8$, $\text{Bi}_2\text{Sr}_2\text{CuO}_6$) and under doped ($\text{Bi}_2\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_{12}$, $\text{Bi}_2\text{Sr}_2\text{Ca}_4\text{Cu}_5\text{O}_{14}$) phases.

<Research theme>

- (1) Characterization and Application of Superconducting Thin Films Synthesized by Atomic Layer-by-Layer and Epitaxial Growth Methods.

(Multi-core project: April 1995 to March 2002, Takeshi Hatano)

2.1.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) T.Hatano: High- T_c Superconductors - Layer-by-Layer Growth, Metals and Technology 68, 505 (1998).
- (2) T.Hatano, S.Labat, A.Ishii, S.Arisawa, K.Togano: Stress Induced Change in Hole Concentration in Superlattice Films of Bi-based Superconductors, Applied Superconductivity Conference, Palm Springs Desert, Sept. 13-18, 1998.
- (3) T.Hatano, A.Ishii, S.Labat, S.Arisawa, K.Togano: Strain Effects in Artificially Layered BSCCO Superconductors, Trans. MRS-J 24 (1999)
- (4) A.Ishii, T.Hatano, K.Nakamura, Superconducting Properties and *in-plane* Orientation of $\text{Bi}_2\text{Sr}_2\text{Ca}_{(n-1)}\text{Cu}_n\text{O}_y$ Thin Films Synthesized by Pulsed Laser Ablation, J. Japan Inst. Metals 61, 879 (1997). (in Japanese)

- (5) S.Arisawa, T.Hatano, K.Nakamura, K.Togano: Effect of the Substrate of *in-situ* Fabrication of Borocarbide Thin Films, *Physica C*, **308**, 67(1998).
- (6) S.Arisawa, T.Hatano, K.Togano: Fabrication and Characterization of Borocarbide Thin Films Grown by in-situ Process, *J. Korean Physical Society*. (in press.)
- (7) S.Arisawa, H.Miao, H.Fujii, A.Ishii, S.Labat, T.Hatano, K.Togano: Preparation and Superconducting Properties of Extremely Thin $\text{Bi}_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_x$ Ribbon-like Thin Films on Silver Substrates, *Physica C* **314**, 155(1999).

•**Oral presentations:**13

- (1) International Conference: 5
- (2) Domestic Conference: 8

•**Patent application:**

- (1) Fabrication Method of Bi-based High- T_c Superconducting Thin Films: A.Ishii, T.Hatano, K.Nakamura, Japan, Sept. 19, 1997, No. 255623, 1997.
- (2) Fabrication Method of Ribbon Like Thin Films of High T_c Superconductors: S.Arisawa, T.Hatano, K.Togano, H.Miao, Japan, Feb. 25, 1999, No. 49114, 1999.

•**Award(s):**

Young Researcher Award: The Japan Institute of Metals, Sep. 1998, S. Arisawa

2.1.5 4th Subgroup

Masao Fukutomi (Subgroup Leader), Kazunori Komori, Kyouko Kawagishi

2.1.5.1 Research work

Studies on high temperature superconducting (HTS) thin films for microwave applications have been carried out using a laser ablation technique and rf magnetron sputtering. To fabricate microwave quality HTS films with a good in-plane epitaxy, suitable in-plane oriented buffer layers have been developed using plasma and ion beam techniques. A sapphire rod resonator has been used to evaluate the microwave surface resistance, R_s , of the films. In order to establish this method as the standard for R_s measurements for HTS films, research into the R_s measurement methods is also conducted as a member of the domestic “round-robin test” committee. Part of this research was performed in collaboration with the High Energy Accelerator Research Organization and MITSUBA Corporation.

<Research themes>

- (1) Control of the In-plane Texture of High- T_c Superconducting Thin Films for Microwave Applications(General Research: April 1998 to March 2001, Masao Fukutomi)
- (2) Texture Controll of $\text{YBa}_2\text{Cu}_3\text{O}_y$ Thin Films by Vapor Deposition Techniques (research subject of “Characterization and Application of Superconducting Thin Films Synthesized by Atomic Layer-by-Layer and Epitaxial Growth Methods”)(R&D on New Superconducting Materials: April 1995 to March 2001, Masao Fukutomi)

2.1.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) M.Fukutomi,S.Kumagai,H.Maeda: Fabrication of $\text{YBa}_2\text{Cu}_3\text{O}_y$ Thin Films on Textured Buffer Layers Grown by Plasma Beam Assisted Deposition, Australian J. Physics 50, 381 (1997)
- (2) J.Liu, K.Asano, E.Ezura, S.Inagaki, S.Isagawa, H.Nakanishi, M.Fukutomi, K.Komori, M.Saitoh: Dependence on the Microwave Field of the Surface Resistance for $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ Films Fabricated on Copper Substrates, Appl. Phys.Lett. 73, 3450 (1998)
- (3) S.Kumagai, S.Aoki, M.Saitoh, M.Fukutomi, K.Komori, K.Togano: Fabrication of $\text{YBa}_2\text{Cu}_3\text{O}_y$ Thin Films on Textured Buffer Layers Grown by Modified Bias Sputtering Technique, Proc. 2nd Japanese Romanian Joint Seminar on Applied Electromagnetics and Mechanics, June,1998.

•Oral Presentation: 8

- (1) International Conference: 1
- (2) Domestic Conference:7

2.1.6 5th Subgroup

Yoshiaki Tanaka (Subgroup Leader), Tsuneo Kuroda, Y.C.Guo (STA fellow)

2.1.6.1 Research work

Fabrication processes for the Bi-system oxide superconductors by the powder-in-tube technique have been studied with emphasis on high mechanical strength and high critical current density properties.

<Research themes>

- (1) Fabrication of Ag-Cu Alloy Sheathed Oxide Superconducting Wires with High Strength and High J_c Properties (General Research: April 1998 to March 2000, Yoshiaki Tanaka)
- (2) Improvement of Electrical and Mechanical Properties in High Temperature Superconductors (STA Fellow Research: May 1997 to July 1998, Y.C.Guo)

2.1.6.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) Y.Tanaka, T.Kuroda, K.Miura, Y.Abe, Y.Suga, M.Ishizuka, T.Yanagiya, S.Yasuhara: Bi-2212 Ag-Sheathed Round Cross Section Wires, Proc. Inter. Symp. on Superconductivity(ISS'97), Gifu, Japan, October 27-30, 1997.
- (2) Y.C.Guo, Y.Tanaka, T.Kuroda, S.X.Dou, Z.Q.Yang: Addition of Nanometer SiC in the Silver-sheathed Bi2223 Superconducting Tapes, Physica C, **311**, 65 (1999).
- (3) Y.C.Guo, J.X.Jin, H.K.Liu, Y.Tanaka, S.X.Dou: Long Lengths of Silver-Clad Bi2223 Superconducting Tapes with High Current Carrying Capacity, Appl. Superconductivity **5**, 163 (1997).
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·Oral Presentation: 11

- (1) International Conference: 3
- (2) Domestic Conference: 7

This project will be performed in collaboration with Sumitomo Heavy Industries, Ltd, Kanagawa and Sukegawa Electric Co. Ltd, Ibaraki.

2.1.7 Researchers in the 1st Research Group

Age and research fields of researchers in the 1st Research Group. (March 31, 1999)

Age of Researchers	Number of Researchers	Materials Science	Physics	Chemistry	Electronics
60 ~	2	0	1	1	0
50 ~ 60	3	2	1	0	0
40 ~ 49	5	4	1	0	0
30 ~ 39	8	6	1	1	0
22 ~ 29	6	5	0	0	1
Summation	24	17	4	2	1

Students and visitors from universities and industry are not included.

2.1.8 Research Budgets in the 1st Research Group

Research budgets in the 1st Research Group (yen)

Kinds of Budget	1997	1998
Core Research for Evolutional Science and Technology	91,000,000	140,690,000
1 st SG	5,000,000	5,000,000
Multi-Core project(Mechanism)	22,983,000	22,889,000
Multi-Core project (Vortices)	12,645,000	12,351,000
Multi-Core project(Junction)		
Joint Research Promotion System	15,483,000	11,500,000
Special Coordination Funds for Promoting Science and Technology	9,000,000	
NASDA: Microgravity	5,400,000	5,400,000
2 nd SG	38,460,000	25,795,000
Multi-Core project(Wire)	29,091,000	43,046,000
Multi-Core project(Magnetic Separation)		
Special Coordination Funds for Promoting Science and Technology	5,000,000	
3 rd ,4 th SG	12,695,000	24,195,000
Multi-Core project(Thin film)		7,150,000
General Research		9,000,000
Supplementary Budget		
5 th SG	3,000,000	1,900,000
General Research		
Summation	249,757,000	308,916,000

2.2 2nd Research Group (Advanced Nuclear Materials)

Koichi Yagi (Supervising Researcher)

2.2.1 Research work

Researches related to nuclear technology and materials are conducted in some Research Groups, Research Divisions and Centers in the NRIM. The nuclear research subjects carried out in NRIM are totally regulated and controlled by the Nuclear Energy and Materials Research Committee. The 2nd Research Group takes charge of some specific research fields of that nuclear research.

Understanding of the interaction between materials and energetic particles, which are produced from nuclear reactions, is an important subject concerning the study on radiation damage of nuclear materials and the development of irradiation resistant materials. Because it is predicted that the materials for advanced nuclear reactors serve under severe conditions, to clarify the mechanisms of radiation damage and to establish its control principals are essential. Therefore, dynamic studies on the radiation damage of materials by energetic particles and the development of innovative irradiation resistant materials are pursued by this Research Group as follows.

- (1) Surface reaction and damage processes studied by real-time measurements
- (2) Dynamic response of materials under irradiation
- (3) Development of isotopically controlled materials
- (4) Data-free-way system for advanced materials
- (5) Understanding the chemical reactivity of molecules on solid surfaces and clusters

The members of the 2nd Research Group, as at March 31, 1999, consisted of 12 researchers, 1 domestic research fellow, 1 secretary and 7 guest researchers.

The research activities of this group are very high since the start of the 5th Long-term Plan owing to the clear purpose of this research group being very clear. The number of publications, including submitted papers, are 109, patents submitted are 7 from April 1, 1997 to March 31, 1999. The details are shown in the research activities of each Subgroup.

2.2.2 1st Subgroup

Masahiro Kitajima (Subgroup Leader), Akiko N. Itakura, Kunie Ishioka, Kouichi Murakami (guest researcher), Yasushiro Nishioka (guest researcher), Akira Miyamoto (guest researcher), Takaya Kawabe (guest researcher), Kiminori Ushida (guest researcher), Hrvoje Petek (guest researcher)

2.2.2.1 Research work

Dynamics and kinetics of chemical reaction and defect growth on material surfaces under irradiation of ions, atomic/molecular beams and light have been studied by using pump-probe reflectivity techniques (coherent phonon measurements), Raman spectroscopy, resonance enhanced multiphoton ionization (REMPI) spectroscopy, and high-sensitivity surface stress measurements.

<Research themes>

- (1) Real-Time Investigation on Surface Reactions and Defect Growth Processes under Irradiation (Nuclear Energy Research: April 1994 to March 1999, Masahiro Kitajima).
- (2) High-Resolution Real-Time Investigation on Defect Formation Processes under Surface-and Interface-reactions (Nuclear Energy Research: April 1999 to March 2004, Masahiro Kitajima).

2.2.2.2 Research products, including papers submitted (April1, 1997 to March 31, 1999)

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- (2) K.G. Nakamura, K. Ishioka, M. Kitajima, K. Murakami: AB INITIO Calculation of the Hydrogen Molecule in Silicon, Solid State Commun., **101**, 735(1997).
- (3) K.G. Nakamura, M. Kitajima: Vibration and Rotation of Hydrogen Molecule in Silicon, Jpn. J. Appl. Phys., **36**, 2004(1997).
- (4) E. Asari, K.G. Nakamura, T. Kawabe, M. Kitajima: Observation of Relaxation Processes of Disorder in Ion-irradiated Graphite Using Raman Spectroscopy, J. Nucl. Mater., **244**, 173(1997).
- (5) K. Ishioka, K.G. Nakamura, M. Kitajima, N. Fukata, S. Sasaki, K. Murakami, S. Fujimura, J. Kikuchi, H. Haneda: Raman Spectroscopic Study on Hydrogen Molecules in Crystalline Silicon Treated with Atomic Hydrogen, Appl. Surf. Sci., **117/118**, 37(1997).
- (6) K.G. Nakamura, I. Kamioka, M. Kitajima: Pulse Molecular Beam Reactive Scattering of O₂ on Semiconductor Surfaces Studied with Resonance Enhanced Multiphoton Ionization, Appl. Surf. Sci., **117/118**, 42(1997).
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- (9) H. Kimura, K. Ishioka, M. Sato: Estimation of SHG Properties on Polycrystalline $\text{Ba}(\text{B}_{1-x}\text{M}_x)_2\text{O}_4$ (M:Al or Ga), *J. Mater. Sci. Lett.*, **16**, 1375(1997).
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- (11) N. Fukata, S. Sasaki, K. Murakami, K. Ishioka, K.G. Nakamura, M. Kitajima, S. Fujimura, J. Kikuchi, H. Haneda: Hydrogen Molecules and Hydrogen-related Defects in Crystalline Silicon, *Phys. Rev.*, **B56**, 6642(1997).
- (12) K.G. Nakamura, M. Kitajima: 2D Trajectory Calculations on the Eley-Rideal Reaction, *J. Sur. Sci. Jpn.*, **18**, 700(1997). (in Japanese)
- (13) N. Fukata, S. Sasaki, K. Murakami, K. Ishioka, K.G. Nakamura, M. Kitajima, S. Fujimura, J. Kikuchi, H. Haneda: Hydrogen Molecules in Defective Silicon, *Jpn. J. Appl. Phys.*, **36**, L1456(1997).
- (14) K.G. Nakamura, K. Ishioka, M. Kitajima: Hydrogen Molecule in Group IV Element Crystal, *Jpn. J. Appl. Phys.*, **36**, L1479(1997).
- (15) A. Endou, A. Stirling, R. Yamauchi, E. Broclawik, M. Kubo, A. Miyamoto, K.G. Nakamura, M. Kitajima: Quantum Chemical Study on SiO Desorption from Si (111) Surface, *Surf. Sci.*, **387**, 59(1997).
- (16) M. Kitajima: Defects in Crystals Studied by Raman Scattering, *Critical Review in Solid State and Materials Sciences*, **22**, 275(1997).
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- (29) A.Yamada, A. Endou, H. Takaba, K. Teraishi, S.S.C. Ammal, M. Kubo, K.G. Nakamura, M. Kitajima, A. Miyamoto: Tight-binding Molecular Dynamics Simulation of Desorbed SiO Molecule during the Oxidation of Si(111) Surface, *Jpn. J. Appl. Phys.*, in press.
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- (33) K.G. Nakamura, T. Narushima, M. Kitajima: Surface Stress in Silicon Oxide Layer, *Transaction of Materials Research Society of Japan*, in press.
- (34) K. Ishioka, K. Ushida, K. Nakamura, S. Hishida, M. Kitajima: Effect of Ion Irradiation on Coherent Phonon Dynamics in Bismuth, *Jpn. J. Appl. Phys.*, part 2(Letters), in press.

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- (36) M. Kitajima, K. Ishioka, K. Nakanoya, S. Tateishi, T. Mori, N. Fukata, K. Murakami, S. Hishida: Three Different Forms of Hydrogen Molecules in Silicon, Jpn. J. Appl. Phys., 38, Part 2, No. 7A, pp. L691(1998).
- (37) K. Murakami, K. Ishioka, M. Kitajima, S. Tateishi, K. Nakanoya, T. Mori, S. Hishida: A New Type of Hydrogen Molecules in Silicon, Physica B, submitted
- (38) M. Kitajima, K. Ishioka, K. Murakami, K. Nakanoya, T.Mori: Temperature Dependence of the Hydrogen Molecules in n- and p- Type Silicon, Physica B, submitted
- (39) K. Ishioka, M. Kitajima, S. Tateishi, K. Nakanoya, N. Fukata, K. Murakami, S. Hishida: Hydrogen Molecules in Multivacancies in Silicon, Phy. Rev. Lett., submitted

Oral Presentation: 68

- (1) International Conference: 20
- (2) Domestic Conference: 48

Patent Application:

- (1) Formation Method of Ultra-thin Silicon Oxide Film Having a MOS Device Characteristics, M. Kitajima, et al. Oct. 17, 1997, 09-282413

2.2.3 2nd Subgroup

Johsei Nagakawa (Subgroup Leader), Norikazu Yamamoto, Yasumitsu Fukuzawa, Yoshiharu Murase, Shigeo Okuda (guest researcher)

2.2.3.1 Research work

- (1) Fatigue fracture of SUS 316 SS under irradiation in load-control mode was found by light-ion irradiation experiments and FEM calculation to be different not only from the unirradiated but also from post-irradiation behavior.
- (2) The transient nature of the significant enhancement of irradiation creep at 60°C was investigated by computer simulation and proved by light-ion irradiation experiments.

- (3) A low radio-activation martensitic steel, F82H (Fe-8Cr-2W-V,Ta), demonstrated its fairly good resistance against helium embrittlement through post helium implantation creep testing.
- (4) Helium induced grain boundary fracture of Fe-Ni-Cr austenitic materials was shown to be triggered by unstable growth of intergranular bubbles with over critical sizes.

<Research themes>

- (1) Research on the Deformation and Fracture of Materials under Irradiation (Nuclear Energy Research: April 1993 to March 1998, Johsei Nagakawa)
- (2) Understanding and Improvement of Radiation-Induced Degradation in the Advanced Nuclear Materials (Nuclear Energy Research: April 1998 to March 2003, Johsei Nagakawa)
- (3) Influence of Nuclear Transmutations on Low Radio-Activation Structural Materials for Fusion Reactor Application (Nuclear Energy Research: April 1996 to March 2001, Norikazu Yamamoto)

2.2.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) J. Nagakawa: Materials Deformation Induced by Radiation Damage, Nucl. Engi. 43, 46(1997).
- (2) Y. Murase, J. Nagakawa, N. Yamamoto, H. Shiraishi: Void Swelling in Proton Irradiated Fe-Cr-Ni Ternary Alloys, Fusion Technology 1996, 503(1997).
- (3) J. Nagakawa: Materials Deformation produced by Atomic Displacement Damage, Proc. Research Topics NRIM 1997, 7(1997).
- (4) J. Nagakawa: Research on the Materials Deformation and Fracture under Irradiation, Progress Report on the Nuclear Research in National Laboratories, 37, 90-1(1997).
- (5) Y. Murase, J. Nagakawa, N. Yamamoto, H. Shiraishi: Void Swelling in Fe-15Cr-xNi Ternary Alloys under Proton Irradiation, J. Nucl. Mater. 255, 34 (1998).
- (6) J. Nagakawa: Calculation of Radiation-Induced Deformation in the ITER Vacuum Vessel, J. Nucl. Mater. 258-263, 289(1998).
- (7) Y. Murase, J. Nagakawa, N. Yamamoto, H. Shiraishi: Effect of Cold-Work on Void Swelling in Proton Irradiated Fe-15Cr-20Ni Ternary Alloys, J. Nucl. Mater. 258-263, 1639(1998).
- (8) J. Nagakawa: Research on the Materials Deformation and Fracture under Irradiation, Progress Report on the Nuclear Research in National Laboratories,

38, 89(1998).

- (9) J. Nagakawa et al.: Materials Deformation and Fracture under Irradiation, NRIM Project Report 20, (1998), in press.
- (10) J. Nagakawa, Y. Murase, N. Yamamoto: Development of In-Beam Creep-Fatigue Machine, Proc. IEA/JUPITER Joint Symposium on Small Specimen Technologies for Fusion Research, (1999), in press.
- (11) J. Nagakawa: Radiation Damage in Fusion Reactor Materials, in *Computational Materials Design*, ed. by T. Saito, (Springer 1999), in press.
- (12) J. Nagakawa: Low Temperature Irradiation Creep, in *Problems in Materials System for the Next-step Fusion Devices*, ed. by H. Matsui, (Tohoku Univ. 1999), in press.
- (13) J. Nagakawa: On the Creep Deformation and Fatigue Fracture under Irradiation, Progress Report of Materials Tech. Committee for LWR, JRCM, (1999), in press.
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- (15) N. Yamamoto, J. Nagakawa, Y. Murase, H. Shiraishi: Bubble Microstructure and its Correlation to Creep Rupture Properties in Austenitic Alloys Implanted with Helium, Proc. 4th Japan-China Symp. on Materials for Advanced Energy Systems and Fission and Fusion Engineering, 161 (1997).
- (16) N. Yamamoto: Influence of Nuclear Transmutations on Low Activation Structural Materials for Fusion Reactor Application, Progress Report on the Nuclear Research in National Laboratories , 37, 2-1 (1997).
- (17) N. Yamamoto, J. Nagakawa, Y. Murase, H. Shiraishi: Helium Embrittlement of Ti and P Added Austenitic Alloys Crept at 923 K, Fusion Engrg. Des., 41, 111 (1998).
- (18) N. Yamamoto, J. Nagakawa, Y. Murase, H. Shiraishi: Microstructural Observation of Helium Implanted and Creep Ruptured Fe-25%Ni-15%Cr Alloys Containing Various MC and MN Formers, J. Nucl. Mater. , 258-263, 1628 (1998).
- (19) N. Yamamoto, J. Nagakawa, Y. Murase, H. Shiraishi: Effect of Combined Addition of Ti and P on Creep Rupture Properties of Helium Implanted Fe-25%Ni-15%Cr Alloy, J. Nucl. Mater. , 258-263, 1634 (1998).
- (20) N. Yamamoto: Influence of Nuclear Transmutations on Low Activation Structural Materials for Fusion Reactor Application, Progress Report on the Nuclear Research in National Laboratories, 38, 2-1 (1998).
- (21) N. Yamamoto, J. Nagakawa, Y. Murase, H. Shiraishi: Creep Rupture Properties of Titanium- and Phosphorus- Modified Fe-25%Ni-15%Cr Alloys Preimplanted with

Helium, Proc. IEA/JUPITER Joint Symposium on Small Specimen Technologies for Fusion Research, (1999), in press.

- (22) N. Yamamoto, J. Nagakawa, K. Shiba: An Evaluation on Helium-Embrittlement-Resistance of Low Activation Ferritic/Martensitic Steel F82H, J. Nucl. Sci. Technol, (1999), in press.

Oral Presentation: 23

- (1) International Conference: 8
- (2) Domestic Conference: 15

2.2.4 3rd Subgroup

Mitsutane Fujita (Subgroup Leader), Hiroshi Araki, Hiroshi Suzuki, Tetuji Noda (Director of Planning Office), Koichi Yagi (Supervising Researcher)

2.2.4.1 Research work

- (1) The isotope separation method has been developed using by a CO₂ laser with an oxygen isotope, and its application to materials has been studied by using synthetic isotopically controlled materials, such as silicon and boron compounds, or isotopes by transmutation, including simulation studies.
- (2) The preparation of isotopically purified silicon, for determining standard molar mass, was carried out by using highly purified silicon enriched with ²⁸Si.
- (3) The development of SiCf/SiC composites has been studied by using chemical vapor infiltration processes.
- (4) The distributed material database, named "Data-Free-Way", has been built in cooperation between the NRIM and the other three organizations.
- (5) The NRIM discussed with FIZ Karlsruhe and MPA Stuttgart to mutually exchange materials information.

<Research themes>

- (1) Isotope Separation and Its Application to Materials (Nuclear Energy Research: April 1997 to March 2002, Koichi Yagi)
- (2) Preparation of Isotopically Purified Silicon for Determining Standard Molar Mass Image (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1999, Koichi Yagi)
- (3) Development of Chemical Vapor Infiltration Process for SiCf/SiC Composites Image

(Special Coordination Funds for Promoting Science and Technology: April 1997 to March 2001, Koichi Yagi)

- (4) Research on Utilization Technique of "Data-Free-Way" System for Nuclear Materials (Nuclear Energy Research: April 1995 to March 2001, Mitsutane Fujita)
- (5) Building of Globalizing System for Mutual Usage of Material Information (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 1999, Koichi Yagi)

2.2.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

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- (2) T.Noda: High purity SiC/SiC Composites, New Mater. **17**, 62(1997).(in Japanese)
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- (4) A.Sagara, O.Motojima, S.Inagawa, K.Watanabe, H.Yamanishi, H.Chikaraishi, A.Kohyama, H.Matsui, T.Muroga, N.Noda, T.Noda, N.Ohyabu, T.Satow, A.A.Shishkin, S.Tanaka, T.Terai, K.Yamazaki, J.Yamamoto, FFHR Group : Blanket Design Using FLiBe in Helical-type Fusion Reactor FFHR, J. Nucl. Mater. **248**, 147(1997).
- (5) H.Suzuki, H.Araki, N.Ishikawa, T.Noda: Formation of Nano-crystalline SiC Films by Laser CVD at Low Temperatures, J. Surface Analysis, **3**, 426(1997).
- (6) T.Noda, S.Ito: Impurities and Evaluation of Induced Activity of V Alloys, Proc. IEA WS on Vanadium Alloy Development, 677(1997).
- (7) S.Tanaka, I.Shiota, H.Suzuki, T.Noda: Enrichment of ²⁸Si by Infrared Laser Irradiation, Functionally Graded Materials, 725(1997).
- (8) J.Yu, Y.Chen, Y.Wang, W.Zhang, S.Xu, T.Noda: Research on plasma-facing component materials, J. Nucl. Mater. **233-237**, 771(1997).
- (9) A.Sagara, O.Motojima, S.Imagawa, O.Mitarai, T.Noda: Design Studies of Helical-type Fusion Reactor FFHR, Fusion Eng. & Design, **41**, 349(1998).
- (10) T.Noda: High Purity SiC Composites, Nucl. Viewpoints, **44**, 60(1998).
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- (12) T.Noda, H.Suzuki, N.Nogi: Prospect of Isotope Controlled Materials, Bulletin of ISIJ, **3**, 23(1998).

- (13) A.Sagara, K.Y.Watanabe, K.Yamazaki, O.Motojima, M.Fujiwara, O.Mitarai, S.Imagawa, H.Yamanishi, H.Chikaraishi, A.Kohyama, H.Matsui, T.Muroga, T.Noda, N.Ohyabu, T.Satow, A.A.Shishkin, S.Tanaka, T.Terai, T.Uda: LHD-Type Compact Helical Reactors, IAEA-CN-69/FTP103, 1(1998).
- (14) T.Uda, A.Sagara, O.Motojima, H.Yamanishi, S.Tanaka, T.Terai, T.Noda: Study of Safety Concept for a Helical-type Fusion Reactor FFHR, Fusion Eng. & Design, 42, 115(1998).
- (15) H.Araki, H.Suzuki, W.Yang, S.Sato, T.Noda: Effect of High Temperature Heat Treatment in Vacuum on Microstructure and Bending Properties of SiC/SiC Composites Prepared by CVI, J. Nucl. Mater. 258-263, 1540(1998).
- (16) T.Hinoki, W.Zhang, A.Kohyama, S.Sato, T.Noda: Effect of Fiber Coating on Interfacial Shear Strength of SiC/SiC by Nano-indentation Technique, J. Nucl. Mater. 258-263, 1567(1998).
- (17) W.Zhang, T.Hinoki, Y.Katoh, A.Kohyama, T.Noda, T.Muroga, J.Yu: Crack Initiation and Growth Characteristics in SiC/SiC under Indentation Test, J. Nucl. Mater. 258-263, 1577(1998).
- (18) T.Noda, H.Suzuki, H.Araki: Silicon Isotope Enrichment for Low Activation, Fusion Eng. & Design, 41, 173(1998).
- (19) A.Sagara, T.Muroga, O.Motojima, T.Noda, S.Tanaka, T.Terai, A.Kohyama, H.Matsui: Materials Design and Related R&D Issues for the Force-free Helical Reactor (FFHR), J. Nucl. Mater. 258-263, 2079(1998).
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- (41) Y.Tachi, J.Saito, M.Fujita, J.Kinugawa, H.Tsuji, Y.Kaji, K.Shimura, R.Nakajima, S.Iwata: Utilization of Image Data in Distributed Material Database named Data-Free-Way, J. Nucl. Sci. Technol. (1999) in press

Oral Presentation: 82

- (1) International Conference: 27
- (2) Domestic Conference: 55

Patent:

- (1) Synthesis of Pure Disilicon Hexafluoride: T. Noda, H. Suzuki, H. Araki, 1997, No.2664048
- (2) Synthesis of Pure Disilicon Hexafluoride: T. Noda, H. Suzuki, H. Araki, 1997, S.N.08/526919 in USA.
- (3) High Purity SiC Composites and the Production: T. Noda, H. Araki, A. Kohyama: 1998, H10-143044
- (4) Production Method of SiCf/SiC Composites: T. Noda, H. Araki, A. Kohyama, 1998, H10-143045
- (5) Isotopical Silicon Film and the Production Method: H. Suzuki, H. Araki, T. Noda: 1999, requesting
- (6) Retrieval System of CCT Diagrams: M. Fujita, T. Kasugai, 1998, H10-217278.

Award:

- (1) Commendation by the Minister of State for Science and Technology 1997, May 29 from Science and Technology Agency, T. Noda.
- (2) Best of Report Award 1998 Spt.20, from The Japan Institute of Metals, H. Suzuki, H.

Araki, T. Noda.

- (3) Persons of Scientific and Technological Information Research Merits, 1998 Oct.14
from Japan Science and Technology Corporation, M. Fujita.

2.2.5 4th Subgroup

Masanori Yata (Subgroup Leader)

2.2.5.1 Research Work

Our experimental program is aimed to understand the chemical reactivity of molecules on solid surfaces and clusters and to understand the elementary processes of the reactions using “state-selected” molecular beams. Our final goal is to obtain the answers to the following questions: (1) how will the initial state of reactants affect the elementary process of the reactions? And how can we control the branch ratio of the product yield by the way in which the reactants are excited ?

<Research theme>

- (1) Control of the synthetic process of transition-metal oxides by “state-controlled” molecular beam techniques (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 2000, Masanori Yata)

2.2.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) M. Yata, H. Rouch, K. Nakamura: Kinetics of Oxygen Surfactant in Cu(001) Homoepitaxial Growth, *Phys. Rev.*, **B56**, 10576(1997).
- (2) M. Yata, H. Rouch, K. Nakamura: Role of Oxygen Adatoms in Homoepitaxial Growth of Cu(001), *Mater. Res. Soc. Symp.Proc.* **528**, 59(1998).
- (3) M. Yata, H. Rouch, K. Nakamura: Kinetics of Oxygen Surfactant-mediated Epitaxial Growth of Cu(001), *J. Surf. Sci. Soc. Jpn.*, **19**, 92(1998).
- (4) K. Nakamura and M.Yata: An Interpretation of the Temperature-dependent Growth Mode of Copper on the Cu(001)-(2 $\sqrt{2}\times\sqrt{2}$)R45° -O Surface, *Surf. Sci.* **417**, 268 (1998).
- (5) M.Yata and H. Rouch: Control of the Initial Oxidation on Cu(001) Surface by Selection of Translational Energy of O₂ Molecules, *Appl. Phys. Lett.*, **75**, 1(1999).
- (6) M. Yata and Y.Saito: The Interaction of O₂ Molecules with Clean and O-covered Cu(001), *Surf. Sci.*, in press.

- (7) M. Yata and Y.Saito: Dynamics of the Initial Oxidation of Cu(001) Surface, J. Chem. Phys. submitted.

Oral Presentation: 10

- (1) International Conference: 3
(2) Domestic Conference: 7

2.2.6 Researchers in the 2nd Research Group

Age and research fields of researchers in the 2nd Research Group. (March 31,1999)

Age of Researchers	Number	Metal-lurgy	Solid-state physics	Process-ing	Data-base	Mechanics
60 ~						
50 ~ 59	3	1			1	1
40 ~ 49	5	2	2	1		
30 ~ 39	4	1	2	1		
22 ~ 29						
Summation	12	4	4	2	1	1

2.2.7 Research Budgets in the 2nd Research Group

Research budgets in the 2nd Research Group (yen)

Kinds of budgets	1997	1998
Nuclear Energy Research	113,727,000	117,742,000
Special Coordination Funds for Promoting Science and Technology	15,750,000	45,477,000
Supplementary Budget	0	25,000,000
Summation	129,477,000	188,219,000

2.3 3rd Research Group (Advanced High Temperature Materials)

Morihiro Nakamura (Supervising Researcher)

2.3.1 Research Work

We need to improve high temperature materials for the efficient use of thermal energy in order to conserve natural resources and to protect the environment. Following the 5th Long-term Plan, the R&D of advanced high temperature materials for high temperature applications has been promoted at the 3rd Research Group. Efforts for fabrication and improvement of advanced high temperature materials have been made. The research work at the 3rd Research Group are divided into two groups: in the first group, ultra-high temperature materials, like Ni base superalloys and “refractory superalloys”, are studied, and in the other, light-weight high temperature alloys like TiAl base, Al₃Ti base and Ti₂AlNb base alloys, are studied. Examples of the outcomes are shown next.

- (1) Advanced Ni base superalloys for higher temperature uses have been developed with the alloy design system which we developed.
- (2) “Refractory superalloys” for ultra-high temperature applications have been proposed and realized using Pt group metals like Ir and Rh as base metals.
- (3) The high temperature deformability of TiAl base alloys has been improved through isothermal forging and macroalloying, and the room temperature ductility of Al₃Ti base alloys like (Al, Mn)₃Al are studied by the addition of a 4th element.
- (4) The high temperature strength of Ti₂AlNb base alloys has been improved through compositional and microstructural modification, and fabrication of the alloys has been carried out using powder metallurgy techniques.

The member of the 3rd Research Group, as at March 31, 1999, consisted of 17 researchers, 3 STA fellows, 1 domestic research fellow, 4 visiting researchers, 1 graduate student and 4 guest researchers.

The output of the research activities has been increasing since the start of the 5th long-term plan. The number of publications including submitted papers is 111, patents submitted or registered are 12 from April 1, 1997 to March 31, 1999. The details are shown in the research activities of each laboratory.

2.3.2 1st Subgroup

Hiroshi Harada (Subgroup Leader), Shizuo Nakazawa, Yoshikazu Ro, Yutaka Koizumi, Hideyuki Murakami, Yoko Yamabe-Mitarai, Yuefeng Gu (STA Fellow), Chang-Seok Oh (STA Fellow), Xi-Hong Yu (visiting researcher), Makoto Osawa (visiting researcher), Takayuki Sakai (visiting researcher), Takehisa Hino (visiting researcher), Chiaki Tanaka (guest researcher), Yomei Yoshioka (guest researcher).

(Fifth Laboratory, Computational Materials Division)

Toshihiro Yamagata (Head), Tadaharu Yokokawa, Toshiharu Kobayashi

2.3.2.1 Research work

The 3rd and 4th generation Ni-base single crystal (SC) and directionally solidified (DS) superalloys for advanced power generation gas turbines and aeroengines have been (/are being) developed under collaborations with Rolls Royce, NEDO (MITI), IHI, Toshiba, Kawasaki, and so on. NiTi-base high temperature alloys were newly proposed as high specific-strength materials and evaluated. “Refractory superalloys” for ultra-high temperature uses were also proposed and being realized with using Pt group metals, e.g., Ir and Rh, as base metals. Atomistic level computer modellings using CVM and Monte Carlo simulations supported by our experimental work with APFIM, High Temperature X-ray Diffractometry, etc, as well as more empirical alloy design method based on databases, were established and used mainly in the Ni-base superalloy developments.

The work above in this Subgroup led to “High Temperature Materials 21 Project” launched in June 1999, as a Leading Project for “Dokuritu Gyousei Hojinnka” taking place in April 2001.

<Research themes>

(1) Atomic Arrangement Design of Ultra-high Temperature Alloys

(Computational Materials Research: April 1995 to March 2000, Hiroshi Harada)

(2) Virtual Experiment for Materials Design (Phase 1)

- Design of High Temperature Materials by Using Statistical Thermodynamics Calculation -

(Special Coordination Funds for Promoting Science and Technology: April 1995 to March 1998, Hiroshi Harada)

(3) Virtual Experiment for Materials Design (Phase 2)

- Design of Refractory Superalloys -

(Special Coordination Funds for Promoting Science and Technology: April 1998 to

- March 2000, Hiroshi Harada)
- (4) Development of Single Crystal Superalloys for High Efficiency Gas Turbines
(NEDO Research Grant for Advanced Technology: October 1997 to March 2000, Hiroshi Harada)

2.3.2.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) Yuefeng Gu, Y. Yamabe-Mitarai, Y. Ro, T. Yokokawa, H. Harada: Microstructures and Compressive Properties of Ir-15Nb Refractory Superalloys Containing Nickel, *Scripta Mater.*, **39**, 723 (1998)
- (2) Yuefeng Gu, Y. Yamabe-Mitarai, Xihong Yu, Y. Ro, H. Harada: Effects of Nickel Addition on Strength and Fracture Behavior of Ir-15Nb Two Phase Refractory Superalloy, Oxford-Kobe Materials Seminar 1998, Poster paper, p. 1 (1998)
- (3) Yuefeng Gu, Y. Yamabe-Mitarai, Y. Ro, T. Yokokawa, H. Harada: Effects of Nickel Content on Microstructures and Compressive Properties of Ir-15Nb Refractory Superalloy, JSPS 123 Committee Research Report, **39**, 137 (1998)
- (4) Yuefeng Gu, Y. Yamabe-Mitarai, Y. Ro, H. Harada: Microstructures and Deformation Behavior of Ir-Nb Two Phase Refractory Superalloys for Various Nb Content, *Scripta Mater.*, **40**, 1313 (1999)
- (5) Yuefeng Gu, Y. Yamabe-Mitarai, Y. Ro, T. Yokokawa, and H. Harada, Superior Mechanical Properties and Fracture Behavior of Ir-15at% Nb Two-phase Refractory Superalloys with Nickel Additions, *Met. Trans.A*, accepted
- (6) Yuefeng Gu, Y. Yamabe-Mitarai, Y. Ro, Y. Yokokawa, H. Harada, Influence of Nickel Addition on Strength and Fracture Behavior of Ir-15Nb Two-phase Alloy, *Mat. Res. Soc. Symp. Proc.*, (1998)
- (7) Yuefeng Gu, Y. Yamabe-Mitarai, Y. Ro, and H. Harada, Microstructures and Mechanical Properties of Ir-Nb-Ni fcc-L12 Two-phase Refractory Superalloys, *IUMRS-ICAM'99*, in press
- (8) Yuefeng Gu, Y. Yamabe-Mitarai, Xihong Yu, and H. Harada, Microstructures and Compressive Properties of Ni-doped Rh-15Nb Two-phase Refractory Superalloys, *Materials Letters*, in press
- (9) Yuefeng Gu, Y. Yamabe-Mitarai, Y. Ro, T. Yokokawa, T. Maruko, H. Harada, Microstructures and Fracture Behavior of Ir-15Nb-XNi and Rh-15Nb-XNi Two-phase Refractory Superalloys, *ATEM'99*, in press
- (10) H. Harada, Y. Yamabe-Mitarai: Superalloys for Gas Turbines; Present and Future

- (Review paper), *Zairyo Kagaku* (Journal of Japan Institute of Materials Science), **34**, 63 (1997) (in Japanese)
- (11) H. Harada : High Temperature Materials Design (Review paper), JSPS 123 Committee Research Report, **38**, No.3 (Special Issue for the 40th Anniversary Seminar) 185 (1997) (in Japanese)
 - (12) H. Harada, T. Yamagata: Researches on Superalloys for Gas Turbines at National Research Institute for Metals (Review paper), *J. Gas Turbine Soc. of Japan*, **25**, 110 (1997) (in Japanese)
 - (13) H. Harada: Development of Superalloys for High Efficiency Gas Turbines (Review paper), *Kogyo Zairyo*, **46**, 114 (1998) (in Japanese)
 - (14) H. Harada, H. Murakami : Materials Design; Its Application to High Temperature Materials (Review paper), *JIM Seminar Textbook*, p. 61 (1998) (in Japanese)
 - (15) H. Harada: Design of Ni-base and Ir-base Superalloys, Oxford-Kobe Materials Seminar 1998, p. 131 (1998)
 - (16) H. Harada, T. Yamagata: GT-Related Research at NRIM (Review paper), *Bulletin of Gas Turbine Society of Japan*, **46** (1998) (in Japanese)
 - (17) H. Harada and H. Murakami, Design of Ni-base Superalloys (Review paper), *Computational Materials Design*, Springer-Verlag, ed. by T. Saito, in press
 - (18) T. Hino, Y. Yoshioka, K. Nagata, H. Kashiwaya, T. Kobayashi, Y. Koizumi, H. Harada, T. Yamagata: Design of High Re Containing Single Crystal Superalloys for Industrial Gas Turbines, 6th Liege Conf. on "Materials for Advanced Power Engineering 1998", p. 1129 (1998)
 - (19) T. Hino, Y. Yoshioka, K. Nagata, H. Kashiwaya, T. Kobayashi, Y. Koizumi, H. Harada, T. Yamagata: Design of High Re Containing Single Crystal Superalloys for Industrial Gas Turbines, *Met. Mat. Trans. A*, submitted
 - (20) T. Hino, Y. Ishikawa, Y. Yoshioka, K. Nagata, T. Kobayashi, Y. Koizumi, H. Harada, T. Yamagata: Design of Single Crystal Superalloys for Industrial Gas Turbines, Inter. Gas Turbine Congress 1999 Kobe, November (1999), submitted.
 - (21) T. Kobayashi, Y. Koizumi, S. Nakazawa, H. Harada and T. Yamagata: Design of a High Rhenium Containing Single Crystal Superlloy with Balanced Intermediate and High Temperature Creep Strengths, 4th Inter. Charles Parsons Turbine Conf. of Advances in Turbine Materials, Design and Manufacturing, p. 766 (1997)
 - (22) T. Kobayashi, Y. Koizumi, T. Yokokawa, S. Nakazawa, H. Harada, T. Yamagata: Design of a Third Generation Single Crystal Superlloy with Excellent Phase Stability, JSPS 123 Committee Research Report, **39**, 9 (1998) (in Japanese)
 - (23) T. Kobayashi, Y. Koizumi, H. Harada, T. Yamagata, A. Tamura, S. Nitta: Design of

- 3rd Generation DS Superalloy TMD-103, 6th Liege Conf. on "Materials for Advanced Power Engineering 1998", p. 1079 (1998)
- (24) Y. Koizumi, T. Kobayashi, T. Kimura, M. Osawa and H. Harada: 3rd Generation Single Crystal Superalloys with Excellent Processability and Phase Stability, 6th Liege Conf. on Materials for Advanced Power Engineering 1998, p. 1089 (1998)
 - (25) Y. Koizumi, T. Kobayashi, H. Harada, T. Yamagata: Creep Life Extension of a Single Crystal Superalloy by Re-heat-treatment, 4th Inter. Charles Parsons Turbine Conf. of Advances in Turbine Materials, Design and Manufacturing, p. 679 (1997)
 - (26) M. Maldini, T. Yamagata, H. Harada, V. Lupinc, M. Yamazaki: Effect of a Thermo-mechanical Treatment on Gamma' Morphology and Creep Behavior Single Crystal Nickel-base Superalloys, 11th Congress Inter. Federation for Heat Treatment and Surface Engineering, p. 131, Florence, Italy, October (1998)
 - (27) H. Murakami, P.J. Warren, T. Kumeta, Y. Koizumi, H. Harada: Microstructural Evolution of β/β' Two Phase Quaternary Alloys, J. Surface Analysis, 56(1997)
 - (28) H. Murakami, P.J. Warren, T. Kumeta, Y. Koizumi, H. Harada: Microstructural Characterization of NiTi-Based $\beta_2/\text{Ni}_2\text{TiAl}$ -based β' Two Phase Alloys, Proc. the 2nd Inter. Symp. on Structural Materials, Seven Springs, U.S.A, p. 877 (1997)
 - (29) H. Murakami, P.J. Warren, T. Kumeta (Ibaraki University), Y. Koizumi, H. Harada: Microstructural Evolution of β_2/NiTi -Based β' Ni_2TiAl -based Two Phase Alloys, The Asian Science Seminar, p. 323 (1997)
 - (30) H. Murakami, Y. Koizumi, T. Yokokawa, Y. Yamabe-Mitarai and H. Harada: Atom-probe Microanalysis of Ir-bearing Ni-base Superalloys, Materials Science and Engineering A250, p. 109 (1998)
 - (31) H. Murakami, M. Osawa, T. Yokokawa, Y. Koizumi, T. Yamagata, H. Harada: The Location of Atoms in Ir-containing Ni-base Single Crystal Superalloys, 6th Liege Conf. on Materials for Advanced Power Engineering 1998, p. 1139 (1998)
 - (32) H. Murakami, T. Yokokawa, M. Osawa, Y. Koizumi, H. Harada: Atomistic Investigation of Ir-containing Ni-base Single Cryatal Superalloys, Oxford-Kobe Materials Seminar 1998, p. 13 (1998)
 - (33) H. Murakami, H. Harada, Y. Saito: Design and Analysis of Ni-base Superalloys on an Atomistic Basis, J. Japan Inst. Metals, 63, 723 (1999) (in Japanese)
 - (34) M. Osawa, T. Yokokawa, T. Kobayashi, Y. Koizumi, H. Harada: X-ray Diffraction Measurement of Gamma/Gamm-prime Lattice Misfit and Lattice Distortion in Ni-base Superalloys, Oxford-Kobe Materials Seminar 1998, Poster paper, p. 8 (1998)

- (35) H. Sato, K. Itoh, M. Shimizu, Y. Yamabe-Mitarai, H. Harada, T. Maruko, Y. Nakamura: Solar Thermal Thruster Made of Iridium, 21st Inter. Symp. on Space Technology and Science, 98-a-2-02, p. 1, Omiya, Japan, May (1998)
- (36) Y. Yamabe-Mitarai, Y. Koizumi, H. Murakami, Y. Ro, T. Maruko, H. Harada: Rh-base Refractory Superalloys for Ultra-high Temperature Use, Scripta Mater., **36**, 393 (1997)
- (37) Y. Yamabe-Mitarai, Y. Ro, T. Yokokawa, T. Maruko, H. Harada: Growth of L12 Precipitates in FCC Matrix in Ir Binary Alloys, The Asian Science Seminar, p. 277 (1997)
- (38) Y. Yamabe-Mitarai, Y. Koizumi, H. Murakami, Y. Ro, T. Maruko, H. Harada: Platinum Group Metals Base Refractory Superalloys, Mat. Res. Soc. Symp., vol. 460, p. 701 (1997)
- (39) Y. Yamabe-Mitarai, Y. Ro, T. Maruko, T. Yokokawa, H. Harada: Platinum Group Metals-base Refractory Superalloys for Ultra-high Temperature Use, Structural Intermetallics 1997, p. 805 (1997)
- (40) Y. Yamaba-Mitarai, Y. Ro, T. Maruko, H. Harada: Ir-base Refractory Superalloys for Ultra-high Temperature Use, Met. Mat. Trans. A, **29A**, 537 (1998)
- (41) Y. Yamabe-Mitarai, Y. Ro, T. Maruko, T. Yokokawa, H. Harada: Precipitate Shape Dependence of Strength in Ir-base Refractory Superalloys, 6th Liege Conf. on "Materials for Advanced Power Engineering 1998", p. 1147 (1998)
- (42) Y. Yamabe-Mitarai, Y. Ro, T. Maruko, H. Harada: Microstructure Dependence of Strength of Ir-base Refractory Superalloys, Intermetallics, **7**, 49 (1999)
- (43) Y. Yamabe-Mitarai, H. Harada: Design of Refractory Superalloys Based on Ir and Rh, U.S.-Japan Workshop on Very High Temperature Structural Materials, p. 117, Hawaii, December 9-11 (1998)
- (44) Y. Yamabe-Mitarai, Y. Ro, T. Maruko, H. Harada: Precipitation hardening of Ir-Nb and Ir-Zr alloys, Scripta Mat., **40**, 109 (1999)
- (45) Y. Ymabe-Mitarai, Xihong Yu, Yuefeng Gu, Y. Ro, S. Nakazawa, T. Maruko, H. Harada, Microstructures and High temperature strengths of Ir-based and Rh-based Refractory Superalloys, ATEM'99, in press
- (46) T. Yokokawa, M. Osawa, H. Murakami, T. Kobayashi, Y. Koizumi, T. Yamagata, H. Harada: High Temperature Measurement of Gamma/Gamma-prime Lattice Misfit in a 3rd Generation Ni-base Superalloy, 6th Liege Conference on Materials for Advanced Power Engineering 1998, p. 1121 (1998)
- (47) T. Yokokawa, K. Ohno, H. Murakami, T. Kobayashi, T. Yamagata, H. Harada: Accurate Measurement of Lattice Misfit between Gamma and Gamma-prime

Phases in Nickel-base Superalloys at High Temperatures, *Advances in X-ray Analysis*, Vol.39, p. 449 (1997)

- (48) T. Yokokawa, H. Harada, K. Ohno: X-ray Diffractometric Measurement of Lattice Misfit between Gamma/Gamma-prime Phases of Ni-base Superalloys at High Temperatures, *J. Japan Welding Society*, **67**, 34 (1998) (in Japanese)
- (49) S. Yoshitake (Mitsubishi Material), V. Narayan, H. Harada, H.K.D.H. Bhadeshia, D.J.C. Mackay: Estimation of the Gamma and Gamma-prime Lattice Parameters in Nickel-base Superalloys Using Neural Network Analysis, *ISIJ Inter.*, **38**, 495 (1998)
- (50) Xihong Yu, Y. Yamabe-Mitarai, Yuefeng Gu, H. Harada: The Investigation on Ir-Nb-Ni-Al Quaternary Refractory Superalloys, *Aerospace materials*, The 1st Oxford-Kobe Materials Seminar 1998, Poster paper, p. 17 (1998)
- (51) Xihong Yu, Y. Yamabe-Mitarai, Y. Ro, Yuefeng Gu, H. Harada: Development of Quaternary Ir-Ta-Ni-Al Refractory Superalloys, *Scripta Mater.*, in press
- (52) Xihong Yu, Y. Yamabe-Mitarai, Y. Ro, H. Harada: Newly Developed Quaternary Refractory Superalloys, *IUMRS-ICAM'99*, in press
- (53) Xihong Yu, Y. Yamabe-Mitarai, Y. Ro, H. Harada: Design of Quaternary Ir-Nb-Ni-Al Refractory Superalloys, *Met. Mat Trans. A*, in press
- (54) H. Fujii (Osaka University), D.J.C. MacKay (University of Cambridge), H.K.D.H. Bhadeshia (University of Cambridge), H. Harada, K. Nogi (Osaka University): Estimation of Creep Rupture Strength in Nickel-base Superalloys, 6th Liege Conf. on Materials for Advanced Power Engineering 1998, p. 1401 (1998)

• **Oral Presentation:** 60

(1) International: 26

(2) Domestic Conference: 34

• **Patent Applications**

- (1) Nickel-based Single Crystal Alloy and a Method of Manufacturing the Same:
T. Kobayashi, Y. Koizumi, S. Nakazawa, H. Harada, T. Yamagata,
Japan: October 31, 1997, 09-316111
EU(5 countries): October 30, 1998, 98308931.9
USA: September 23, 1998, 09/159.494
- (2) Ni-base Superalloy, a Method of Manufacturing the Same, and a Gas Turbine Parts made of the Same:
T. Hino, Y. Yoshioka, T. Suzuki, T. Kobayashi,

- T. Yokokawa, Y. Koizumi, H. Harada,
Japan: August 13, 1998, 10-228986
- (3) Ni-base Superalloy, a Method of Manufacturing the Same, and a Gas Turbine Parts made of the Same:
T. Hino, Y. Yoshioka, T. Suzuki, T. Kobayashi,
T. Yokokawa, Y. Koizumi, H. Harada,
Japan: March 4, 1998, 10-067671
USA: March 3, 1999, 09/261.230
- (4) Ni-base Superalloy, a Method of Manufacturing the Same, and a Gas Turbine Parts made of the Same:
T. Hino, Y. Yoshioka, K. Nagata, T. Kobayashi,
Y. Koizumi, S. Nakazawa, T. Yokokawa, H. Harada,
Japan: February 25, 1999, 11-049031
- (5) Nickel-base Superalloys with Iridium Addition:
T. Kobayashi, Y. Koizumi, H. Murakami, Y. Ro, Y. Yamabe, S. Nakazawa, H. Harada, T. Yamagata
October 28, 1998, 09-295552
- (6) A Method of Manufacturing Nickel-base Directionally Solidifies Superalloys:
T. Kobayashi, Y. Koizumi, H. Harada, T. Yamagata, A. Tamura, S. Nitta,
Japan: March 2, 1998, 10-066204
USA: February 26, 1999, 09/257910
EU(7 countries): March 2, 1999, 99104190.6
- (7) Refractory Superalloy and a Manufacturing Method of the Same:
Y. Yamabe-Mitarai, Yuefeng Gu, Xihong Yu, Y. Ro, S. Nakazawa, H. Harada,
Japan: Feb. 2, 1999, 11-025540

• **Patent registered**

- (1) NiTi-base High Specific-strength Heat Resistant Alloys:
Y. Koizumi, S. Nakazawa, Y. Ro, H. Harada,
November 11, 1998, Japan 2847177
- (2) A Method of Manufacturing Nickel-base Directionally Solidifies Superalloys:
T. Kobayashi, Y. Koizumi, H. Harada, T. Yamagata, A. Tamura, S. Nitta,
Japan: March 26, 1999, 2905473

• **Award**

- (1) JIM “Shorei-sho” for Encourageing Young Scientist,

- September, 1997, Yoko Yamabe-Mitarai
(2) Commendation for Achievements, STA,
May, 1997, Toshiharu Kobayasi

2.3.3 2nd Subgroup

Aperiodic Materials Research Team

2.3.4 3rd Subgroup

Masuo Hagiwara (Subgroup Leader), Satoshi Emura, Feng Tang (domestic research fellow)

2.3.4.1 Research Work

- (1) The effect of compositional and microstructural modification, and the homogeneous dispersion of fine ceramic particulates such as TiB and Er₂O₃ on the elastic modulus, density and high temperature mechanical properties of orthorhombic Ti₂AlNb intermetallic alloys have been studied.
- (2) The fatigue behavior of conventional titanium alloy-based particulate composites such as Ti-6Al-2Sn-4Zr-2Mo/10%TiB has been evaluated under the creep-fatigue interaction, and the fracture mechanisms have been analyzed.

<Research themes>

- (1) Research and Development of High Temperature Ti₂AlNb Titanium Aluminide-Based Composites (General Research: April 1997 to March 1999, Masuo Hagiwara)
- (2) Evaluation of High Temperature Properties for Titanium-Based Particulate Composites (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1998, Masuo Hagiwara)

2.3.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) M. Hagiwara, S. Emura, Y. Kawabe, S.J. Kim: Low Cost Synthesis of P/M Ti Alloys and Ti-based Particulate Composites, *Non-Aerospace Application of Titanium*, ed.

- by F. H. Froes, P.G. Allen and M. Niinomi, p.21 (TMS, Warrendale 1998), TMS Annual Meeting, San Antonio, February 16-19, 1998
- (2) M. Hagiwara, S. Emura, S.J. Kim, M. Niinomi: P/M synthesis of Ti Alloy-Based Particulate Composites and Their Properties, *Advanced Materials and Processings*, ed. by M.A. Imam, R. Denale, S. Hanada, Z. Zhong and D.N. Lee, p.361 (TMS, Warrendale 1998), 3rd Pacific Rim Inter. Conf. on Advanced Materials and Processing (PRICM-3), Hawaii, July 12-16, 1998
 - (3) M. Hagiwara, S.J. Kim, S. Emura, F. Tang: Improved High Cycle Fatigue Strength of BE P/M Ti-6Al-1.7Fe-0.1Si Alloy and Ti-6Al-1.7Fe-0.1Si/10TiB Composites, Powder Metallurgy, World Congress and Exhibition 1998, Granada, October 18-22, 1998, in press
 - (4) F. Tang, S. Emura, M. Hagiwara: Effect of Compositional Modification on the Density, Young's Modulus and Mechanical Properties of Orthorhombic Ti₂AlNb-Based Alloys, Xi'an Inter. Titanium Conf., Xi'an, September 15-18, 1998, in press
 - (5) M. Hagiwara, S. Emura, Y. Kawabe: Synthesis and Property Evaluation of P/M Ti-6Al-2Sn-4Zr-2Mo/TiB Particulate Composites, *Tetsu-to-Hagane*. 83, 821(1997) (in Japanese)
 - (6) M. Niinomi, S. Takahashi, M. Hagiwara, S. Emura, Y. Kawabe, S.J. Kim: Fracture Toughness and Microstructure in TiB Particulate-reinforced Ti-6Al-2Sn-4Zr-2Mo Composites, *Tetsu-to-Hagane*. 84, 452 (1998) (in Japanese)
 - (7) M. Hagiwara, S.J. Kim, S. Emura, Y. Kawabe: Improvement of High Cycle Fatigue Strength of P/M Ti-6Al-2Sn-4Zr-2Mo/TiB Particulate Composites by the Modification of Matrix Microstructure, *Tetsu-to-Hagane*, 84, 678 (1998) (in Japanese)
 - (8) S. Emura, M. Hagiwara, Y. Kawabe: Microstructural Control and Improvement of Fatigue Properties in Ti₃Al-Nb Based Alloys Produced by Blended Elemental Powder Metallurgy, *J. Japan Inst. Metals*, 62, 621 (1998) (in Japanese)
 - (9) S. Emura, M. Hagiwara, Y. Kawabe: Effect of a Matrix Microstructure on the High Cycle Fatigue Properties of TiB Particulate Reinforced Ti₃Al-Nb Matrix Composites, *J. Japan Inst. Metals*, 63, 383 (1999) (in Japanese)
 - (10) S. Wanikawa, S. Emura, M. Hagiwara: MA Synthesis of TiC-Reinforced Titanium Composites and Particle Size Dependence of Tensile Properties, *J. Japan Soc. Powder and Powder Metall.*, 46, 484 (1999) (in Japanese)
 - (11) M. Hagiwara, S.J. Kim: Low Cost Synthesis and Property Evaluation of BE P/M Ti Alloys and Ti-Based Particulate Composites, *Metals Mater.* 4, 141 (1998)
 - (12) M. Hagiwara, S.J. Kim: Blended Elemental P/M Synthesis of Ti-6Al-1.7Fe-0.1Si

- Alloy with Improved High Cycle Fatigue Strength, *Scripta Mater.*, **39**, 1185 (1998)
- (13) F. Tang, S. Emura, M. Hagiwara: Modulated Microstructure in Ti-22Al-11Nb-4Mo Alloy, *Scripta Mater.* **40**, 471 (1999)
 - (14) L. Wang, M. Niinomi, M. Hagiwara, S. Emura, Y. Kawabe, S.J. Kim: Relationship Between Fracture Toughness and Microstructure of Ti-6Al-2Sn-4Zr-2Mo Alloy Reinforced with TiB Particles, *Mater. Sci. Eng.*, **A263**, 319 (1999)
 - (15) S. Emura, M. Hagiwara: Blended Elemental P/M Synthesis and Property Evaluation of an Orthorhombic Ti-22Al-27Nb Alloy, submitted to *J. Japan Inst. Metals* (in Japanese)
 - (16) F. Tang, M. Hagiwara: Tensile and Fracture Behavior of Ti-22Al-11Nb-4Mo Alloy, submitted to *Scripta Mater.*

• **Oral Presentation: 10**

- (1) International Conference: 4
- (2) Domestic Conference: 6

• **Patent Application**

- (1) Particulate-Reinforced Titanium-Based Composites and Their Manufacturing Method: M. Hagiwara, S. Emura, Y. Kawabe,
Registered on November 20, 1998. Patent No:2852414
- (2) Light Weight High Strength Titanium Alloy for High Temperature Application:
M. Hagiwara, F. Tang, S. Emura,
Applied on September 14, 1998. Application No:260683

2.3.5 4th Subgroup

Minoru Nobuki(Subgroup Leader), Kenki Hashimoto, Kazuo Kasahara, Tatuo Kumagai, Eiji Abe, Kewei Gao (STA fellow), Yasuhisa Yamamoto (visiting researcher), Tokuzo Tsujimoto (guest researcher)

2.3.5.1 Research work

- (1) The $\alpha \rightarrow \gamma$ phase transformation kinetics and structure evolution during the rate control cooling process of gamma TiAl alloys have been studied as a function of temperature by using SEM, EPMA and HRTEM.
- (2) Microstructure modification of gamma TiAl alloys has been performed by

isothermal die forging after heat-treatment and their mechanical properties have been examined with tensile tests at high temperatures.

- (3) The microstructure and room temperature ductility of titanium trialuminides single-phase alloys have been studied by EPMA, SEM, laser-micrography, X-ray diffractometry, hardness tests and 3-point bend tests.
- (4) Structure analyses of quasi-crystals are performed using HRTEM.

<Research themes>

- (1) Microstructure Control and Properties of Intermetallic Alloys in Ti-Al Alloy Systems (General Research: April 1997 to March 2001, Minoru Nobuki)
- (2) Local Fluctuation of Structure for Intermetallic Phases with Non-equilibrium and Non-stoichiometric Compositions (Special Coordination Funds for Promoting Science and Technology: April 1, 1997 to March 31, 1998, Eiji Abe)
- (3) Role of Hydrogen in Environmental Embrittlement of Structural Materials (Hydride Formation and Embrittlement Behavior of Intermetallic Compounds) (Special Coordination Funds for Promoting Science and Technology: April 1, 1998 to March 31, 2001, Morihiko Nakamura)

2.3.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) E. Abe, S. Kajiwarra, T. Kumagai, M. Nakamura: High-resolution Electron Microscopy of Twin Interfaces in Massively Transformed γ -TiAl, *Phil. Mag. A*, **75**, 975 (1997).
- (2) E. Abe, M. Nakamura: The Structure of Antiphase Domain Boundaries in Massively Transformed γ -TiAl Studied by High-resolution Electron Microscopy, *Phil. Mag. Lett.*, **75**, 65 (1997).
- (3) E. Abe, T. Kumagai, M. Nakamura: The α - γ Phase Transformation Mechanisms in Two-phase γ -TiAl Based Intermetallic Alloys, *Proc. of Int. Symp. Structural Intermetallics-2 (TMS-AIME, Warrendale)*, p. 167-176 (1997).
- (4) E. Abe, T. Kumagai, S. Kajiwarra, M. Nakamura: Microstructure of Massively Transformed γ -TiAl Phase Studied by High-resolution Electron Microscopy, *MRS Symp. Proc. Vol. 460*, p. 201 (1997).
- (5) T. Kumagai, E. Abe, M. Takeyama, M. Nakamura: Microstructural Evolution of Massively Transformed γ -TiAl during Isothermal Aging, *Scripta Mater.* **35**, 495 (1997).

- (6) T. Kumagai, E. Abe and M. Nakamura: Formation of Fine γ Grain Structure through Fine α_2/γ Lamellar Structure in Ti-rich TiAl Alloy, MRS Symp. Proc. vol. 460, 135 (1997).
- (7) E. Abe: Ultra-fine Lamellar Structure and Local Structure of α_2 Ordered Phase with Supersaturated Al, in *Ultra-fine world: Materials – Solid state physics based on microscopic observation*, (Japan Electron Microscopy Society), p 54 (1997)
- (8) E. Abe, M. Ohnuma and M. Nakamura: Structure of a New Phase Formed during Crystallization of Titanium-aluminide Amorphous Film, Proc. Int. Congress on Electron Microscopy-14, Vol II, p.77 (1998)
- (9) T. Kumagai, E. Abe and M. Nakamura: Microstructure Evolution through the $\alpha - \gamma$ Phase Transformation in a Ti-48at.%Al Alloy, Metall. Trans. A, 29A, 19 (1998).
- (10) E. Abe, M. Ohnuma and M. Nakamura: The Structure of a New Phase Formed during the Early Stage of Crystallization of Ti-48at.%Al Amorphous Film, Acta Mater. (1999) in press.
- (11) E. Abe, K. Niinobe, M. Nobuki, M. Nakamura and T. Tsujimoto: High-temperature Phase Transformation in Cr Added TiAl Base Alloy, MRS Symp. Proc. vol. 460, p. 201 (1999).
- (12) E. Abe, K. Niinobe, T. Kumagai, M. Nakamura and T. Tsujimoto; Transmission Electron Microscope Study of the Non-equilibrium $\alpha - \gamma$ Phase Transformation in Ti-Al Intermetallic Alloys, Proc. PTM'99 (1999) in press.
- (13) K. Kasahara, K. Hashimoto, T. Kimura, M. Nakamura, T. Tsujimoto: Influence of Aluminizing Treatment on Oxidation Resistance and on Room Temperature Ductility of TiAl Based Alloys, Zairyou-to-Kankyo, 47, 29 (1998)
- (14) K. Kasahara, K. Hashimoto, T. Kimura, M. Nakamura, T. Tsujimoto: Effects of Aluminizing on Tensile Property and High-Temperature Oxidation of TiAl-base Alloys, Corrosion Engineering 47, 23 (1998)
- (15) K. Niinobe, Y. Tomoda, T. Tsujimoto, M. Nobuki: Superplasticity in Titanium Aluminides Containing Chromium, Mater. Sci. Forum, vol. 304-306, p. 201 (1999)
- (16) Y. Yamamoto, K. Hashimoto, T. Kimura, H. Moriya, M. Nobuki, N. Khono: Solubility Limits of Additional Element, Zr, V, Ag, Ga in $L1_2-(AlMn)_3Ti$ Phase at 1450 K, J. Japan Inst. Metals, 62, 844 (1998) (in Japanese)
- (17) T. Kimura, Y. Yamamoto, K. Hashimoto, H. Moriya: Residual Strain in Powders of $L1_2-(AlMn)_3Ti$ Titanium Trialuminides with Additions of Zr, Ga, Ag or V, J. Japan Inst. Metals, 63, 174 (1999) (in Japanese)
- (18) K. Hashimoto, Y. Yamamoto, T. Kimura, H. Moriya, M. Nakamura: Effect of Vanadium Addition on Lattice Parameter and Distortion of $L1_2-(Al, X)_3Ti$ Phase, J.

- Japan Inst. Metals, **63**, 808 (1999) (in Japanese)
- (19) K. Hashimoto, Y. Yamamoto, T. Kimura, M. Nobuki: Effect of Vanadium on Residual Strain in $L1_2$ -Type $(AlMn)_3Ti(V)$ Alloy Powders and Bend Ductility of Pre-Milling Alloys, *Met. Trans., JIM*, **40**, 400 (1999)
 - (20) M. Nakamura, M. Nobuki, T. Tanabe, T. Kumagai, I. Mutoh, E. Abe: Microstructure Control and High Temperature Properties of TiAl Base Alloys, *Intermetallics*, **6**, 637-641 (1998).
 - (21) T. Kumagai, M. Nakamura: Microstructure and Environmental Embrittlement of TiAl Base Alloys, in *Report on Role of Hydrogen in Environmental Embrittlement of Structural Materials-I*, The Iron and Steels Institute of Japan, 1999, p. 92. (in Japanese)
 - (22) M. Nakamura: Intermetallic Compounds in *Material Science on High Temperature Strength*, ed. by K. Maruyama (Uchida Roukakuho, Tokyo 1997), p. 314.
 - (23) T.J. Sato, E. Abe, A. P. Tsai: A Novel Decagonal Quasicrystal in Zn-Mg-Dy System, *Jap. J. Appl. Phys.*, **36**, L1038 (1997).
 - (24) M. Ohnuma, K. Hono, E. Abe, H. Onodera, S. Mitani, H. Fujimori: Microstructure of Co-Al-O Granular Films, *J. Appl. Phys.*, **82**, 5646 (1997).
 - (25) E. Abe, T. J. Sato, A. P. Tsai: The Structure of Frank-Kasper Decagonal Quasicrystal in Zn-Mg-Dy System : A Comparative Study with Al-Ni-Co System, *Phil. Mag. Lett.*, **77**, 205 (1998).
 - (26) E. Abe, A. P. Tsai: High-resolution Electron Microscopy Study on Zn-Mg-Y Icosahedral Quasicrystal, *Proc. of Aperiodic 97'*, p. 235 (1998).
 - (27) E. Abe, T. J. Sato, A. P. Tsai: HRTEM Study of the Zn-Mg-rare-earth Decagonal Quasicrystal, *Int. Congress on Electron Microscopy-14*, Vol. III, p. 23 (1998).
 - (28) Alok Singh, E. Abe, A. P. Tsai: A Hexagonal Phase Related to Quasicrystalline Phases in Zn-Mg-rare Earth System, *Phil. Mag. Lett.*, **77**, 95 (1998).
 - (29) Alok Singh, E. Abe, A. P. Tsai: An Electron Diffraction Study of a Hexagonal Phase in a Zn-Mg-Sm alloy, *Int. Congress on Electron Microscopy-14*, Vol. III, p.61 (1998).
 - (30) Alok Singh, E. Abe, H. Takakura, A. P. Tsai: An Electron Microscopic Study of Hexagonal Phases in Zn-Mg-RE alloys, *MICRON*, submitted.
 - (31) T. J. Sato, E. Abe, A. P. Tsai, Composition and Stability of Decagonal Quasicrystal in Zn-Mg-RE System, *Phil. Mag. Lett.*, **77**, 213 (1998).
 - (32) T. Mochiku, M. Nakahara, E. Abe, T. Kamiyama, H. Asano, K. Hirata, F. Izumi: Crystal Structure of $Sr_{1.9}Nd_{1.1}Cu_{2.1}Nb_{0.9}O_8$ and $Sr_2(Nd_{0.75}Ce_{0.25})Cu_2NbO_{10}$: Ba_2YCu_3)+d - related Compounds with NbO_6 Octahedron, *Proc. of 10th Int. Symp. on Superconductivity* (1998) in press.

- (33) P.J. Steinhardt, H.-C. Jeong, K. Saitoh, M. Tanaka, E. Abe, A.P. Tsai: Experimental Verification of the Quasi-unit Cell Model of Quasicrystal Structure, *Nature* (London) **396**, 55 (1998).
- (34) E. Abe, A. Singh, H. Takakura, A. P. Tsai: Hexagonal Superstructures in Zn-Mg-Rare-earth Alloys, *J. Alloys. Compd.* **283**, 169 (1999).
- (35) E. Abe, T. J. Sato, A. P. Tsai: Structure of a Quasicrystal without Atomic Clusters, *Phys. Rev. Lett.* **82**, 5269 (1999).
- (36) E. Abe, A.P. Tsai: Quasicrystal-Crystal Transformation in Zn-Mg-rare-earth Alloys, *Phys. Rev. Lett.* (1999) in press.
- (37) E. Abe, K. Saitoh, H. Takakura, A.P.Tsai, P.J.Steinhardt, H.-C. Jeong: A Quasi-Unit Cell Model for Al-Ni-Co Ideal Quasicrystals Based on Clusters with Broken 10-fold Symmetry (1999) submitted to *Phys. Rev. Lett.*
- (38) E. Abe, A.P.Tsai: The Atomic Structure of the Zn-Mg-RE Quasicrystals Studied by HRTEM, *MRS Symp. Proc.* vol. 553, p.123 (1999).
- (39) E. Abe, A.P.Tsai: Structure of a Quasicrystal without Atomic Clusters, *Acta Crystallogr. A* (Proc. of IUCr'99), in press.
- (40) J.G. Guo, E. Abe, T.J. Sato, A.P. Tsai: Production of a Single Decagonal Quasicrystal in Al-Co-Cu system, *Jpn. J. Appl. Phys.* (1999) in press.
- (41) E. Abe, A.P. Tsai: Five Petal Flower of Metal – Why are quasi-crystals formed? *BOUNDARY* (1999) in press.

• **Oral Presentation:** 43

- (1) International conferences: 9
- (2) Domestic conferences: 34

• **Patent Application**

- (1) TiAl Intermetallic Compound Base Alloys and their Fabrication Methods:
Minoru Nobuki, Tokuzo Tsujimoto, Kouichi Niinobe and Akihide Akutu,
August 18, 1997, Application No. 1997 221657

• **Award:**

- (1) JIM (Japan Institute for Metals) “Shorei-sho” for Encouraging Young Scientist,
September, 1998, Eiji Abe

2.3.6 Researchers in the 3rd Research Group

Age and research fields of researchers in the 3rd Research Group (March 31, 1999)

Age of researcher	Number	Metallurgy	Physics	Chemistry	Others
60 ~	2	2	0	0	0
50 ~ 59	10	3	3	1	3
40 ~ 49	4	2	0	0	2
30 ~ 39	13	10	2	0	1
22 ~ 29	1	1	0	0	0
Summation	30	18	5	1	6

Others: Electric engineering, mechanical engineering

2.3.7 Research Budgets in the 3rd Research Group

Research budgets in the 3rd Research Group (yen)

Kinds of budgets	1997	1998
General Research	32,500,000	12,000,00
Special Coordination Funds for Promoting Science and Technology	28,133,000	40,171,000
Computational Materials Research	9,592,000	9,592,000
NEDO	45,600,000	28,800,000
Supplementary Budgets	0	47,914,000
Summation	115,825,000	138,477,000

2.4 4th Research Group (Energy Conversion Materials)

Kiyoshi Inoue (Supervising Researcher)

2.4.1 Research work

It is necessary for supporting a civilized lifestyle to convert efficiently one form of energy into another, useful, one; magnetic, chemical, thermal, kinetic, or light energies. The development of high-efficiency energy-conversion materials is expected to be effective solutions of environmental, resource, and energy problems in the 21st century. Following the 5th Long-term Plan, the R & D of energy conversion materials have been promoted by the 4th Research Group. We have made numerous efforts searching for new energy conversion materials, investigating their characteristics, and improving the conversion efficiency of conventional energy conversion materials. Examples of the main outcomes are shown as follows.

- (1) Addition of Al or Ga was found to improve the nonlinear optical properties of BaB₂O₄ crystal.
- (2) For stabilizing Nb₃Al multifilamentary superconducting wire, which has 3-5 times larger J_c (critical current density) than those of the commercial superconductors, Cu-ion plating was found to be very effective.
- (3) We have developed Nb₃(Al, Ge) multifilamentary wire showing J_c (4.2 K, 25T) of 150 A/mm², which is the highest J_c value of multifilamentary superconductors reported to date.
- (4) We have developed a reactive ion-etching process for fabricating the nanostructures of ferromagnetic materials.
- (5) Some V alloys were found to have a higher hydrogen permeability than that of commercially-used Pd-Ag alloys.
- (6) The effective maximum power P_{\max} of p-type AgSbTe₂ was improved by 2nd phase precipitation, four times as large as that of AgSbTe₂ without precipitation.
- (7) The P_{\max} of joined n-type PbTe with different carrier concentrations was 20% larger than that of homogeneous PbTe.

The members of 4th Research Group, as at March 31, 1999, consisted of 22 researchers, 4 visiting researchers, 7 guest researchers, and 4 graduate students.

The output of the research activities has been relatively high since the start of the 5th Long-term Plan. The number of publications, including submitted papers, patents submitted, and awards received were 110, 18, and 5, respectively, between

April 1, 1977 to March 31, 1999. The details are shown in the research activities of each Subgroup. Because the many elder researchers in 4th Research Group will retire in the near future, recruitment of young researchers will become urgently necessary.

The 4th Research Group is composed of Subgroups, which have the following objectives.

(1) 1st Subgroup:

Nonlinear optical crystals for laser wavelength modulation and superconductors for micro-SMES (Superconducting Magnetic Energy Storage).

(2) 2nd Subgroup:

Shape memory alloys with gradient composition, wear proof materials with low friction, diffusion phenomena in intermetallic compounds, and surface modifications through plasma-source ion implantation.

(3) 3rd Subgroup:

Ferromagnetic materials with nano-scale controlled structures.

(4) 4th Subgroup:

Hydrogen storage alloys based on Mg and other light metals.

(5) 5th Subgroup:

Thermoelectric semiconductors with heterogeneous structure.

2.4.2 1st Subgroup

Hideo Kimura (Subgroup Leader), Yuji Yoshida, Akimitsu Miyazaki, Yasuo Iijima, Akihiro Kikuchi, Xiaopeng Jia (visiting researcher), Tsuguo Fukuda (guest researcher), Takatomo Sasaki (guest researcher), Tooru Katsumata (guest researcher), Mitsunori Sato (guest researcher), Kiyoshi Inoue (Supervising Researcher)

2.4.2.1 Research work

In this subgroup, nonlinear optical crystals for photo-frequency modulation and superconducting materials for micro-SMES (Superconducting Magnetic Energy Storage) have been investigated as follows.

- (1) Single crystals for frequency modulation devices are required to have crystal asymmetry. An effective method to improve the asymmetry is by substitution of a large element for a small element in the surroundings of a pair of large and small elements. In this study, these single crystals (BaB_2O_4 -based crystals), which have

crystal asymmetry, have been grown by the floating-zone-pulling-down method and characterized.

- (2) For realizing micro-SMES, we have been developing stabilized superconducting cables (Nb_3Al , $\text{Nb}_3[\text{Al}, \text{Ge}]$ cables) with high J_c in high fields, current lead superconducting materials (Y123, Tl1223 films) showing both high J_c at 77 K and poor thermal conductivity at 4.2-77 K, and superconducting vessels (Bi2223 thick films) for magnetic shielding.

<Research themes>

- (1) Search of New Nonlinear Optical Crystals for Wavelength Modulation, Single Crystal Growth and Principle Technology Development for Optical Devices, (General Research: April 1997 to March 2002, Hideo Kimura)
- (2) Investigation of Wavelength Modulation on Nonlinear Optical Oxides by Atomic Distribution Control, (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 1999, Hideo Kimura)
- (3) Development of Basic Superconducting Technology for High Field Micro-SMES, (General Research: April 1997 to March 2002, Kiyoshi Inoue)
- (4) Development of Nb_3Al Multifilamentary Superconductor (Special Coordinated Funds for Promoting Science and Technology: April 1998 to March 1999, Kiyoshi Inoue)
- (5) Development of Essential Technique for Improving Coil Current Density in Superconducting Magnet, (Special Coordinated Fund for Promoting Science and Technology: April 1998 to March 2001, Kiyoshi Inoue)

2.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) H. Kimura, T. Numazawa, M. Sato: Melt Supercooling Behavior and Crystal Growth of $\text{Ba}(\text{B}_{1-x}\text{M}_x)_2\text{O}_4$ (M: Al or Ga), *J. Crystal Growth* **174**, 308 (1997)
- (2) H. Kimura, M. Sato, K. Shimamura, T. Fukuda: Viscosity and Surface Tension Change in BaB_2O_4 Melt by Substitution of Al or Ga for B, *J. Mater. Sci. Lett.* **16**, 911 (1997)
- (3) H. Kimura, K. Ishioka, M. Sato: Estimation of SHG Properties on $\text{Ba}(\text{B}_{1-x}\text{M}_x)_2\text{O}_4$ (M: Al or Ga), *J. Mater. Sci. Lett.* **16**, 1375 (1997)
- (4) H. Kimura, T. Numazawa, M. Sato, T. Ikeya, T. Fukuda, K. Fujioka: Single Crystals of RAlO_3 (R: Dy, Ho and Er) for Use in Magnetic Refrigeration Between 4.2 and 20 K, *J. Mater. Sci* **32**, 5743 (1997)

- (5) H. Kimura, K. Ishioka, M. Sato: Advantages of Substitution of M (M: Al or Ga) for B on BaB_2O_4 , Proc. Inter. Symp. on Laser and Nonlinear Optical Materials '97, Singapore November 3-5, 162, 1997
- (6) T. Katsumata, R. Sakai, Y. Ohshiba, S. Komuro, T. Morikawa, H. Kimura: Growth and Characteristics of Long Persistent Phosphor Crystals, Proc. Inter. Symp. on Laser and Nonlinear Optical Materials '97, Singapore November 3-5, 337, 1997
- (7) H. Kimura, M. Sato, Y. Terada, K. Shimamura, T. Fukuda, S. Miyashita: Single Crystals of $(\text{Dy}_{1-x}\text{Gd}_x)\text{VO}_4$ for Magnetic Applications, J. Mater. Sci. **33**, 2379 (1998)
- (8) T. Katsumata, R. Sakai, Y. Ohshiba, Y. Isoda, S. Komuro, T. Morikawa, H. Kimura: Growth and Characteristics of Long Duration Phosphor Crystals, J. Crystal Growth **198/199**, 869 (1999)
- (9) A. Miyazaki, H. Kimura, X. Jia, K. Shimamura, T. Fukuda: Growth of Small Diameter $\text{Ba}(\text{B}_{1-x}\text{Al}_x)_2\text{O}_4$ Single Crystals, Cryst. Res. Technol. **34**, 817 (1999)
- (10) H. Kimura, X. Jia, K. Shoji, R. Sakai, T. Katsumata: Crystal Growth of $\text{Ba}(\text{B}_{1-x}\text{Al}_x)_2\text{O}_4$ Using a New Double Ring Halogen Lamp Heater Fz Furnace, submitting to J. Crystal Growth
- (11) Y. Iijima, M. Kosuge, T. Takeuchi, K. Inoue: Critical Current Density Characteristics of Nb_3Al Multifilamentary Wires Continuously Fabricated by Rapid-Quenching, Proc. ICEC16/ICMC, Kitakyushu-Shi 1695 (1997)
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- (17) K. Nakagawa, K. Fukuda, G. Iwai, H. Moriai, Y. Iijima, T. Takeuchi, K. Inoue, N. Kobayashi: Development of Nb₃Al Superconducting Wires Fabricated by Rapid-Quenching and Transforming Process, Proc. MT-15, 977 (1998)
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 - (21) A. Ichinose, A. Kikuchi, K. Tachikawa, S. Akita: Deposition of Y₂O₃ Buffer Layers on Biaxially-Textured Metal Substrate, Physica C 302, 51 (1998)
 - (22) Y. Iijima, A. Kikuchi, K. Inoue, and T. Takeuchi: Effect of Additional Elements to Nb₃Al Multifilamentary Wire Fabricated by the Rapid-Heating Process, to be published in IEEE Trans. on Supercond. (1999)
 - (23) A. Ichinose, C-Y. Yang, D. C. Labalestier, S. E. Badcock, A. Kikuchi, K. Tachikawa, S. Akita: YBCO Films Grown on Y₂O₃-Buffered Cube-Textured Ni Substrate, to be published in Advances Cryogenic Engineering, (1999)
 - (24) A. Kikuchi, K. Inoue, K. Tachikawa: Structure and Irreversibility Field of Tl-1223 phase Synthesized through the Substitution of TlF for Tl₂O₃, Physica, C, (1999), in press
 - (25) A. Ichinose, G. Daniels, N. Heing, D. C. Larbalestier, A. Kikuchi, K. Tachikawa, S. Akita: Preparation and Characterization of Y₂O₃ Buffer Layer and YBCO Films on Textured Ni Tape, IEEE Trans. on Appl. Supercond. (1999), in press
 - (26) S. Akita, A. Ichinose, A. Kikuchi, K. Tachikawa, D. C. Larbalestier, G. Daniels, N. Heing: Characterization of Y₂O₃ Buffer Layers and YBCO Films on Textured Ni Tape, Trans. IEEE of Japan, in press

• Oral Presentation: 46

- (1) International Conference: 13
- (2) Domestic Conference: 33

• Patent

- (1) BaM₂O₄ Oxide Single Crystal Having Non-Linear Optical Property and Manufacturing Method Thereof: H. Kimura, M. Sato, USA Patent, 08.01.97, USA Patent Application 08/904.610
- (2) Fabrication Method of Bulk Single Crystals: H. Kimura, T. Katsumata, R. Sakai, JP Patent 09.22.97, Patent Application 09-257087
- (3) Wavelength Conversion Materials and Their Fabrication Method: H. Kimura, T. Katsumata, R. Sakai, JP Patent 01.28.98, JP Patent Application 10-015214, 01.22.99, JP Patent Registration 2876527
- (4) An Amorphous Substance for Wavelength Conversion and a Making Process of the Same: H. Kimura, R. Sakai, T. Katsumata, USA Patent, 01.26.99, USA Patent Application 09/236.610
- (5) Optical Waveguide and Its Fabrication Method: H. Kimura, A. Miyazaki, JP Patent 02.01.99, JP Patent Application 11-024287
- (6) Nb₃Al Superconducting Compound Wire and Its Fabrication Process: A. Kikuchi, K. Inoue, M. Fukutomi, G. Iwaki, K. Nakagawa, JP Patent Application, 10-262727, 10.09.17
- (7) Nb₃Al Superconducting Compound Wire and Its Fabrication Process: T. Takao, T. Kiyoshi, K. Itoh. H. Wada, Y. Iijima, K. Inoue, JP Patent Application, 10-287491, 10.10.09
- (8) Nb₃Al Superconducting Compound Wire and Its Fabrication Process: T. Takao, T. Kiyoshi, K. Itoh. H. Wada, Y. Iijima, K. Inoue, JP Patent Application, 10-287492, 10.10.09
- (9) Eutectic Two-Phase Alloys with Ductility and Their Fabrication Process: K. Inoue, Y. Yoshida, Y. Iijima, A. Sai, JP Patent Application, 11-059907, 11.03.08
- (10) Fabrication Process of Nb₃(Al, Ge) and Nb₃(Al, Si) Multifilamentary Superconducting Wires: A. Kikuchi, Y. Iijima, K. Inoue, JP Patent Application, 11-158826, 11.06.04

Awards:

- (1) Fabrication Process of V₃Si Multifilamentary Wire: K. Inoue, Noteworthy Invention, April 14, 1997.
- (2) Development of Extremely High Field Magnets: K. Inoue, Persons of Scientific and Technological Research Merit, April 13, 1999.
- (3) Improvement of Cold-Drawing Process: Y. Iijima, Commendation for Achievements, May 19, 1999.

2.4.3. 2nd Subgroup

Hisaoki Sasano (Subgroup Leader), Shigeaki Uehara, Hitoshi Shinno, Susumu Ikeno, Masatsugu Kaise, Masahiko Kato (graduate student)

2.4.3.1 Research work

The research subjects of this subgroup are concerned with the following four fields.

- (1) The composition dependency of diffusivity in NiAl, Ti₃Al and TiAl have been investigated by using solid-AlCl₃ gas diffusion couple.
- (2) In order to obtain shape memory alloys with recovery temperature, changing along the longitudinal direction, we have investigated fabricating composition-graded shape memory alloys of TiNi and Cu-Zn-Al by a chemical transportation technique.
- (3) The wear properties of TiAl composites containing ceramics, such as AlN, TiB₂ and SiC have been studied for high temperature use.
- (4) A new surface modification apparatus (plasma source ion implantation), in which specimens are immersed in plasma and implanted by a high negative pulse potential applied to the specimens, has been fabricated. By using the apparatus, a large complex-shaped specimen can be treated with at lower cost than the conventional ion beam ion implantation.

<Research themes>

- (1) Formation of Functionally Graded Shape Memory Alloys and Others
(General Research: April 1997 to March 1999, Hisaoki Sasano)
- (2) Research for Creation of New Functions of Thin Films by Annealing in Magnetic Field
(General Research: April 1995 to March 1998, Hitoshi Shinno)
- (3) Research for Surface Modification of Materials by Plasma Source Ion Implantation
(General Research: April 1998 to March 2001, Hitoshi Shinno)

2.4.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

• Publication

- (1) M. Kaise, K. Saito : Synthesis of Superconducting BiSrCaCuO Ultrathin Films of Thicknesses below 10 nm, J. Jpn. Inst. Metals, 61, 873 (1997) (in Japanese)
- (2) H. Shinno, M. Uehara, K. Saito: Synthesis of α' -Fe₁₆N₂ Iron Nitride by Means of Nitrogen Ion Implantation into Iron Thin Films, J. Mat. Sci., 32, 2255 (1997)
- (3) H. Shinno, K. Saito: Synthesis of Fe₁₆N₂ by Means of Nitrogen Ion Implantation into Sputter Deposited Fe Thin Films, Proc. 4th Inter. Symp. on Sputtering &

Plasma Processes, Kanazawa, June, 1997, pp.105-109.

- (4) M. Kaise, K. Saito : Synthesis of Superconducting BiSrCaCuO Ultrathin Films by Ion Implantation, Proc. 13th Symp. on Surface Layer Modification by Ion Implantation, 13, Tokyo November 21, 1997
- (5) H. Shinno, K. Saito: Effects of Film Thickness and Low Substrate Temperature on Formation Processes of Fe_{16}N_2 during Nitrogen Ion Implantation into Fe Films, Proc. 13th Symp. on Surface Layer Modification by Ion Implantation, Tokyo, November, 1997, pp.9-15.
- (6) M. Kato, H. Sasano, T. Suzuki : Control of Aluminum Concentration on Titanium Surface by Means of Chemical Transportation Technique, Mater. Sci. Eng., A243, 212(1998).
- (7) M. Kato, H. Sasano, K. Honma, T. Suzuki : Single-Phase Interdiffusion in Intermetallic Compound NiAl, J. Japan Inst. Metals, 62, 761(1998). (in Japanese)
- (8) M. Kato, H. Sasano, T. Suzuki: Coating of Ni-Al alloys having Various Compositions on Ni_3Al and Ni by Means of Chemical Transportation Technique, 11th Congress of Inter. Fed. for Heat Treatment and Surface Engineering, Vol.2, 53. Florence July 7-11, 1998.
- (9) S. Ikeno, K. Hashimoto : Wear Properties of Surface-Oxidized TiAl-Sb, 5th Inter. Symp.on Functionally Graded Materials 295, Dresden October 26-29, 1998.
- (10) K. Saito, M. Kaise: Superconductivity and Structure of a Few Unit-Cells Thick Bi-Sr-Ca-Cu-O Ultrathin Films, Phys. Rev. B 57, 11786(1998)
- (11) H. Shinno, K. Saito: Effects of Film Thickness on Formation Processes of Fe_{16}N_2 in Nitrogen Ion-Implanted Fe Films, Surface and Coatings Technology, 103-104, 129 (1998)
- (12) M. Kato, H. Sasano, T. Suzuki : Single-Phase Interdiffusion in Intermetallic Compound Ti_3Al , J. Japan Inst. Metals, 63, 656(1999). (in Japanese)
- (13) M. Kato, H. Sasano, T. Suzuki : Phase Separation in Nearly Stoichiometric NiAl, J. Japan Inst. Metals, 63, 549(1999). (in Japanese)
- (14) M. Kaise: Synthesis of Superconducting BiSrCaCuO Ultrathin Films, J. Jpn. Chemical Industry, 50, 30(1999) (in Japanese)

• Oral presentation:13

- (1) International Conference: 4
- (2) Domestic Conference: 9

2.4.4 3rd Subgroup

Isao Nakatani (Subgroup Leader), Takao Furubayashi, Hiroaki Mamiya, Takeshi Kikuchi

2.4.4.1 Research work

The objective of the research work in this subgroup is to explore basic research, technology, and the applications of novel magnetic materials with mesoscopic sizes, which include three types of magnetic materials with mesoscopic sizes of dimensions around 100nm, 10nm and 1nm regime, respectively. This work has two main components.

- (1) First, there is the exploration of fabrication methods for mesoscopic magnetic materials. These are electron-beam nanolithography to fabricate nanostructures of ferromagnetic substances with dimensions in several hundreds nm or sub-100nm, syntheses of magnetic colloids (magnetic fluids) of ferromagnetic ion-nitride particles, 100nm or sub-10nm in diameter, dispersed in liquids of mineral oils, and preparations of nanogranular magnetic materials with ferromagnetic fine particles, several nm or 1nm in size, embedded in solid matrix of insulator.
- (2) Second, there is the challenge that one can explore mesoscopic magnetic phenomena, from the classical to the quantum limit. Although its roots are in the quantum description of solids, the new fabrication techniques for sophisticated materials described above allow widespread studies of the phenomenon and development of device applications.

<Research themes>

- (1) Studies on Micro-Fabrications and Developments for Advances Magnetic Materials (Special Coordination Funds for Promoting Science and Technology : April 1996 to March 998 (Period II) , Isao Nakatani)
- (2) Research on Quantum Magnetic Properties and Spinic Functions of Mesoscopic Magnetic Materials (Special Research: From April 1996 to March 31, 2003, Isao Nakatani)

2.4.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) H. Mamiya, M. Onoda, T. Furubayashi, J. Tang, I. Nakatani: Structural and Magnetic Studies on Vanadium Spinels MgV_2O_4 , J. Appl. Phys. 81, 5289 (1997)
- (2) H. Mamiya, I. Nakatani: Effects of Cooling Field on Magnetic Relaxation for an

- Iron-Nitride Fine Particle System, J. Appl. Phys. 81, 4733 (1997).
- (3) Nakatani: Fabrications of Microstructures of Magnetic Materials, J. Magn. Soc. Jpn. 22,1383(1998).
 - (4) H. Mamiya, I. Nakatani: Magnetization Curve for Iron-Nitride Fine Particle System with Random Anisotropy, IEEE Trans. Magn. 34, 1126 (1998).
 - (5) H. Mamiya, I. Nakatani, T. Furubayashi: Blocking and Freezing of Magnetic Moments for Iron-Nitride Fine Particle Systems, Phys. Rev. Lett, 80, 177 (1998).
 - (6) H. Mamiya, I. Nakatani: Dynamic Study of an Iron-Nitride Fine Particle System, - Field Dependence of the Blocking Temperature -, J. Magn. Magn. Mater, 177-181, 966 (1998).
 - (7) T. Taniyama and I.Nakatani: Magnetic Relaxation in $\text{Ga}_{0.6}\text{Mo}_2\text{S}_4$ Spinel, J. Appl. Phys. 83,1 (1998).
 - (8) T. Taniyama and I.Nakatani: Nonlinear Susceptibility of Ferromagnetic $\text{Ga}_{0.6}\text{Mo}_2\text{S}_4$ Spinel, J. Magn. Magn. Mater. 177-181, 263 (1998).
 - (9) T. Furubayashi, I. Nakatani: Superparamagnetism of Granular Fe-MgF₂ Films, IEEE Trans. Mag. 34, 1117 (1998)
 - (10) T.Taniyama, I.Nakatani, H.Yanagihara, and E.Kita: Magnetoresistance of Zigzag-Shaped Cobalt Wires, J. Magn. Magn. Mater.196-197, 77 (1999).
 - (11) T. Taniyama, I.Nakatani, T.Mamikawa, and Y.Yamazaki: Resistivity due to Domain Walls in Co Zigzag Wires, Phy. Rev. Lett. 82, 2780 (1999).
 - (12) H. Mamiya I. Nakatani, T. Furubayashi: Slow Dynamics for Spin-Glass-Like Phase of a Ferromagnetic Fine Particle System, Phys. Rev. Lett. 82, 4332 (1999).
 - (13) H. Mamiya, I. Nakatani: Phase Diagram of an Iron-Nitride Magnetic Fluid: Effects of temperature and weak magnetic field, IEEE Trans. Magn., submitted.
 - (14) H. Mamiya, I. Nakatani: Critical Phenomena of an Iron-Nitride Fine Particle System, Nanostructured Materials, in press.
 - (15) T. Kikuchi, S. Kajiware, H. Pal, K. Inoue, T. Asano, M. Kosuge, M. Yuyama, H. Wada: Athermal and Isothermal Martensitic Transformation at Room Temperature Induced by Ultra High Magnetic Field, Proc. 4th ESMT (the Netherlands, 1997)

• Oral Presentation:69

- (1) International Conference: 17
- (2) Domestic Conference: 52

• **Patent Application:**

- (1) Reactive-Ion Etching Method, Isao Nakatani, Jul. 25, 1997, JP Patent 2677321.
- (2) Inductor and Transformer, Isao Nakatani, Masayuki Hijikata, Masaru Saito, Aug.15, 1997, JP Patent 2556461.
- (1) Reactive-Ion Etching Apparatus, Isao Nakatani, Sept.22, 1997, JP Patent Application 09-256635.
- (2) Reactive-Ion Etching Mask, Isao Nakatani, Sept.22, 1997, JP Patent Application 09-256636.
- (3) Reactive-Ion Etching Method, Isao Nakatani, Dec.2, 1998, JP Patent Application 10-343287.
- (4) Magnetic Damascene Structure, Isao Nakatani, Mar.29, 1999, JP Patent Application 11-86118.
- (5) Method for Reactive-Ion Etching and Apparatus therefor, Isao Nakatani, Sept.21,1998, US Patent Application 09/157421.
- (6) Method for Reactive-Ion Etching and Apparatus therefor, Isao Nakatani, Sept.21,1998, Korea Patent Application 38946.
- (7) Method for Reactive-Ion Etching and Apparatus therefor, Isao Nakatani, Sept.21,1998, UK Patent Application 9820639.4.

• **Award:**

- (1) Reactive-Ion Etching Method: Isao Nakatani, Noteworthy Invention, April 13, 1998

2.4.5 4th Subgroup

Chikashi Nishimura (Subgroup Leader), Hideo Numata, Masao Komaki, Isao Tomizuka (guest researcher), Masayuki Itagaki (guest researcher), Yoshihiro Momose (guest researcher)

2.4.5.1 Research Work

In order to obtain the guidelines for developing high-performance light alloys for hydrogen storage, hydrogen diffusion, hydrogen trapping, hydrogen absorption, and hydrogen behaviors in Mg-based and V-based alloys have been investigated by means of a hydrogen permeation technique, thermal desorption spectroscopy (TDS), and pressure-composition-isotherms (PCT) measurements. We found that some V alloys show higher hydrogen permeability than that of commercially-used Pd-Ag alloys.

<Research theme>

- (1) Research and Development of High-Performance Light Alloys for Hydrogen Storage
(General Research: April 1997 to March 2001, Chikashi Nishimura)

2.4.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) M. Komaki, M. Amano, H. Numata, C. Nishimura: Effects of Oxidation of Mg-Ni-H and Mg-Ni-Al-H Alloys on Hydrogen-Thermal-Desorption Characteristics, J. Japan Inst. Metals **62**, 111(1998) (in Japanese)
- (2) H. Numata, M. Amano, I. Tomizuka, T. Tsujimoto: Thermal Hydrogen Desorption Spectroscopy of Hydrides Formed in the Surface Layer of an Electrochemically Treated TiAl Based Alloy, J. Japan Inst. Metals **61**, 105(1997) (in Japanese)
- (3) H. Numata, T. Tsujimoto: Hydride Formation on Ti₃Al by Cathodic Hydrogen Charging in Sulfuric Acid Containing Sb₂O₃, J. Japan Inst. Metals **63**, 561 (1999). (in Japanese)
- (4) C. Nishimura, M. Komaki, M. Amano: Hydrogen Permeation through Magnesium, J. Alloys and Compounds (accepted)

Oral Presentation: 10

- (1) International Conference: 1
- (2) Domestic Conference: 9

Award:

- (1) Invention of a Versatile Sample-Holder in the Hydrogen Permeation Measurements
Equipment: Masao Komaki, Persons who have proposed technical ideas in relation to their job, STA, April 13, 1998.

2.4.6 5th Subgroup

Isao A. Nishida (Subgroup Leader), Yoshio Imai, Yukihiro Isoda, Yoshikazu Shinohara, Masafumi Miyajima (visiting researcher), Yasuo Nakahara (visiting researcher), Kenji Taguchi (visiting researcher), Seiji Yoneda (graduate student), Masayasu Hashimoto (graduate student), Nobuhiro Yuhashi (graduate student)

2.4.6.1 Research work

Thermoelectric materials, such as Bi_2Te_3 , PbTe , AgSbTe_2 and FeSi_2 , have been investigated in this subgroup. We have been improving the thermoelectric performances related with precipitation of the second phases, the addition of rare earth elements, and the joining conditions of segments.

Thermoelectric properties of the unidirectionally solidified p-type AgSbTe_2 were found to be affected directly by precipitation of high temperature second phase. The effective maximum power P_{\max} of AgSbTe_2 was improved by second phase precipitation, and was four times as large as that of AgSbTe_2 without the precipitation. Furthermore, the P_{\max} of joined n-type PbTe with different carrier concentrations was 20% larger than that of a homogeneous PbTe . There is a high possibility to realize a superior thermoelectric semiconductor through controlling the structure and composition.

<Research themes>

(1) Energy Conversion Materials Fabricated with Composite Structure

(General Research: April 1997 to March 2002, Isao A. Nishida)

(2) Development of Energy Conversion Materials with Formation of Graded Structure

(Special Coordination Funds for Promoting Science and Technology: April 1993 to March 1998, Isao A. Nishida)

2.4.6.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) Y. Noda, M. Orihashi, I. A. Nishida: Thermal and Electrical of p-type Lead Telluride Doped with Ag or K, J. Japan Inst. Metals, **61**,180(1997). (in Japanese)
- (2) M. Orihashi, Y. Noda, H. T. Kaibe, I. A. Nishida: Thermoelectric Properties of Impurity-Doped PbTe , J. Japan Inst. Metals, **61**,214(1997). (in Japanese)
- (3) Y. Shinohara, Y. Imai, Y. Isoda, I. A. Nishida: Study of Electrical Power Properties for FGM, Proc. Functionally Graded Material in 1997 (FGM'97), Tokyo, 117 (1997).
- (4) E. Koshigoe, K. Kudou, M. Hashimoto, I. Shiota, I. A. Nishida: FGM of Low Temperature Thermoelectric Materials, Proc. FGM '97, Tokyo, 129(1997).
- (5) Y. Isoda, Y. Shinohara, Y. Imai, I. A. Nishida, M. Hashimoto, H. T. Kaibe, O. Ohashi: Thermoelectric Properties of Joined PbSnTe Systems with Different Carrier Concentrations, Proc. FGM '97, Tokyo, 135(1997).
- (6) S. Yoneda, E. Ohota, Y. Imai, Y. Shinohara, Y. Isoda, T. Kimura, I. A. Nishida, H. T. Kaibe: Thermoelectric Properties of AgSbTe_2 System Compound, Proc. FGM '97,

- Tokyo, 159(1997).
- (7) Y. Noda, I. A. Nishida, H. S. Kang, M. Niino: Preparation and Thermoelectric Properties of *p*-type Thermoelectric Material in the Temperature Range, Proc. FGM '97, Tokyo, 165(1997).
 - (8) M. Hashimoto, I. Shiota, O. Ohashi, H. T. Kaibe, Y. Imai, Y. Shinohara, I. A. Nishida: Liquid Phase Bonding of PbTe System Thermoelectric Materials, Proc. FGM '97, Tokyo, 171(1997).
 - (9) M. Kurosaki, M. Hashimoto, I. Shiota, I. A. Nishida: Relationship between Thermoelectric Properties and Preparations of *n*-type PbTe, Proc. FGM '97, Tokyo, 177(1997).
 - (10) Y. Imai, Y. Shinohara, Y. Isoda, I. A. Nishida: Thermoelectric Properties of Joined PbTe with Different Carrier Concentrations, Proc. FGM '97, Tokyo, 183(1997).
 - (11) Y. Imai, Y. Shinohara, Y. Isoda, I. A. Nishida, H. T. Kaibe, S. Yoneda, I. Shiota: Study of FGM for $\text{Pb}_{1-x}\text{Sn}_x\text{Te}$, Proc. Symp. Thermoelectric Energy Conv. in 1997(TEC'97), Tokyo, 28(1997).
 - (12) S. Yoneda, E. Ohota, H. T. Kaibe, T. Okumura, I. Shiota, Y. Imai, Y. Shinohara, Y. Isoda, I. A. Nishida: Thermal Hysteresis Control of *n*-type PbTe, Proc. TEC'97, Tokyo, 32 (1997).
 - (13) I. J. Ohosugi, T. Kojima, H. T. Kaibe, M. Sakata, I. A. Nishida: Consideration on the Thermoelectric Performance of Thermoelectric Semiconductor, Proc. FGM '97, Tokyo, 44(1997).
 - (14) Y. Shinohara, Y. Imai, Y. Isoda, I. A. Nishida: Effect on Peltier and Joule Heats to Resistivity Measurement, Proc. TEC '97, Tokyo, 48(1997).
 - (15) Y. Isoda, O. Ohashi, I. A. Nishida: Refractory and Thermal Shock Resistance of Iron Disilicide with Boron, Proc. TEC '97, Tokyo, 68(1997).
 - (16) M. Hashimoto, I. Shiota, O. Ohashi, S. Yoneda, H. T. Kaibe, Y. Imai, Y. Shinohara, Y. Isoda, I. A. Nishida: Liquid Phase Bonding of *p*-type $\text{Pb}_{1-x}\text{Sn}_x\text{Te}$, Proc. TEC '97, Tokyo, 92(1997).
 - (17) H. T. Kaibe, T. Okumura, S. Yoneda, T. Miyamoto, I. Shiota, Y. Isoda, I. A. Nishida: Thermoelectric Properties of *n*-type PbTe by Evaporation Method, Proc. TEC '97, Tokyo, 98(1997).
 - (18) S. Yoneda, T. Miyamoto, H. T. Kaibe, E. Ohota, I. Shiota, Y. Isoda, I. A. Nishida: Effect on Crystal Grain Size to Thermoelectric Properties of Sintered PbTe by Spark Plasma Sintering Technique, Proc. TEC '97, Tokyo, 100(1997).
 - (19) I. A. Nishida: Thermal Physics on Thermoelectric Conversion Technique, J. Thermal Phys. 11, 95(1997).

- (20) I. A. Nishida: Thermoelectric Materials, Bull. Jpn. Inst. Metals Metar., **36**, 958 (1997).
- (21) I. A. Nishida: Preparation of Thermoelectric Materials, Ceramics, **33**, 176(1998).
- (22) Y. Noda, M. Orihashi, I. A. Nishida: Thermoelectric Properties of *p*-type Lead Telluride Doped with Silver or Potassium, Materials Trans. JIM, **39**, 602(1998).
- (23) M. Orihashi, Y. Noda, H. T. Kaibe, I. A. Nishida: Evaluation of Thermoelectric Properties of Impurity-Doped PbTe, Materials Trans. JIM, **39**, 672(1998).
- (24) S. Yoneda, E. Ohta, H. T. Kaibe, I. Shiota, K. Takahashi, Y. Imai, I. A. Nishida: Crystal Grain Size Dependence of Thermoelectric Properties for Sintered PbTe by Spark Plasma Sintering Technique, Proc. 16th ICT, Dresden, 247(1997).
- (25) H. T. Kaibe, Y. Noda, Y. Isoda, I. A. Nishida: Temperature Dependence of Thermal Conductivity for $\text{Mg}_2\text{Si}_{1-x}\text{Ge}_x$, Proc. 16th ICT, Dresden, 279(1997).
- (26) I. Shiota, I. A. Nishida: Development of FGM Thermoelectric Materials in Japan - The Sate of the Art-, Proc. 16th ICT, Dresden, 364(1997).
- (27) Y. Shinohara, Y. Imai, Y. Isoda, I. A. Nishida, H. T. Kaibe, I. Shiota: Thermoelectric Propertied of Segmented Pb-Te Systems with Graded Carrier Concentrations, Proc. 16th ICT, Dresden, 386(1997).
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Oral Presentation: 49

- (1) International Conference: 19
- (2) Domestic Conference: 30

2.4.7 Researchers in the 4th Research Group

Age and research fields of researchers in the 4th Research Group. (March 31, 1999)

Age of Researchers	Number	Metal-lurgy	Solid-state Physics	Crystal Growth	Cryogen-ics	Chemis-try
60 ~	4	3	0	0	0	1
50 ~ 60	12	3	3	2	2	2
40 ~ 49	10	3	3	3	1	0
30 ~ 39	5	0	4	1	0	0
22 ~ 29	5	0	4	0	1	0
Summation	36	9	14	6	4	3

2.4.8 Research Budgets in the 4th Research Group

Research budgets in the 4th Research Group. (yen)

Kinds of budgets	1997	1998
Special Research	11,917,000	13,433,000
General Research	40,000,000	37,420,000
Special Coordination Funds for Promoting Science and Technology	8,053,000	17,885,000
Collaboration with Hiroshima Pre.	4,000,000	0
Supplementary Budgets	0	68,300,000
Summation	63,970,000	137,038,000

2.5 5th Research Group (Intelligent Materials)

Norio Shinya (Supervising Researcher)

2.5.1 Research Work

Intelligent materials are new materials with multiple and intelligent functions based on a new concept. The multiple and intelligent functions materials are necessary to develop highly integrated electronics and machines, and to improve the reliability of structures. In order to realize materials with innovative functions, many approaches are being tried.

According to the 5th Long-term Plan, the research work in the 5th Research Group is focussed on development of key technologies for assembling powder particles and integrating functions, actuators for micro-machines and structures, and functional structural materials for aerospace and high temperature use. The main research aims of the 5th Research Group are as follows.

- (1) Development of ① technology for assembling powder particles, ② powder particle composites, ③ periodical structures by charged beams and ④ three-dimensional powder particle arrays by micro-probe manipulation.
- (2) Creation of ① photonic band gap materials and multiple functional materials, ② photonic band gap materials by three-dimensional fabrication techniques and ③ flexible PTCR, PTC-NTC materials.
- (3) Development of ① shape memory alloys (SMA) for actuators, Ti-Ni SMA thin films and ② training-free Iron based SMA.
- (4) Development of ① self-healing and light weight structural materials, ② self-healing of creep voids in heat resisting alloys and ③ metallic closed cellular materials.

Development of a charged powder particle beam apparatus and evaluation of photonic band gap materials are carried out jointly with APCO Ltd. and the University of Tokyo, respectively.

The members of the 5th Research Group consist of 13 researchers, 1 STA fellow, 1 domestic research fellow, 1 technician, 1 secretary and 5 guest researchers.

The number of publications, including submitted papers, are about 80, patents submitted are 12 between April 1, 1997 to March 31, 1999. The details are shown in the research activities of each Laboratory.

2.5.2 1st Subgroup

Mikihiko Kobayashi (Subgroup Leader), Takehiro Dan, Junro Kyono, Satoshi Kishimoto, Mitsuru Egashira, Masashi Hase, Takeshi Konno, Hiroshi Fudouzi, Hideki Miyazaki (domestic research fellow), Liu Daozhi (STA fellow), Setsuo Kajiware (guest researcher), Makoto Kuwabara (guest researcher), Kyoko Saito (technician), Yumiko Asaka (secretary)

2.5.2.1 Research work

- (1) Four kinds of handling techniques are developed to place accurately particles at prescribed positions. The first one is a method to produce ordered mixtures by mixing two kinds of particles, which are electrified positively and negatively. Packing of the ordered mixtures of semiconducting BaTiO_3 and In particles shows good PTC properties. It will be applied to the shape free heater. The second is to arrange particles one by one using a tungsten microprobe. Metallic particles can weld on a metallic substrate by applying high voltage between the particles and the substrate. Three dimensional micro-structures are produced from gold and nickel particles. The third one is to arrange numerous particles at a time. The principle is to draw fine electrified patterns by an electrified beam or an ion beam on an insulating substrate, and particles adhere to the patterns by electrostatic force. The last one is to make particle chains at a selected position on an electrode, where micro pillars are prepared, using the pearl chain effect.
- (2) Fine particles are manipulated in a vacuum chamber, observed with SEM. Three dimensional structures are constructed with sub-micron particles and their optical properties are measured. This study is pursued in collaboration with the University of Tokyo.
- (3) Particles of polystyrene coated with nickel alloy are sintered and three dimensional micro-honeycomb materials are constructed. The material is very light and has a high energy absorption and a large ultrasonic attenuation coefficient. This result shows that this 3D micro-honeycomb material can be utilized for mechanical parts or structures with damping functions.
- (4) Creep tests are carried out for the alloys, which are prepared by adding B and N to the standard heat resisting alloy. The life of some alloys is longer than that of the standard alloy. It is considered that BN is precipitated in the creep voids and prevents the voids from growing.
- (5) Surface relief in martensitic transformation of Fe-Mn-Si shape memory alloys is investigated by AFM. The preferential formation of a single martensitic variant in a

grain is observed, even in a non-trained sample.

<Research themes>

- (1) Creation of Multi-functional Materials by Assemblage of Primitive Functions (Intelligent Materials Research: April 1996 to March 2002, Norio Shinya)
- (2) Measurement and Analytical Evaluation of Interface Properties (Special Coordination Funds for Promoting Science and Technology: April 1995 to March 1997, Norio Shinya)
- (3) Creation of Micro Cellular Materials (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 1999, Satoshi Kishimoto)

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Oral presentation: 60

- (1)International Conference: 22
- (2)Domestic Conference: 38

Patent Application

- (1) Metallic Closed Cellular Materials and the Production Method: S.Kishimoto, N.Shinya, 1997.11.04, Application Number: 09-301596
- (2) Drawing Apparatus by Charged Powder Particles: M.Egashira, N.Shinya, H.Saito, 1998.04.06, Application Number: 10-092988
- (3) High Luminance Gun of Powder Particles: M.Egashira, N.Shinya, H.Saito, 1998.04.06, Application Number: 10-092989
- (4) Detector of Powder Particles: M.Egashira, N.Shinya, H.Saito, 1998.04.06, Application Number: 10-092990
- (5) Control Apparatus for Powder Particles: M.Egashira, N.Shinya, H.Saito, 1998.04.06, Application Number: 10-092991
- (6) Control Apparatus for Fine Particle Beam: M.Egashira, N.Shinya, H.Saito, 1998.04.06, Application Number: 10-092992
- (7) Polarized Electron Beam Generator: M.Egashira, H.Saito, 1998.04.06, Application Number: 10-093000
- (8) Method and Apparatus for Coating on Powder Particles: 1998.10.01, Kobayashi, M.Egashira, T.Dan, N.Shinya, Application Number: 10-280105
- (9) Accurate Three Dimensional Positioning of Fine Objects: M.Hase, M.Egashira, N.Shinya, 1998.10.02, Application Number: 10-281778
- (10) Method for Deformation Measurement and Grid Sheet for the Measurement: S.Kishimoto, H.Xia, N.Shinya, 1998.11.20, Application Number: 10-375230
- (11) Accurate Positioning of Fine Objects: H.Fudouzi, T.Konno, M.Egashira, M.Kobayashi, N.Shinya, 1998.12.01, Application Number: 10-342134

Awards:

- (1) Persons who have proposed technical ideas in relation to their job, 1997.4.14, Science and Technology Agency, M. Egashira
- (2) Persons who have proposed technical ideas in relation to their job, 1998.4.13, Science and Technology Agency, T. Konno:
- (3) JIM Award for Encouragement of Research, 1998.9.28, M. Hase
- (4) MRS Outstanding Poster Award, 1998.123.8, S. Kishimoto

2.5.3 2nd Subgroup

Akira Ishida (Subgroup Leader), Morio Sato, Atsushi Takei (guest researcher),

Hachiro Imai (guest researcher), Tsuguto Yamashita (guest researcher), Shuichi Miyazaki (guest researcher)

2.5.3.1 Research work

The mechanical properties of sputter-deposited Ti-Ni thin films have been evaluated. Tensile tests revealed that sputter-deposited thin films, especially Ti-rich Ti-Ni thin films, show good ductility (40% elongation) as well as high strength (1.2 GPa yield stress) owing to their fine microstructure, peculiar to thin films. The shape memory behavior of these thin films has been also studied. The results prove that sputter-deposited thin films of Ti-Ni possess stable shape memory effects and reliable mechanical properties for practical applications.

<Research theme>

- (1) Development of Advanced Shape Memory Thin Films by Sputtering

(General Research: April 1997 to March 2002, Akira Ishida)

2.5.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) A. Ishida, M. Sato, T. Kimura, S. Miyazaki: Microstructure of Ti-rich Ti-Ni Thin Films, 2nd Inter. Conf. on SMST (Shape Memory and Superelastic Technologies) p.161, Pacific Grove, March 3-7, 1997
- (2) A. Ishida, M. Sato, T. Kimura, S. Miyazaki: Microstructure of Ti-48.2at.%Ni Shape Memory Thin Films, Metall. Mater. Trans. A, 28A, 1985 (1997)
- (3) M. Sato, A. Ishida, S. Miyazaki: Two-way Shape Memory Effect of Sputter-deposited Thin Films of Ti-51.3at.%Ni, Thin Solid Films, 315, 305 (1998)
- (4) A. Ishida: "Sputtering", in *Handbook of Surface Finishing*, ed. by the Surface Finishing Society of Japan (Nikkan Kogyo Shinbunsha, Tokyo 1988) (in Japanese)
- (5) A. Ishida, S. Miyazaki: Microstructure and Mechanical Properties of Sputter-deposited Ti-Ni Alloy Thin Films, Trans. ASME, 121, 2 (1999)
- (6) A. Ishida, M. Sato, T. Kimura, S. Miyazaki: Stress-Strain Curves of Sputter-Deposited Ti-Ni Thin Films, Phil. Mag., in press (1999)
- (7) A. Ishida, M. Sato, S. Miyazaki: Mechanical Properties of Ti-Ni Shape Memory Thin Films Formed by Sputtering, J. Mater. Sci. & Eng. A, in press (1999)

Oral Presentation: 7

- (1) International Conference: 1
- (2) Domestic Conference: 6

Patent Application:

- (1) A Shape Memory Alloy with Ductility and a Making Process of the Same: A. Ishida, S. Miyazaki, March 15, 1999, 09/267, 612

Award:

- (1) Persons who have proposed technical ideas in relation to their job: STA, 1998, M. Sato

2.5.4 Researchers in the 5th Research Group

Age and research fields of researchers in the 5th Research Group. (March 31, 1999)

Age of Researchers	Number of Researchers	Metallurgy	Functional Materials	Solid-state Physics	Mechanical Properties
60 ~	2	0	2	0	0
50 ~ 59	6	1	3	0	2
40 ~ 49	7	1	5	0	1
30 ~ 39	4	0	2	2	0
22 ~ 29	0	0	0	0	0
Summation	19	2	12	2	3

2.5.5 Research Budgets in the 5th Research Group

Research budgets in the 5th Research Group. (yen)

Kinds of budgets	1997	1998
Intelligent Materials Research	35,145,000	43,436,000
Special Coordination Funds for Promoting Science and Technology	5,600,250	8,550,000
General Research	9,960,000	5,000,000
Supplementary Budget	0	85,300,000
Summation	50,705,250	142,286,000

Chapter 3: Research in Advanced Physical Fields

3.1 Center for Advanced Physical Fields

Kazuhiro Yoshihara (Director of Center for Advanced Physical Fields)

The advanced physical fields, such as high magnetic field, high resolution beam technology, and extreme high vacuum, are suitable environments to create and observe the nano-structure materials, and we can expect these nano-structure materials exhibit unique behaviors in these environments. To execute research in these advanced physical fields, we established the Center for Advanced Physical Fields, which has the three research stations mentioned above, and large scale facilities have been set up.

The objectives of the Center is, (1) to improve techniques to establish these advanced physical fields, (2) to execute research on materials with atomic-scale controlled structures which show new quantum effects, and (3) to open these large scale facilities to all scientists around the world. In sections 3.1.1, 3.1.2 and 3.1.3, the recent research results in High Magnetic Field Station, High Resolution Beam Technology Station and Extreme High Vacuum Station are shown respectively. At present, the facilities in the High Magnetic Field Station have been open to researchers from outside of the NRIM, and other facilities in the High Resolution Beam Technology Center and the Extreme High Vacuum Center will be similarly open in the near future. The present status of user facilities in the High Magnetic Field Station is explained in section 3.1.1.9.

One specially allocated budget to the Center is for "Materials Research under Advanced Physical Fields", which supports two research projects (High magnetic field chemistry and Luminescent micro crystal) in the Center. Other research projects in the Center are supported by "R&D on New Superconducting Materials", "Materials Research using Synchrotron Radiation", "Special Research", "Nuclear Energy Research", and "Special Coordination Funds for Promoting Science and Technology" budget.

One of the major research projects in the Center is "Research on materials with atomic-scale controlled structures", which is supported by the "Special Coordination Funds for Promoting Science and Technology". This project, started in 1995, is promoted as one of the Center of Excellence Development Programs (COE Project) of the Japanese Government. The project is in fact designed to make use of the advantages of these facilities in the Center for Advanced Physical Fields.

The COE Project is roughly divided into two parts. One is "Improvement of

techniques to establish extreme physical fields". The techniques provided by the Center for Advanced Physical Fields are important to carry out research on the creation of materials with atomic scale structures. We call this research "COE supporting research". The other one is "Creation and physical properties of atomic scale structure materials". We call this research "COE core research". Atomic scale structure materials are categorized into quantum dot, quantum wire and thin film. Therefore, we divided "COE core research" into 5 sub-projects, which cover quantum dot, quantum wire and thin film. We have created new nano-dimension crystals, fabricated single atomic width lines which exhibited electron transport properties, and found interesting quantum phenomena in the high magnetic fields.

Since the starting stage of the project, the research fellow positions of the COE project have been opened to the world. Over 20 research fellows are employed from all over the world. However, the number of fellows is still not sufficient, though participation of young scientists, both from foreign countries and from Japan has substantially increased. To encourage mutual communication, an International Symposium on Advanced Physical Fields is held in the NRIM once a year, which has enhanced research activities in this research field.

3.1.1 High Magnetic Field Research Station

Hitoshi Wada (Supervising Researcher)

3.1.1.1 Research Work

Based on the 5th Long-term Plan, a variety of R&D activities are going on that include the generation and application of high magnetic fields:

- (1) R&D studies on the technologies for generating high fields and cryogenic temperatures to continually improve the capacity of the generated magnetic fields.
- (2) Application studies on magnetic fields for different purposes, including protein crystal growth, etc.
- (3) Participation in and contribution to international standardization activities in superconducting materials.

The research organization of the Station includes 14 member researchers, 1 researcher, who concurrently holds a post at a different national institute, 4 domestic fellows, 2 COE fellows, 4 visiting researchers from private companies and 2 special technical staffs. In addition, there are 8 technicians, for the operation of large machines, 1 assistant, for experiments, and 2 assistants, for database and office work.

The high magnetic field facilities of the Station have been opened to external users since April 1998. These two aspects of the Station's activities are described below.

3.1.1.2 Magnet Development Unit

Tsukasa Kiyoshi (Unit Leader), Toshihisa Asano, Hiroshi Morita (domestic research fellow), Hiroyuki Nakayama (domestic research fellow), Osamu Ozaki (visiting researcher), Jun-ichi Fujihira (visiting researcher)

3.1.1.2.1 Research work

(1) Superconducting magnets

(1-1) 1 GHz class NMR spectrometer

A 1 GHz class NMR magnet (generated magnetic field is 23.5 T) development program is in progress in the second stage of the Multi-core Project. The project was started in fiscal 1995. The 23.5 T superconducting magnet system is being fabricated by combining a metallic superconductor magnet (outer magnet) and an oxide superconductor magnet (inner magnet). The developed high-field NMR spectrometer is expected to be a powerful tool in the protein structure analysis project to be

challenged by the Institute of Physical and Chemical Research.

(1-2) Protein crystal growth

In collaboration with the National Institute of Bioscience and Human-Technology and the National Institute of Materials and Chemical Research, a unique NbTi superconducting magnet has been developed which generates a uniform magnetic force field of $232 \text{ T}^2/\text{m}$ (0.17 G) in a space of $10 \text{ mm } \phi \times 10 \text{ mm}$ in which protein crystal growth experiments can be conducted.

(2) Development of resistive magnets

(2-1) Water-cooled magnets

In order to generate dc magnetic fields higher than 30 T, a hybrid magnet is necessary consisting of a superconducting outsert magnet and a water-cooled insert magnet.

Two main structural types of water-cooled magnets for high-field use have been studied these are the polyhelix type and the Bitter type.

A three co-axially stacked Bitter type magnets were developed in collaboration with the National High Magnetic Field Laboratory, USA, generating a magnetic field of 28.8 T, the Japanese record for a water-cooled magnet, in September 1997.

(2-2) Magnetic stimulation

Magnetic stimulation by a pulsed magnet field is considered to be due to eddy currents induced in an organism. It has however not been clarified how the induced eddy current is distributed in the organism. Three-dimensional eddy current analysis was made by applying the finite element analysis method. Calculations were compared with the experimental results, measured by Kagoshima University to define the optimal coil shape capable of localizing the eddy current. This is a joint study with Brown University, in the USA, and the Shimadzu Corporation.

<Research themes>

- (1) Development of 1 GHz Class NMR Spectrometer (R&D on New Superconducting Materials: April 1995 to March 2002, Hitoshi Wada)
- (2) Superconducting Magnet Development for Very high Field Use (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1998, Hitoshi Wada)
- (3) Protein Crystal Growth under Variable Gravity Conditions (Core Research for Evolutional Science and Technology: October 1997 to September 2002, Hitoshi

Wada)

- (4) Improvement of Magnet Technology for High Field Magnets (General Research: April 1998 to March 2001, Tsukasa Kiyoshi)
- (5) High-Strength and Conductivity Materials for High Field Magnet Use (General Research: April 1995 to March 1998, Hitoshi Wada)

3.1.1.2.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999

- (1) T. Kiyoshi, K. Inoue, M. Kosuge, H. Wada, H. Maeda: Current Decay Evaluation of Closed HTS Coil Circuits, *IEEE Trans. Appl. Superconductivity*, **7**, 877 (1997)
- (2) H. Kumakura, H. Kitaguchi, T. Kiyoshi, K. Inoue, K. Togano, M. Okada, K. Fukushima, K. Tanaka, K. Kato, J. Sato: Performance Tests of Bi-2212 Insert Magnets Fabricated by Ag Sheath Method and Dip-coating Method, *IEEE Trans. Appl. Superconductivity*, **7**, 646 (1997)
- (3) T. Hase, K. Shibutani, S. Hayashi, M. Shimada, R. Ogawa, Y. Kawate, T. Kiyoshi, K. Inoue: Operation of Superconductively Jointed Bi-2212 Solenoidal Coil in Persistent Current Mode, *Cryogenics*, **37**, 201 (1997)
- (4) K. Fukushima, M. Okada, J. Sato, T. Kiyoshi, H. Kumakura, K. Togano, H. Wada: Persistent Mode Operation of $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x/\text{Ag}$ Stacked Double Pancake Coils with Superconducting Joints, *Japanese J. Appl. Phys.* **36**, L1433 (1997)
- (5) T. Hase, K. Shibutani, S. Hayashi, M. Shimada, R. Ogawa, Y. Kawate, T. Kiyoshi, K. Inoue: Fabrication of Double-sheathed $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ Multifilamentary Wire for Solenoidal Coil, *J. Japan Inst. Metals*, **61**, 842 (1997) (in Japanese)
- (6) T. Kiyoshi, K. Inoue, A. Sato, H. Aoki, K. Itoh, H. Wada, H. Maeda: 1GHz NMR Spectrometer Project at National Research Institute for Metals, in *High magnetic fields: applications, generation and materials*, ed. by H. J. Schneider-Muntau, (World Scientific, Singapore 1977)
- (7) M. Morita, S. Ito, K. Inoue, T. Kiyoshi, H. Maeda: Development of a 40 T Class Hybrid Magnet, in *High magnetic fields: applications, generation and materials*, ed. by H. J. Schneider-Muntau, (World Scientific, Singapore 1977)
- (8) H. Kumakura, H. Kitaguchi, T. Kiyoshi, K. Inoue, K. Togano, H. Maeda, M. Okada, K. Fukushima, K. Tanaka, K. Kato: High-Tc Superconductor Research for High Field Applications at National Research Institute for Metals, in *High magnetic fields: applications, generation and materials*, ed. by H. J. Schneider-Muntau, (World Scientific, Singapore 1977)
- (10) K. Inoue, T. Kiyoshi, K. Itoh, H. Kumakura, H. Kitaguchi, A. Sato, Y. Iijima, H.

- Wada: Development of 1 GHz NMR Spectrometer Magnet at NRIM, in *Advances in Superconductivity*, ed. By S. Nakajima and M. Murakami, IX (Springer, Tokyo 1997)
- (11) M. Okada, K. Tanaka, K. Fukushima, J. Sato, H. Kitaguchi, H. Kumakura, T. Kiyoshi, K. Inoue, K. Togano: Development of Bi-2212/Ag Superconducting Insert Magnets for High Magnetic Field Generation, in *Advances in Superconductivity*, ed. by S. Nakajima and M. Murakami, IX (Springer, Tokyo 1997)
- (12) M. Okada, K. Fukushima, J. Sato, K. Nomura, H. Kitaguchi, H. Kumakura, T. Kiyoshi, K. Togano, H. Wada: Fabrication and Transport Properties of Bi-2212/Ag Multifilamentary Tapes and Coils for High Magnetic Field Generation, 3rd European Conf. on Applied Superconductivity, Eindhoven, Netherlands, June 30- July 3, 1997
- (13) M. D. Bird, S. Bole, Y. M. Eyssa, H.-J. Schneider-Muntau, T. Kiyoshi, T. Asano, Y. Sakai, K. Inoue, H. Wada: NRIM/NHMFL Joint Development of a 30 T Magnet, 15th Int. Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997
- (14) K. Fukushima, M. Okada, T. Kiyoshi, H. Kumakura, K. Togano, K. Inoue, H. Wada: Development of Bi-2212/Ag Closed Circuit for 1 GHz-NMR Spectrometer, 15th Int. Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997
- (15) T. Hase, O. Ozaki, K. Shibutani, S. Hayashi, M. Shimada, Y. Kawate, K. Takabatake, T. Kiyoshi, M. Yuyama, K. Inoue, H. Wada: Normal-zone Propagation in Bi-2212 Solenoidal Coil at 4.2 K, 15th Int. Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997
- (16) R. Hirose, T. Kamikado, O. Ozaki, M. Yoshikawa, T. Hase, M. Shimada, Y. Kawate, K. Takabatake, M. Kosuge, T. Kiyoshi, K. Inoue, H. Wada: 21.7 T Superconducting Magnet Using (Nb,Ti)3Sn Conductor with 14%-Sn Bronze, 15th Int. Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997
- (17) T. Kiyoshi, H. Kitaguchi, H. Kumakura, K. Inoue, H. Wada, T. Hase, S. Hayashi, K. Ohkura, K. Sato, K. Tanaka, M. Okada: High-Field Performance of BSCCO Coils with an Outer Diameter of 130 mm, 15th Int. Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997
- (18) O. Ozaki, R. Hirose, M. Yoshikawa, S. Ito, T. Hase, M. Shimada, Y. Kawate, K. Takabatake, T. Kiyoshi, M. Kosuge, K. Inoue, H. Wada: Quench Behavior of Multi-Sectional Superconducting Magnet in Superfluid Helium, 15th Inter. Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997
- (19) T. Asano, T. Kiyoshi, K. Inoue, H. Wada, K. Nakagawa, G. Iwaki: A New High Strength Conductor with High Conductivity for High Magnetic Fields, 15th

- Int. Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997
- (20) Y. Sakai, T. Asano, T. Kiyoshi, K. Inoue, H. Wada: Development of the Ultra-High Strength, High Conductivity Cu-Ag Alloy, 15th Int. Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997
 - (21) T. Kamikado, R. Hirose, M. Shimada, T. Kiyoshi, K. Inoue, H. Wada: Stabilities of Superconducting Split Pair Coils, 15th Int. Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997
 - (22) K. Inoue, Y. Iijima, T. Takeuchi, T. Kiyoshi, K. Fukuda, K. Nakagawa, G. Iwaki, H. Moriai: New Nb₃Al Multifilamentary Conductor and Its Application to High Field Superconducting Magnet, *Physica B*, **246-247**, 364 (1998)
 - (23) T. Hase, K. Shibutani, S. Hayashi, M. Shimada, Y. Kawate, T. Kiyoshi, H. Kitaguchi, H. Wada: Persistent-current-mode Operation of Bi-2212 Solenoidal Coil with a Clear Bore of 60 mm, in *Advances in Superconductivity*, ed. by K. Osamura and I. Hirabayashi, X (Springer, Tokyo 1998)
 - (24) M. Okada, K. Tanaka, S. Matsuda, K. Sato, J. Sato, H. Kitaguchi, T. Kiyoshi, H. Kumakura, K. Togano, H. Wada: Bi-2212/Ag Superconducting Magnet for High-Field Applications, in *Advances in Superconductivity*, ed. By K. Osamura and I. Hirabayashi, X (Springer, Tokyo 1998)
 - (25) K. Togano, H. Kumakura, H. Kitaguchi, H. Fujii, T. Kiyoshi, H. Wada, M. Okada, J. Sato, T. Hasegawa: Development of Bi₂Sr₂Ca₁Cu₂O_x/Ag Tapes and Coils, in *Advances in Superconductivity*, ed. By K. Osamura and I. Hirabayashi, X (Springer, Tokyo 1998)
 - (26) T. Kiyoshi, H. Wada: Development of an Inner Coil for a 1 GHz NMR Magnet, *OYO-BUTURI*, **67** 431 (1998) (in Japanese)
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 - (28) T. Miyazaki, Y. Murakami, T. Hase, T. Miyatake, M. Shimada, Y. Kawate, T. Kiyoshi, K. Itoh, H. Wada: Development of Superconductors for 1 GHz NMR Magnet -High Critical Current Dendity (Nb,Ti)₃Sn Conductor-, *J. Cryogenic Society of Japan*, **33**, 725 (1998) (in Japanese)
 - (29) T. Kiyoshi, A. Sato, H. Wada: Development of 1GHz Superconducting NMR Magnet at TML/NRIM, *IEEE Trans. Appl. Superconductivity*, in press
 - (30) T. Kiyoshi, O. Ozaki, H. Nakayama, H.-B. Jin, H. Wada, N. Wakayama, M. Ataka, Superconducting Magnets for Generating Uniform Magnetic Force Field, *IEEE Trans. Appl. Superconductivity*, in press

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- (32) T. Miyazaki, Y. Murakami, T. Hase, M. Shimada, K. Itoh, T. Kiyoshi, T. Takeuchi, K. Inoue, H. Wada: Development of Nb₃Sn Superconductors for a 1 GHz Class NMR Magnet - Tin Content in Bronze Matrix Dependence of High-field Characteristics-, IEEE Trans. Appl. Superconductivity, in press
- (33) O. Ozaki, M. Kosuge, T. Kiyoshi, M. Yuyama, H. Wada, T. Kamikado, Y. Murakami, T. Miyazaki, S. Hayasi, Y. Kawate: R&D Studies on Mechanical Stress of 1GHz NMR Magnet, IEEE Trans. Appl. Superconductivity, in press
- (34) L.Y. Xiao, T. Kiyoshi, O. Ozaki, H. Wada: Case Study on Quench Evolution and Passive Protection of High Tc Superconducting Pancake Coil, Cryogenics, in press

Oral Presentation: 98

- (1) International Conference: 37
- (2) Domestic Conference: 61

Patent Application

- (1) Uniform Magnetic Force Magnet: Tsukasa Kiyoshi, Hitoshi Wada, Nobuko Wakayama, May 19, 1998, No. 10-137220

Award

- (1) The Ichimura Prizes in Technology-Meritorious Achievement Prize, April 1998: Tsukasa Kiyoshi

3.1.1.3 Cryogenic Technology Unit

Akio Sato (Unit Leader), Takenori Numazawa, Bunmei Matsumoto, Hideo Nagai, Michio Kosuge, Michinari Yuyama, Takashi Miki (COE fellow), Sigeki Nimori (special technical staff)

3.1.1.3.1 Research work

Studies have been carried out on a superfluid stirling cycle, capable of directly cooling a superconducting magnet at a superfluid helium temperature below 2 K, a cooling system to generate cryogenic temperatures below 1 K in high fields, and a magnetic

refrigerator to be mounted in a space experiment base. In addition, techniques needed for measuring specific heat and thermal conduction at a level of 100 mK have been studied. A challenging program is in progress with respect to an extensive cryogenic engineering database in cooperation with the Cryogenic Association of Japan.

<Research themes>

- (1) Refrigerator for Ultra-Low Temperature Generation (General Research: April 1995 to March 1998, Akio Sato)
- (2) Measurement of Thermal Properties of Cryogenic Materials (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 2002, Akio Sato)
- (3) Standard materials and Measurement Methods for Cryogenic Materials (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 2002, Akio Sato)

3.1.1.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) A.Sato, T.Kiyoshi, H.Wada, H. Maeda, S.Ito, Y.Kawate: Design of Superfluid-cooled Cryostat for 1 GHz NMR Spectrometer, Proc. 6th Int. Cryogenic Eng. Conf., ed. by T.Haruyama et al. (Elsevier Science, Oxford 1997) p.431
- (2) A.Sato, K.Fujioka, T.Haruyama, H.Hirabayashi, T.Ishigohoka, A.Ishiyama, Y.Kawate, K.Nishigaki, S.Nishijima, T.Noguchi, O.Ogino, H.Ogiwara, O.Okazaki: What Happened to Cryogenic and Superconducting Equipment in the Great Hanshin Earthquake?, Proc. 16th Inter. Cryogenic Eng. Conf., ed. by T.Haruyama et al. (Elsevier Science, Oxford 1997) p.513
- (3) A. Sato, T.Miki, F.Matsumoto, H.Nagai, H.Wada, S.Ito, Y.Kawate: Development of Superfluid-cooled Cryostat for 1 GHz NMR Spectrometer, 15th Inter. Conf. on Magnet Technology (MT-15), Beijing October 20-24, 1997
- (4) T. Miyazaki, Matsukura, T.Miyatake, M.shimada, K.Itoh, T.Kiyoshi, A. Sato, H. Wada, K.Inoue: Improvement of Critical Current Density in the Bronze-processed Nb₃Sn Superconductors, Advances in Cryogenic Engineering (Materials), Vol.44, 935, 1997
- (5) T. Miyazaki, Matsukura, T.Miyatake, M.Shimada, K.Itoh, T.Kiyoshi, A. Sato, H.Wada, K.Inoue: Development of Bronze-processed Nb₃Sn Superconductors for 1 GHz NMR Magnets, Advances in Cryogenic Engineering (Materials), 44, 943, (1997)
- (6) T. Kiyoshi, A.Sato, H.Wada, S.Hayashi, M.Shimada, Y.Kawate: Development of 1

GHz Superconducting NMR Magnet at TML / NRIM, IEEE Trans. Appl. Superconductivity, in press

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- (8) A.Sato, T.Miki, F.Matsumoto, H.Nagai, H.Wada: Development of Superfluid-Cooled Cryostat for 1 GHz NMR Spectrometer - Detailed Design-, Proc. 17th Int. Cryogenic Eng. Conf., ed. by D. Dew-Hughes, (Institute of Physics Publishing, Bristol and Philadelphia, 1998) p.613
- (9) T.Miki, A.Sato, F.Matsumoto, H.Nagai: Stability Analysis for Pressurized He II Cooling System with Tube Heat Exchangers, Proc. 17th Int. Cryogenic Eng. Conf., ed. by D. Dew-Hughes, (Institute of Physics Publishing, Bristol and Philadelphia, 1998) p.835
- (10) T. Numazawa, A. Sato, K. Shimamura: Thermal Conductivity of Rare-earth Oxides for Heat Switch Application, Proc. 16th Inter. Cryogenic Eng. Conf., ed. by T.Haruyama et al. (Elsevier Science, Oxford 1997) p.2073
- (11) T. Numazawa: Recent Progress on Magnetic Refrigeration, Cryogenic Eng. 32, 192-202(1998)
- (12) T. Numazawa, A. Sato, K. Shimamura: Thermal Conductivity of RAl_3 (R=Dy, Er and Ho), J. Mater. Sci., 33, 827 (1998)

Oral Presentation: 17

- (1) International Conference: 8
- (2) Domestic Conference: 9

Patent Application

- (1) Superfluid Helium Refrigerator: Akio Sato, Hideo Nagai, May14, 1997, No.09-122554

Award

- (1) The 57th (1998) Noteworthy Inventions: Michinari Yuyama

3.1.1.4 Superconductor Development Unit

Takao Takeuchi(Unit Leader), Michio Kosuge, Michinari Yuyama, Masahiro Uda
(visiting scientist)

3.1.1.4.1 Research work

R & D studies have been carried out on new superconducting materials for generation of a field of 25 T. Nb₃Al wires manufactured by a newly developed method has demonstrated excellent strain tolerance and Jc characteristics up to a magnetic field region far above 20T.

In addition, a new superconducting material is required for the superconducting magnet for the Large Elementary Particle Accelerator (LHC) of CERN. We are collaborating with the High Energy Particle Accelerator Research Organization of the Ministry of Education to make a contribution to this huge scientific challenge.

<Research theme>

- (1) Stability of Superconducting Materials (Nuclear Energy Research: April1994 to March 1999, Hitoshi Wada)

5.1.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999

- (1) M. Kosuge, T. Takeuchi, M. Yuyama, Y. Iijima, K. Inoue, H. Wada, K. Fukuda, G. Iwaki, S. Sakai, H. Moriai, B. ten Haken, h. H. J ten Kate: High Field Performance of Nb₃Al Multifilamentary Conductors Prepared by Phase Transformation from bcc Solid Solution, Inst. Phys. Conf. Ser. 158, 1607(1997).
- (2) T. Takeuchi, Y. Iijima, K. Inoue, H. Wada, B. ten Haken, H. H. H. ten Kate, K. Fukuda, G. Iwaki, S. Sakai, H. Moriai : Strain Effects in Nb₃Al Multifilamentary Conductors Prepared by Phase Transformation from bcc Supersaturated-Solid-Solution, Appl. Phys. Lett., 71, 122 (1997).
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- (4) T. Takeuchi, Y. Nemoto, H. Maeda, K. Itoh, K. Inoue, M. Kosuge, M. Yuyama, H. Wada: Microstructure and Electromagnetic Characteristics of Multifilament V₃Si Superconductors, J. Japan Inst. Metals, 62, 727 (1998). (in Japanese)
- (5) T. Takeuchi: Characteristics of Nb₃Al Multifilamentary Superconductors Prepared by Phase Transformation from a Supersaturated Solid Solution, Cryogenic

Engineering, **33**, 614 (1998).

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- (7) M. Rabara, Y. Yoshida, T. Takeuchi, P. Miranovic, K. Miya: Irreversibility Field Analysis for $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ Tapes by Using Axial Probe, *Physica C*, **305**, 285 (1998).
- (8) K. Nakagawa, K. Fukuda, G. Iwaki, K. Chida, H. Moriai, t. Takeuchi, Y. Iijima, K. Inoue, K. Tagawa, T. Kiyoshi, K. Itoh, H. Wada, N. Kobayashi: Development of Nb_3Al Superconducting Wires Fabricated by the RHQT Process, *Cryogenic Engineering (Japan)*, **33**, 623(1998). (in Japanese)
- (9) M. Rabara, T. Takeuchi, K. Miya: Systematic Study of Angular Hysteresis of Critical Current Density in $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ Tapes, *Physica C*, **313**, 213 (1999).
- (10) T. Takeuchi, K. Tagawa, T. Kiyoshi, K. Itoh, M. Kosuge, M. Yuyama, H. Wada, Y. Iijima, K. Inoue : Enhanced Current Capacity of Jelly-roll Processed and Transformed Nb_3Al Multifilamentary Conductors, to be published in *IEEE Appl. Superconductivity* (1999).
- (11) K. Nakagawa, K. Fukuda, G. Iwaki, H. Moriai, Y. Iijima, T. Takeuchi, T. Kiyoshi, K. Itoh, K. Inoue, H. Wada, N. Kobayashi: Development of Nb_3Al Superconducting Wires Fabricated by Rapid-Quenching and Transforming Process, 15th Conf. on Magnet Technology, Beijing, Oct. 20-24, 1997, 977-980.
- (12) Y. Murakami, K. Itoh, M. Yuyama, H. Wada: Field-angle Dependence of Critical Current in Ag-sheathed Bi-2212 and Bi-2223 Tapes, *Adv. Cryog. Engin.* **42**, 529 (1997).
- (13) T. Kuroda, Y. Murakami, K. Itoh, M. Yuyama, H. Wada, D. Maos: Temperature Dependence of Critical Current Density of Nb_3Al Multifilamentary Wires Fabricated by Nb-tube and its Improved Process, *Cryogenics*, **38**, 785 (1998).

Oral presentation: 17

- (1) International Conference: 4
- (2) Domestic Conference: 13

Patent application:

- (1) Nb_3Al -compound Superconducting Wire and Fabrication Process thereof:
T. Takeuchi, T. Kiyoshi, K. Itoh, H. Wada, Y. Iijima, K. Inoue, K. Nakagawa, G. Iwaki, H. Moriai, m. Kimura, S. Inaba, K. Aihara, Y. Wadayama, October 9, 1998, 1998-287491

- (1) Nb₃Al-compound Superconducting Wire and Fabrication Process thereof, T. Takeuchi, T. Kiyoshi, K. Itoh, H. Wada, Y. Iijima, K. Inoue, K. Nakagawa, G. Iwaki, H. Moriai, M. Kimura, S. Inaba, K. Aihara, Y. Wadayama, October 9, 1998, 1998-287492

Award:

- (1) The 56th (1997) Noteworthy Inventions: T. Takeuchi
(2) 1998 Ohshima Award for Excellent Presentation: T. Takeuchi

3.1.1.5 High Field Application Unit

Kikuo Itoh (Unit Leader), Hideyuki Ohtsuka, Takeshi Ohara (guest researcher from ETL), X. Wang (COE fellow), Ya Xu (domestic research fellow), Hidehiko Okada (visiting researcher)

3.1.1.5.1 Research work

(1) International Standardization of superconducting materials

We proposed standard test methods on the critical current measurement of Nb₃Sn and Bi oxides as the Chairing Organization (Chairman: Hitoshi Wada) in the field of superconducting materials in the international cooperative VAMAS (Versailles Project on Advanced Materials and Standards). These proposals were adopted as the draft standards of the International Electrotechnical Commission (IEC).

(2) Magnetic separation

Development of a Bi-oxide based superconducting magnet for a magnetic separator is in progress in the second stage of the Multi-core Project, which was started in fiscal 1995. This study includes a new concept magnetic analysis method (magnetic chromatography) and is joint work with the Electrotechnical Laboratory of MITI.

(3) High field metallurgy

The effects of high fields on the solid/solid phase transformation and the microstructures of metallic materials have been examined. It has been shown that the magnetic fields effects transformation of the nucleation mechanism as well as the kinetics.

In addition, it has also been found that the phase diagram is changed in the magnetic

field, diffusional transformation is largely promoted, and the recrystallization texture is changed.

<Research themes>

- (1) Evaluation of superconducting wires: R & D on New Superconducting Materials, April 1995 to March 2000, Hitoshi Wada)
- (2) Evaluation Methods for Superconducting Materials: Special Coordination Funds, April 1997 to March 2002, Hitoshi Wada)
- (3) Development of Magnetic Separation System (R & D on New Superconducting Materials: April 1995 to March 2000, Takeshi Ohara)
- (4) Control of Martensitic Transformations and Microstructures in Ferrous Alloys: (General Research, April 1995-March 1998, Hideyuki Ohtsuka)
- (5) Materials Development through Control of Phase Transformation by Magnetic Fields (General Research: April 1998 to March 2000, Hideyuki Ohtsuka)

3.1.1.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999

- (1) H.Wada, L.F.Goodrich, H.J.J.ten Kate: VAMAS Activities for Standardization of Measurement Methods for Superconducting Materials, Adv. Superconduct. X, 1417(1998)
- (2) K.Aihara, R.Tachikawa, T.Matsushita, K.Itoh, S.Murase, T.Saitoh, H.Moriai, K.Ohmatsu, K.Osamura: Standardization of the Test Method for Hysteresis Loss Measurement of Cu/Nb-Ti Composite Superconductors. Part1: VSM/SQUID method, Adv. Superconduct. X, 1425 (1998)
- (3) T.Kumano, M.Hiraoka, T.Shimtomi, K.Osamura, N.Higuchi, K.Itoh, Y.Kubo, M.Shimada: Adv. Superconduct. X, 1433 (1998)
- (4) K.Itoh, T.Kuroda, M.Yuyama, Y.Iijima, H.Wada, Y.Murakami, D.Mao, Field and Temperature Dependences of Critical Current in Direct-heated Nb-tube Processed Nb₃Al Wires, IEEE Trans. Appl. Supercond. 1576 (1997)
- (5) H.Kitaguchi, K.Itoh, H.Wada, K. Togano: Study on Reproducibility of Ttransport Critical Current for HTSC, Adv. Cryog. Engin. 44, 601 (1998)
- (6) H.Wada, K.Itoh, VAMAS Guideline for the Determination of Critical Currents in Bi-based Oxide Superconductors, Adv. Science and Technology, 16, 887 (1999)
- (7) M.Hiraoka, T.Kumano, K.Osamura, T.Shintomi, N.Higuchi, K.Itoh, Y.Kubo, Standardization of the Method for the Determination of the Cu/non-Cu Ratio of Cu/Nb₃Sn Composite Superconductors, to be published in Adv. Superconduct. XI, (1999)
- (8) T. Ohara : Feasibility of Using Magnetic Chromatography for Ultra-Fine Particle

- Separation, *High Magnetic Fields: Applications, Generation, Materials* (World Scientific Publishing Co. Pte. Ltd.) 43-55 (1997)
- (9) T. Ohara, X. Wang, E. R. Whitby, K. C. Karki, C. H. Winstead: Computer Simulation of Magnetic Chromatography System for Ultra-Fine Particle Separation, Proc. 9th Sym.on Electromagnetics and Dynamics, 445-450 (1997)
 - (10) X. Wang, T. Ohara, E. R. Whitby, K. C. Karki, C. H. Winstead: Computer Simulation of Magnetic Chromatography System for Ultra-Fine Particle Separation, IEE of Japan, 117-B, 1466-1474 (1997)
 - (11) T. Ohara : Foundation of Magnetic Separation, Proc. 1st Sym. on New Magnetic Science, 1-19 (1997)
 - (12) T. Ohara , X. Wang: Computer Simulation of Magnetic Chromatography System for Ultra-Fine Particle Separation, Proc. 1st Sym. on New Magnetic Science, 20-27 (1997)
 - (13) E. R. Whitby, X. Wang, T. Ohara: Modeling Transport in Particle Classification Systems: Application to Magnetic Chromatography” 16th Annual Conf. of the AAAR, Denver, Colorado, October 13-17 (1979)
 - (14) T. Ohara: Future of the Magnetic Separation Technology, Proc. Special Symp. on Cryogenics and Applied Superconductivity, 110-111 (1997)
 - (15) T. Ohara: Practical Application and Superconductorization of Magnetic Separation, Proc. 24th Sym. on Superconductivity Science and Technology, Forum of Superconductivity Science and Technology, 55-70 (1998)
 - (16) H. Wada, A. Fukuzawa, H. Otsuka, T. Ohara, H. Abe, T. Kiyoshi: Research Programs on Material Processing in High Magnetic Field at Tsukuba Magnet Laboratory, the American Ceramic Society, the 23rd Annual Cocoa Beach Conf. and Expo., Cocoa Beach, Florida, January 24-29 (1999)
 - (17) H.Ohtsuka:Effects of Magnetic Field on Nucleation in Phase Transformations, J. Japanese Association of Crystal Growth 24, 2(1997) (in Japanese)
 - (18) H.Ohtsuka, G.Ghosh, K.Nagai, S.Kajiware, H.Kitaguchi, M.Uehara: Effects of High Magnetic Field and Tensile Stress on Martensitic Transformation Behavior and Microstructure at 4.2K in Fe-Ni-C Alloys,in High Magnetic Fields: Applications, Generation, Materials (World Scientific 1997)
 - (19) H.Ohtsuka, G.Ghosh, K.Nagai, H.Kitaguchi, M.Uehara: Effects of High Magnetic Field and Tensile Stress on Martensitic Transformation Behavior and Microstructure at 4.2K in Fe-Ni-C Shape Memory Alloys, 16th Int. Cryogenic Engineering Conf./Int. Cryogenic Mater. Conf. (ICEC16/ICMC), Kitakyushu, May 20-24, 1996.

- (20) H.Ohtsuka, G.Ghosh, K.Nagai: Effects of Cu on Diffusional Transformation Behavior and Microstructure in Fe-Mn-Si-C Steels, *ISIJ Int.* **37**, 296 (1997)
- (21) H.Ohtsuka: Effects of Cu on Diffusional Transformation Behavior in High Purity Si-Mn Steels, *Materials Science of Steels*, Tokyo (1997)
- (22) H.Ohtsuka, K.Takashima, G.B.Olson: Nonthermoelastic and Thermoelastic Martensitic Transformation Behavior Characterized by Acoustic Emission in an Fe-Pt Alloy, *Mat. Res. Soc. Symp.*, Boston Dec., 1997
- (23) V.Saravanan, M.Sekar, K.Govinda Rajan, P.Ch.Sahu, Mohammad Yousuf, N.V.Chandra Shekar, N.Victor Jaya, H.Ohtsuka: Pressure Induced Electrical Resistance Behaviour of Fe-24Mn-6Si Shape Memory Alloy, *Int. Conf. on High Pressure Science and Technology*, Doshisya University, Kyoto 1997
- (24) M.Sekar, P.Ch.Sahu, N.V.Chandra Shekar, Mohammad Yousuf, K.Govinda Rajan, V.Saravanan, N.Victor Jaya, H.Ohtsuka: Pressure Induced Electrical Resistance Behaviour of Fe-24Mn Shape Memory Alloy up to ~ 6 GPa at 300K, *NCHST-IV*, IGCAR Kalpakkam, India 1997
- (25) D.M.Baskin, K.T.Faber, H.Ohtsuka: Forming Tough, Single Phase Laminates of Fe_2TiO_5 via Magnetic-Assisted Gelcasting, *J. Japan Inst. Metals* **61**, 1306(1997) (in Japanese)
- (26) H.Ohtsuka, G.Ghosh, H.Mamiya, H.Wada: Aging of an Fe-Ni-Al Alloy in Magnetic Field, *ibid.* **61**, 1333(1997) (in Japanese)
- (27) H.Ohtsuka, G.Ghosh, K.Nagai, H.Wada: Growth Behavior of Martensite Plates in Fe-Ni-C Alloys Formed under High Magnetic, *ibid.* **61**, 1337(1997) (in Japanese)
- (28) Y. Xu H. Ohtsuka, K.Ito, H.Wada, Y.Oda, Y.Tanaka: Effects of High Magnetic Field on Recrystallization and Coarsening Behavior in Fe-3%Si: 1st Symp. *New Magnetic Science*, Urawa, Japan, 124(1997)
- (29) J. Choi, H. Ohtsuka, Y. Xu: A Study on the Phase Transformation of Steels Under High Magnetic Field, *Workshop on the Development of High Performance Structural Steels for 21st Century (HIPERS-21)*, Korea, August, 1998
- (30) H. Ohtsuka, Y. Xu: Effects of High Magnetic Field on Phase Transformations in Fe-based Alloys, *Forefront of Computer Assisted Microstructure Control*, Tokyo September 29, 1998
- (31) Y.Xu, H.Ohtsuka, K.Anak, S.Miyazaki, K.Ito, H.Wada: Effects of High Magnetic Field on Recrystallization Texture in Fe-Si Alloys, 2nd Symp. *on New Magnetic Science*, Urawa, Japan, 1998
- (32) H.Ohtsuka, Y.Xu, H.Wada: Effects of High Magnetic Field on Diffusional

Transformation in Steels, *ibid.*

- (33) A.Chiba, H.Ohtsuka, Y.Xu, H.Wada: Effects of Magnetic Field on Strain Aging of Co-Ni Super Alloy, 2nd Symp. on New Magnetic Science (SNMS'98), Urawa November, 1998
- (34) T.Shimozono, Y.Kono, Y.Konishi, K.Shibata, H.Ohtsuka, H.Wada: Effects of Pre-strain, Heat Treatments and Magnetic Fields on α' Martensite Formation in Fe-25.5%Ni-3~5%Cr Alloys, Materials Science and Engineering, in press.
- (35) H.Ohtsuka, G.Ghosh, H.Wada: Size and Aspect Ratio of Martensite in Fe-Ni-C Alloys Formed Under Applied Magnetic Field, *ibid.*, in press.
- (36) H.Wada, A.Fukuzawa, T.Kiyoshi, H.Ohtsuka, K.Ohara: Research Programs on Materials Processing in High Magnetic Fields at Tsukuba Magnet Laboratory, An Int. Conf. on Engineering Ceramics and Structures, 23rd Annual Cocoa Beach Conf. & Exposition, Florida January, 1999.
- (37) K.Tsuchiya, A.Ohashi, M.Umemoto, H.Ohtsuka: Martensitic Transformation and Magnetic Properties of Ni-based Ferromagnetic Shape Memory Alloys, 8th Symp. of Intelligent Materials, Tokyo, March 16, 1999
- (38) H.Ohtsuka, Y.Xu, H.Wada: Solid/solid Phase Transformation Behavior and Microstructures of Materials in High Magnetic Field, Symp. of Microstructures and Magnetic Properties of Materials, Tokyo, March 30, 1999.

Oral Presentation: 55

- (1) International Conference: 14
- (2) Domestic Conference: 41

3.1.1.6 High Field Chemistry Unit

Haruo Abe (Unit Leader), Ken Takazawa, Nobuhisa Hashimoto (special technical staff)

5.1.6.1 Research work

The photochemical reactions of organic molecules and the reactions of high energy gaseous molecules have been measured in high magnetic fields. It has been found that the life-time of benzophenone molecule radical pairs generated in the photochemical reaction at 30 T becomes about one half of that at 2 T and that the yield of the reaction product is reduced by half. This indicates the possibility that the speed of the chemical

reaction and the yield of the product can be controlled by high fields.

<Research theme>

- (1) Control of Chemical Reaction Process by Magnetic Fields (Materials Research under advanced Physical Fields, April 1996 to March 2000, Haruo Abe)

3.1.1.6.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999

- (1) K. Nishizawa, Y. Sakaguchi, H. Hayashi, H. Abe, G. Kido: A Laser Flash Photolysis Study of the Effects of Ultrahigh Magnetic Fields up to 29.6T on Dynamic Behavior of Radical Pairs in Micellar Solutions, *Chem. Phys. Letters*, **267**, 501 (1997).
- (2) N. I. Wakayama, M. Ataka, H. Abe: Effect of a Magnetic Field Gradient on the Crystallization of Hen Lysozyme, *J. Cryst. Grow.* **178**, 653 (1997).
- (3) S. H. Lin, M. Hayashi, H. Kono, H. Hayashi, H. Abe, S. Ikeda: Effect of Magnetic Field on Photophysical Processes of Molecules, *J. Chinese Chem. Soc.*, **44**, 203 (1997).
- (4) M. Wakasa, K. Nishizawa, H. Abe, G. Kido, H. Hayashi: Saturation of Magnetic Field Effects due to the Dg Mechanism under Ultrahigh Fields up to 28T, *J. Am. Chem. Soc.*, **120**, 10565 (1998).
- (5) K. Takazawa, H. Abe: Electronic Spectra of Gaseous Nitric Oxide in the Magnetic Fields up to 10 T, *J. Chem. Phys.*, **110**, 9492 (1999).
- (6) K. Takazawa, H. Abe: Landau Level of Gaseous Nitric Oxide Studied by Two-Color Multiphoton Ionization Spectroscopy, *J. Chem. Phys.*, **110**, (1999) 11682.
- (7) S. Ikeda, H. Abe, H. Hayashi: Fluorescence Quenching of Gaseous Excited Molecules by Large Magnetic Fields: V 1B_2 State of CS₂, *RIKEN Review*, **15**, 53 (1997).
- (8) K. Nishizawa, Y. Sakaguchi, H. Hayashi, H. Abe, G. Kido: Ultrahigh Magnetic Field Effects on the Dynamic Behavior of Radical Pairs in Micellar Solutions, *RIKEN Review*, **15**, 49 (1997).
- (9) K. Takazawa, H. Abe : Dynamics of Electronically Excited Molecules in Strong Magnetic Fields, 7th MR Science International Workshop on Chemical Dynamics, p7, Tokyo, October 31 (1997)
- (10) K. Takazawa, H. Abe: Dynamics of Electronically Excited Molecules under High Magnetic Fields, 2nd Symp. on New Magneto-Science, p.219, Urawa, Nov. 25-27, (1998)
- (11) H. Abe, G. Kido, K. Nishizawa, Y. Sakaguchi, H. Hayashi: Effects of Ultrahigh Magnetic Fields on Photochemical Reactions in Solution, The 5th Int. Symposium on Magnetic Fields and Spin Effects in Chemistry and Related Phenomena, p30,

Jerusalem, October 26-30, (1997).

- (12) K. Takazawa, H. Abe: Electronic Spectra of gaseous NO in magnetic Fields up to 10T, 5th Inter. Symp. on Magnetic Fields and Spin Effects in Chemistry and Related p95, Jerusalem, October 26-30, (1997).

Oral Presentation: 11

- (1) International Conference: 4
(2) Domestic Conference: 7

3.1.1.7 Researchers in the High Magnetic Field Research Station

Age and research fields of researchers in the High Magnetic Field Research Station.

(March 31, 1999)

Age of Researchers	Number	Magnet Technology	Cryogenic Technology	Super- conducting Materials	Chemistry	Metal- lurgy
60 ~	1			1		
50 ~ 60	4		2	2		
40 ~ 49	8	2	4	1	1	
30 ~ 39	12	5	3		2	2
22 ~ 29	3	2		1		
Summation	28	9	9	5	3	2

3.1.1.8 Research Budgets in the High Magnetic Field Station

Research budgets in the High Magnetic Field Research Station.(yen)

Kinds of budgets	1997	1998
R & D on New Superconducting Materials	361,598,000	362,125,000
Nuclear Energy Research	6,726,000	9,394,000
Materials Research under Advanced Physical Field	22,814,000	24,369,000
Special Coordination Funds for Promoting Science and Technology	30,652,000	26,456,000
General Research	5,130,000	4,890,000
Core Research for Evolutional Science and Technology	66,000,000	50,800,000
Supplementary Budgets	0	34,124,000
Summation	492,920,000	507,757,000

3.1.1.9 Facilities for Open Uses

The National Research Institute for Metals has opened its high field generation facilities of the High Magnetic Field Station since April 1998 to overseas and domestic researchers. Every year, proposals from external researchers are publicly invited in the autumn. The submitted proposals are examined and selected at the "users committee" including external researchers, and implemented in the form of joint research. Table 1 shows the magnets now open to the external users and responsible persons in charge. Details are shown in the NRIM's home page (<http://www.nrim.go.jp:8080/tml/tml/maglist.html>). The number of external applications for use of the facility was 68 in fiscal 1998 and will be more than 70 in fiscal year 1999.

The facilities for open uses such as the hybrid magnet are operated by an operation team, which consists of Akio Sato(Unit Leader), Bunmei Matsumoto, Hideo Nagai, Michio Kosuge and Michinari Yuyama. This team takes care of services such as the pre-cooling of superconducting magnets, or production of liquid helium for experiments.

(1) Magnet cooling and helium liquefaction

A helium refrigerator, 450 W/4 K, is used for operating the hybrid magnet and the 21 T superconducting magnet. This refrigerator has a liquefaction capacity of 150L/h. Helium lost due to gas leakage and consumed during gas refining is supplemented by purchased helium gas. A volume of 5,000Nm³ of gas is the annual supplement.

The liquid helium necessary for cooling the compact superconducting magnet and for low temperature experiments is supplied by a 250 l/h liquefier. Liquid helium of approximately 100,000 liter is annually supplied.

(2) Major high-field magnets

(i) 21 T superconducting magnet

This magnet provides a magnetic field up to 21 T. It is cooled with saturated superfluid helium (1.8 K), and can be used in various ways by changing the diameter of coils, such as the outer coil (15 T/314 mm ϕ), the middle coil (18 T/160 mm ϕ) and the inner coil (21 T/61 mm ϕ).

At present, a Bi-2212 double pancake coil is installed as the inner coil and a magnetic field of up to 21 T is steadily provided in a cold bore of 61 mm ϕ . This coil has demonstrated that a high field exceeding 20 T can be generated using an oxide superconducting coil.

(ii) Hybrid magnet

There are two laboratories in Japan (the NRIM, Institute for Materials Research of Tohoku University), two laboratories in Europe and one laboratory in the USA that have hybrid magnets capable of generating a field of 30 T or higher

At present our hybrid magnet is providing a magnetic field of up to 30 T; because spares for the water-cooled copper part are too expensive to be frequently destroyed.

The superconducting part of our hybrid magnet generates magnetic fields up to 15 T at 4.2 K (in a room temperature bore of 400 mm ϕ). The stored energy is 63.3 MJ. This is the largest in the world, among superconducting magnets generating a magnetic field of 14--15 T.

It is planned that the resistive part will finally generate a magnetic field of 25 T in a bore diameter of 30 mm ϕ , with a power consumption of 13.3 MW (40 T in total), and a magnetic field of 20 T in a bore diameter of 50 mm ϕ (35 T in total) with a power

consumption of 10.8 MW.

During development, a magnetic field of 36.5 T and a magnetic field of 32.3 T were generated in the inner diameter of 30 mm ϕ and the inner diameter of 50mm ϕ respectively in September 1995. These values are the world records for steady state magnetic fields for their respective inner diameters. In the future, the level of the whole system including the cooling system will be improved so that a magnetic field of 33 T or over can be steadily provided.

(iii) Resistive (Water-cooled) magnet

A resistive magnet was jointly developed with the National High Magnetic Field Laboratory (NHMFL) in the USA, providing a magnetic field of 25 T. The effective experimental space is 32 mm ϕ and the excitation speed is 5 T/min.

(iv) Pulsed field magnets

The pulse magnet is capable of providing a magnetic field up to 50 T. There are capacitor banks of 1.6 MJ (5 kV), 200 kJ (10 kV), and 50 kJ (2 kV) available. The magnet of a long pulse type capable of generating a magnetic field of 50T/100 msec inside a 15 mm ϕ cylinder and a magnet of the short interval repeating type capable of generating 30 T/10 msec in 30 mm ϕ are also available.

(v) High-precision superconducting magnets (6 sets)

Very uniform magnetic fields are useful for studying solid state physics. Superconducting magnets generating such uniform fields have been installed and are being used for low temperature physics measurements, such as NMR.

(vi) Superconducting magnets of compact size (4 sets)

In addition, small superconducting magnets are installed, which can be easily used for various purposes.

(3) International Cooperation (World network of high-field facilities)

The NRIIM has exchanged a Memorandum of Understanding with the National High Magnetic Field Laboratory, in Tallahassee, Florida, USA, and the European Joint Use High-Field Facility, in Grenoble, France. A similar MOU is in preparation with the High Field Magnet Laboratory of the University of Nijmegen, in the Netherlands. In

these MOUs, it is stated that joint programs are established by a joint coordinating committee. Based on such programs, the mutual utilization of and the joint development at the research facilities and the exchange of researchers shall be regularly carried out. The global network was thus established by the three major high-field facilities. These three parties also agree to hold joint symposiums and workshops regularly, on a take-turns basis.

Table 1: List of magnets

Magnets	Field (T)	Bore (mm ϕ)	Remarks
Large magnets			
21 SC	21	50	15T(314mm ϕ), 18T(160mm ϕ)
Hybrid	30	30/50	^3He cryostat
Resistive	25	32	cryostat(100mK)
Pulsed field magnets			
Long pulse	50	15	100mmsec
High repetition	30	30	10mmsec
High-precision SC magnets			
20T solid state physics(I)	20		dilution refrigerator
20T solid-state physics(II)	20		^3He cryostat
16T solid-state physics	16		dilution refrigerator
Solid-state NMR	15.5		^3He cryostat
Solution-state NMR(I)			500MHz
Solution-state NMR(II)			250MHz
SC magnets (compact)			
18T	18	50	temperature variable cryostat
15T split-paired	15	(25)	temperature variable cryostat
10T cryocooler-cooled(I)	10	100	vertical/horizontal field direction
10T cryocooler-cooled(II)	10	100	vertical/horizontal field direction

3.1.2 High Resolution Beam Research Station

Kazuo Furuya (Supervising Researcher)

3.1.2.1 Research work

The High Resolution Beam Research Station has been promoting the establishment of technologies for measurement and evaluation of material structures in an atomic scale, and of properties in high spatial and time resolution with the application of precise excitation of materials using electrons, ions, light, etc. Associated with the Center of Excellence Development Program (COE Project), direct observations and analysis of atomic structures and atom motions, and the fabrication and evaluation of new materials in extreme particle fields receive especially focused attention. The examples of the outcomes are shown below.

- (1) The atomic structure of nanocrystals in 1-10 nm diameter embedded into a metallic host matrix was directly observed for the first time. Their structural stability under electron and ion irradiation was evaluated using in-situ off-axial high-resolution transmission electron microscopy (HRTEM) with real-time video recording.
- (2) The position and size controlled single crystalline Si nanostructures were successfully fabricated in SiO₂ matrix for the first time by means of focused electron irradiation at high temperatures.
- (3) Research and development of ion technologies have been focused, including various in-situ measurements, and applied to study radiation damage and to develop metal nanocrystal systems.
- (4) Generation and utilization of excited neutral beams of thermal energy have been investigated in order to analyze and to process exactly the outmost surfaces of materials.
- (5) A new grazing-incidence X-ray spectro-reflectometer has been designed for the beamline 39XU, SPring-8. The commissioning was completed in May, 1998. The instrument is now available for public use in ultra trace analysis and the characterization of thin films. Very recently, we succeeded in the direct detection of 8×10^{-14} g selenium in 0.1 μ l solution. Interference effects in X-ray total reflection have been investigated in view of their application to thin film analysis. It has been found that the X-ray fluorescence spectrum from trace metals localized at a specific interface can be selectively enhanced.
- (6) An advanced Johansson-type X-ray fluorescence spectrometer has been developed, and some preliminary experiments have been performed at the SPring-8. It has

been shown that resonant X-ray fluorescence spectra, which contain much information on the chemical environment of each element, have been obtained even for a diluted system.

The members of High Resolution Beam Research Station, as at March 31, 1999, consisted of 12 researchers, 1 STA fellow, 5 COE fellows, 1 domestic research fellow, 2 special technical staff, 1 graduate student, 31 guest researchers and 2 secretaries.

The results of the research activities have been increased and presented, mostly at international meetings around the world, about half of these were the outputs from the COE project. The others were from the projects on nuclear materials, advanced beam technologies, and so on.

Under the COE project, scientific exchanges, including research collaborations and international meetings, have been stimulated. Twenty-three overseas researchers were invited and one international meeting was hosted at the NRIM. These activities clearly indicate the increasing role of the High Resolution Beam Research Station in the international scientific community.

3.1.2.2 Refined Beam Research Unit

Kazuo Furuya (Supervising Researcher), Yoshio Fukuda, Masaki Takeguchi, Kazutaka Mitsuishi, Miyoko Tanaka, Minghui Song (special technical staff), Yuan Wu (COE fellow), Guoqiang Xie (COE fellow), Yingda Yu (COE fellow), Qing Chen (COE fellow), Ulrich Dahmen (guest researcher), Stephen E. Donnelly (guest researcher), Peter A. Crozier (guest researcher), Robert C. Birtcher (guest researcher), Wolfgang Jäger (guest researcher), Charles W. Allen (guest researcher), Fu Rong Chen (guest researcher), Erik Johnson (guest researcher), Masami Terauchi (guest researcher), Nobuo Tanaka (guest researcher), Heishichiro Takahashi (guest researcher), Akira Hasegawa (guest researcher), Hideki Ichinose (guest researcher), Hiroyasu Saka (guest researcher), Hiroto Mori (guest researcher), Yoshitsugu Tomokiyo (guest researcher)

3.1.2.2.1 Research work

- (1) Atomic characterization and fabrication of nanocrystal structures, either embedded into host materials or on their surface, have been developed using transmission electron microscopy (TEM) in combination with stimulated decomposition, ion implantation and materials evaporation.

- (2) A 1000 keV high-voltage TEM system, to which dual ion implanters are attached, was developed and in-situ high-resolution TEM (HRTEM) observation was successfully demonstrated under simultaneous irradiation of ions and electrons.
- (3) The atomic structure of nanocrystals in 1-10 nm diameter embedded into metallic host matrix was directly observed for the first time. Their structural stability under electron and ion irradiation was evaluated using in-situ off-axial HRTEM with real-time video recording.
- (4) A 200 keV ultra-high vacuum field emission TEM (UHV-FE-TEM) system, with a molecular beam deposition apparatus and a UHV specimen transfer system for contamination free observation, was developed and the atomic structure of various non-passivated nanocrystals such as In, Pb, Ge, Mo, etc., investigated.
- (5) The position and size controlled single crystalline Si nanostructures were successfully fabricated in a SiO₂ matrix for the first time using focused electron irradiation at high temperatures with the UHV-FE-TEM.

<Research themes>

- (1) Analysis/Evaluation of Atomic Scale Compositional Change in Materials Due to the Radiation Damage
(Nuclear Energy Research: April 1995 to March 2000, Kazuo Furuya)
- (2) Characterization and Control of the Optoelectronic Properties of Small Crystalline Materials with Electron Probe Analysis
(Materials Research under Advanced Physical Field: April 1995 to March 2000, Kazuo Furuya)
- (3) Analysis and Characterization of Single Atom Layers and Dimensionally Controlled Materials
(Special Coordination Funds for Promoting Science and Technology: April 1995 to March 2000, Kazuhiro Yoshihara)
- (4) Joint Research on the Effects of Electron and Ion Irradiation on the Stability of Ion-implanted Nanocrystals Structure and Properties
(Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1998, Kazuo Furuya)
- (5) Workshop on the Structures and Properties of Multi-lattice Materials
(Special Coordination Funds for Promoting Science and Technology: February 1998, Kazuo Furuya)
- (6) Surface Analysis Database for Characterizing Unknown Materials
(Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1999, Kazuhiro Yoshihara)

- (7) Surface Chemical Analysis (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 2002, Kazuhiro Yoshihara)

3.1.2.2.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) M.Tanaka, K.Furuya, T.Saito: Microstructural Observation of Focused Ion Beam Modification of Silicide/Si Thin Films, *Mat. Res. Soc. Symp. Proc.*, **438**, 313-318 (1997)
- (2) M.Awaji, N.Ishikawa, K.Furuya: Modeling of Precipitated Xe Atoms in Aluminum and Fresnel Effect in HRTEM Imaging, *Nanostruct. Mater.*, **8**, 899(1997)
- (3) K.Furuya, M.Piao, N.Ishikawa, T.Saito: High-resolution Transmission Electron Microscopy of Defects Clusters in 316 Stainless Steel and Al During Electron and Low Irradiation at Room Temperature, *Mat. Res. Soc. Symp. Proc.*, **439**, 331-336 (1997)
- (4) M.Tanaka, K. Furuya, T.Saito: TEM Observation of FIB Induced Damage in $\text{Ni}_2\text{Si}/\text{Si}$ Thin Films, *Nucl. Instr. Meth. B*, **127/128**, 98(1997)
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- (6) Y.Fukuda, K. Furuya, N. Ishikawa, T.Saito: Aging Behavior of Photoluminescence in Porus Silicon, *J. Appl. Phys.*, **82**, 5718(1997)
- (7) M.Tanaka, K.Furuya, T.Saito: Focused Ion Beam Induced Formation of Pd Silicide, *J. Surf. Anal.*, **3**, 435(1997)
- (8) M.Song, Y.Fukuda, K.Furuya: Correlation Between Photoluminescence and Local Chemical States of Porous Si by Means of Electron Energy Loss Spectroscopy, *J. Surf. Anal.*, **3**, 446(1997)
- (9) K.Furuya, M.Song, Y.Fukuda, T.Noda, T.Saito: Chemical Analysis and High-resolution Transmission Electron Microscopy of Porous Silicon with a High-voltage TEM at 1000 keV, *J. Surf. Anal.*, **3**, 233(1997)
- (10) S.E.Donnely, K.Furuya, M.Song, R.C.Birtcher, C.W.Allen: 1 MeV Electron Irradiation of Solid Xe nanoclusters in Al - An In-situ HRTEM Study", *Proc. Intern. Centennial Symp. On Electron*, 306-312 (1998)
- (11) K.Furuya, M.Song, K.Mitsuishi, R.C.Birtcher, C.W.Allen, S.E.Donnely: Direct Imaging for Atomic Structure of Xe Nanocrystals Embedded in Al, *Proc. Intern. Centennial Symp. On Electron*, 341-347 (1998)
- (12) M.Song, W.Zhuo, Y.Fukuda, K.Furuya: Energy Loss Spectroscopy and Electron

- Microscopy of Photoluminescent P-type Porous Silicon Treated with NaOH Solution, *J. Surf. Anal.*, **4**, 365(1998)
- (13) M.Tanaka, K.Furuya, T.Saito: In-situ Observation of Focused Ion Beam Micropattern on Semiconductors and Insulators, *J. J. Appl. Phys.*, **37**, 7010(1998)
 - (14) M.Tanaka, K.Furuya, T.Saito: Focused Ion Beam Interface with 200 keV TEM for In-situ Micro Patterning to Semiconductors, *Microsc. Microanal.*, **4**, 207(1998)
 - (15) H.Hashimoto, H.Endoh, M.Hashimoto, Z.P.Luo, M.Song, Pseudo-aberration Free Focus Condition for Atomic Resolution Electron Microscope Images, *Micron*, **39**, 113(1998)
 - (16) Y.Fukuda, K.Furuya: Room-temperature Aging Effects on Porous Silicon Layer Chemical Bonds, *Hyoumen-gijutsu*, **49**, 63(1998)
 - (17) M.Tanaka, K.Furuya, M.Takeguchi: Fabrication and Structural Observation of Molybdenum and Indium Nanocrystals Deposited on Si(111) Thin Films by UHV-FE-TEM, *J. Surf. Anal.*, **4**, 287(1998)
 - (18) M.Tanaka, K.Furuya, M.Takeguchi, T.Honda: Surface Observation of Mo Nanocrystals Deposited on Si(111) Thin Films by a Newly Developed Ultrahigh Vacuum Field-emission Transmission Electron Microscope, *Thin Solid Films*, **319**, 110(1998)
 - (19) K.Furuya, M.Tanaka, M.Takeguchi: Ultrahigh-vacuum Field-emission Electron Microscope and Its Application to In-situ Observation of Indium and Molybdenum Nanocrystals, *Proc. 14th Inter. Cong. Electron Microsc.*, 513-514 (1998)
 - (20) K.Furuya, M.Song, K.Mitsuishi, C.W.Allen, R.C.Birtcher, S.E.Donnely: In-situ Observation of Xenon Nanocrystals in Aluminum Under Electron and Ion Irradiation in Transmission Electron Microscope, *Proc 14th Inter. Cong. Electron Microsc.*, 507-508 (1998)
 - (21) K.Mitsuishi, M.Song, K.Furuya, R.C.Birtcher, C.W.Allen, S.E.Donnely: In-site Observation of Atomic Self-organization Processes in Xe Nanocrystals Embedded in Al, *Mat. Rec. Soc. Symp. Proc.*, **504**, 417-422 (1998)
 - (22) M.Tanaka, K.Furuya, T.Saito: TEM Observation of FIB Micromilled Ni Silicides Thin Films, *Thin Solid Films*, **319**, 101(1998)
 - (23) M.Song, Y.Fukuda, K.Furuya: EELS and TEM Studies on NaOH Solution Treated P-type Porous Silicon after Anodization, *Proc. 14th Inter. Cong. Electron Microsc.*, 621-622 (1998)
 - (24) M.Tanaka, K.Furuya, M.Takeguchi, T.Honda: Surface Observation on Analysis by Ultrahigh Vacuum Field Emission Transmission Electron Microscope, *Proc. Inter. Centennial Symp. On Electron*, 586-592 (1998)

- (25) K.Furuya, N.Ishikawa, C.W.Allen: In-situ Observation of Shape and Atomic Structure of Xe Nanocrystals Embedded in Aluminum, *J. Microscopy*, **194**, 152 (1999)
- (26) K.Mitsuishi, M.Song, K.Furuya, R.C.Birtcher, C.W.Allen, S.E.Donnely: Observation of Atomic Processes in Xe nanocrystals Embedded in Al Under 1 MeV Electron, *Nucl. Instr. Meth. B*, **148**, 184(1999)
- (27) M.Song, K.Furuya, T.Tanabe, T.Noda, High-resolution Electron Microscopy of γ -TiAl and Al Irradiated with 15 keV Helium Ions at Room Temperature, *J. Nucl. Mater.*, **271/272**, 200(1999)
- (28) M.Takeguchi, K.Furuya, K.Yoshihara: EELS Study of the Formation Process of Si Nanocrystals in SiO₂ Due to ESD-decomposition, *Micron*, **30**, 147(1999)
- (29) K.Mitsuishi, M.Song, M.Takeguchi, K.Furuya: HRTEM and HAADF-STEM Observation of Xe Nanocrystals Formed by Ion-implantation in HVTEM, *J. Electron Microsc.*, (in press)
- (30) M.Song, K.Mitsuishi, K.Furuya: Electron Energy Loss Spectroscopy and Energy Filtered Imaging of Xe Cavities Embedded in Al at 298 and 473 K, *Micron*, **30**, 129 (1999)
- (31) M.Takeguchi, M.Tanaka, K.Furuya: Fabrication and Analysis of Nanometer Size Structure of Semiconductor by UHV FE-TEM, *Appl. Surf. Sci.*, **146**, 257(1999)
- (32) K.Mitsuishi, M.Kawasaki, M.Takeguchi, K.Furuya: Determination of Atomic Positions in a Solid Xe Precipitate Embedded in an Al Matrix, *Phys. Rev. Lett.*, **82**, 3082(1999)
- (33) Q.Chen, M.Tanaka, K.Furuya: Application of a UHV-MBE-TEM System on the Deposition and HRTEM Study of Indium Nanocrystals, *J. Surf. Anal.*, **5**, 348 (1999)
- (34) Q.Chen, M.Tanaka, K.Furuya: Unusual Crystallographic Structure and Its Fluctuation of Indium Nanoparticles As-deposited and Observed with HRTEM Using UHV-DC-TEM System, *Surf. Sci.*, (in press)
- (35) Y.Fukuda, M.Song, K.Furuya: Photoluminescence Change of As-prepared and Aged Porous Silicon with NaOH Treatment, *J. Electrochem. Soc.*, (in press)
- (36) K.Furuya, K.Mitsuishi, N.Ishikawa, C.W.Allen: Imaging and Modeling of Nano-Crystalline Xe in Al Containing Defects, *Mater. Sci. Engr. A*, (in press)
- (37) C.W.Allen, M.Song, K.Furuya, R.C.Birtcher, S.E.Donnely, K.Mitsuishi: Behavior of Nanocrystalline Xe Precipitates in Al, *J. Electron Microsc.*, (accepted)
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- (39) M.Takeguchi, K.Furuya, K.Yoshihara: Novel Fabrication Technique of Si Nanoparticles as Applied to Atomic-resolved Observation of the Nanoparticle Surfaces, J. Electron Microsc., (in press)
- (40) K.Furuya, K.Mitsuishi, M.Song, T.Saito: In-situ, Analytical, High-voltage and High-resolution Transmission Electron Microscopy of Xe Ion Implantation into Al, J. Electron Microsc., (in press)
- (41) M.Tanaka, M.Takeguchi, K.Furuya: In-situ Deposition of Metal Nanocrystals on Si(111) Thin Film Surface in UHV-FE-TEM, Surf. Sci., (in press)
- (42) K.Furuya, K.Mitsuishi, M.Song, T.Saito: In-situ Ion Implantation of Xe into Al with a High-resolution and High-voltage Electron Microscope, IEEE Transactions, (in press)
- (43) M.Song, K.Furuya, T.Tanabe, T.Noda: High-resolution Electron Microscopy of γ -TiAl In-situ Irradiated with 15 keV Helium Ions in HVTEM, J. Electron Microsc., (in press)
- (44) M.Tanaka, K.Furuya, T.Saito: In-situ Observation of Focused Ion Beam Micromilled Si, SiO₂ and GaAs, IEEE Transactions, (in press)

Oral Presentation: 79

- (1) International Conference: 19
- (2) Domestic Conference: 60

Patent Applications

- (1) Fabrication Method of Si Nanocrystals, Kazuo Furuya, Masaki Takeguchi, Kazuhiro Yoshihara, Domestic Patent, H10-186637, July 1, 1998
- (2) Fabrication Method of Si Nanocrystals, Kazuo Furuya, Masaki Takeguchi, Kazuhiro Yoshihara, EC and USA Patent Application, June 22, 1999

Award

- (1) Best Poster at MRS Fall Meeting 1997, Kazutaka Mitsuishi et al., December 1997
- (2) Commendation for Achievements, Kazuo Furuya, May 1998
- (3) Ichimura Prize, Kazuo Furuya, April, 1999

3.1.2.3 Hybrid Beam Research Unit

Naoki Kishimoto (Unit Leader), Hiroshi Amekura, Yoshihiko Takeda, Kenichiro

Kono, Chi-Gyu Lee (special technical staff), Thi Thi Lay (domestic research fellow), Naoki Umeda (joint-doctoral course student), Zhao Jianping (COE fellow), Haruo Yokomichi (guest researcher), Vasily T. Gritsyna (guest scientist), Lee Heatherly (guest scientist), Timothy J. Renk (guest scientist), Oleg A. Plaksin (guest scientist), Volodymyr Voitsenya (guest scientist)

3.1.2.3.1 Research work

- (1) A hybrid particle field of ions and photons, particularly an extreme particle field (EPF), has been developed employing a high-current heavy-ion accelerator (1 mA) and a high-flux YAG laser (5 J/pulse). Material irradiation techniques have been attained for extremely non-equilibrium processes under the EPF.
- (2) In-situ detection techniques, under high-energy ion bombardment, have been developed and applied to study radiation damage on optoelectronic properties of crystalline and amorphous semiconductors. The in-situ methods employed are particle-induced conductivity, DLTS, ion-induced photon emission, etc.
- (3) A quantum-dot system, metal nanoparticles embedded in insulators, has been fabricated by high-current negative-ion implantation. Kinetic control of nanoparticle morphology has succeeded in creating a self-assembled two-dimensional nanoparticle structure.
- (4) Quantum effects, i.e., nonlinear and ultra-fast optical transitions, of nanoparticle systems, have been evaluated with degenerate-four-wave mixing and the femto-second pump-probe methods, respectively. Large optical nonlinearity of the controlled nanostructure is promising for photonic applications.

<Research themes>

- (1) Study on Detection and Evaluation of Radiation Damage in Extreme Particle Fields
(Nuclear Energy Research: April 1992 to March 1999, Naoki Kishimoto)
- (2) Measurements of Quantum Effects Using High Resolution Beam Probes
(Special Coordination Funds for Promoting Science and Technology: April 1995 to March 2000, Kazuhiro Yoshihara)
- (3) Study on Detection, Evaluation and Utilization of Non-equilibrium Processes Under Extreme Particle Fields
(Nuclear Energy Research: April 1999 to March 2004, Naoki Kishimoto)

3.1. 2.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) N. Kishimoto, V.T. Gritsyna, K. Kono, H. Amekura, T. Saito: Negative Copper Ion Implantation into Silica Glasses at High Dose Rates and the Optical Measurements, *Nucl. Instr. & Meth.*, **B127/128**, 579 (1997)
- (2) K. Kono, N. Kishimoto, H. Amekura, T. Saito: Evaluation of Proton-induced Deep Levels in N-Si, *Mat. Res. Soc. Symp. Proc.*, **442**, 287 (1997)
- (3) N. Kishimoto, V.T. Gritsyna, K. Kono, H. Amekura, T. Saito: High Current Implantation of Negative Copper Ions into Silica Glasses, *Mat. Res. Soc. Symp. Proc.*, **438**, 435 (1997)
- (4) N. Kishimoto, H. Amekura, K. Kono, T. Saito: Particle-induced and Photo-conductivities in Amorphous Si:H Under Proton Irradiation, *Mat. Res. Soc. Symp. Proc.*, **439**, 679 (1997)
- (5) N. Kishimoto, V.T. Gritsyna, K. Kono, H. Amekura, T. Saito: Material Modification of Optical Properties Due to Intense Negative Cu Ions, *IONICS*, **23**, 95 (1997)
- (6) H. Amekura, N. Kishimoto, K. Kono: Persistent Excited Conductivity Induced by Proton Irradiation in a-Si:H, *Materials Science Forum*, **258-263**, 599 (1997)
- (7) N. Kishimoto, H. Amekura, K. Kono, C.G. Lee: Stable Photoconductivity in Metastable a-Si:H Under High-energy Proton Irradiation, *J. Non-cryst. Solids*, **227-230**, 238 (1998)
- (8) H. Amekura, N. Kishimoto, K. Kono: Radiation-induced Two-step Degradation of Si Photoconductors and Space Solar Cells, *IEEE Trans. Nuclear Science*, **45**, 1508 (1998)
- (9) N. Kishimoto, V.T. Gritsyna, Y. Takeda, C.G. Lee, T. Saito: Dose-rate Dependence of Negative Copper Ion Implantation into Silica Glasses and Effects on Colloid Formation, *Nucl. Instrum. & Method in Phys. Res.*, **B141**, 299 (1998)
- (10) N. Kishimoto, H. Amekura, K. Kono, C.G. Lee: Radiation Resistance of Amorphous Silicon in Opto-electric Properties Under Proton Bombardment, *J. Nucl. Mater.*, **258-263**, 1903 (1998)
- (11) N. Kishimoto, H. Amekura, K. Kono, C.G. Lee: Applicability of Shallow-impurity Doped Silicon to Proton-flux Sensors Using Stable Particle-induced Conductivity, *Mat. Res. Soc. Symp. Proc.*, **487**, 423 (1998)
- (12) H. Amekura, A. Eckau, R. Carius, Ch. Buchal: Visible Photoluminescence from Tb-Ions Implanted in SiO₂ Film and Its Concentration Dependence, *Rare Earths*, **32**, 146 (1998)
- (13) N. Kishimoto, V.T. Gritsyna, Y. Takeda, C.G. Lee: Fabrication of Metal Nanospheres and the Kinetics Controlled with High-flux Negative Ions and the Optical Properties, *J. Surf. Anal.* **4**, 220 (1998)

- (14) H. Amekura, A. Eckau, R. Carius, Ch. Buchal: Room Temperature Photoluminescence from Tb-ions Implanted in SiO₂ on Si, J. Appl. Phys., 84, 3867 (1998)
- (15) H. Amekura, N. Kishimoto, K. Kono: Particle-induced conductivity and photoconductivity of silicon under 17 MeV-proton irradiation, J. Appl. Phys., 84, 4834 (1998)
- (16) N. Kishimoto, V.T. Gritsyna, Y. Takeda, C.G. Lee, N. Umeda, T. Saito: Nanocrystal Growth at High Dose Rates in Negative Copper-ion Implantation into Insulators, Mat. Res. Soc. Symp. Proc., 504, 345 (1999)
- (17) K. Kono, N. Kishimoto, H. Amekura: In-situ DLTS Measurement of Metastable Coupling Between Proton-induced Defect and Impurities in N-Si, Mat. Res. Soc. Symp. Proc. 504, 15 (1999)
- (18) S. Wang, H. Amekura, Eckau, R. Carius, Ch. Buchal: Luminescence from Er and Tb, Implanted into MOS Tunnel Diodes, Nucl. Instr. & Meth, B148, 481 (1999)
- (19) N. Kishimoto, N. Umeda, Y. Takeda, C.G. Lee, V.T. Gritsyna: Self-assembled Two-dimensional Distribution of Nanoparticles with High-current Cu⁺- implantation into Insulators, Nucl. Instr. & Meth, B148, 1017 (1999)
- (20) Y. Takeda, C.G. Lee, N. Kishimoto, N. Umeda, V.T. Gritsyna: Linear and Nonlinear Optical Properties of Cu Nanoparticles Fabricated by High-current Cu-implantation in Silica Glass, Nucl. Instr. & Meth, B148, 1029 (1999)
- (21) N. Kishimoto, N. Umeda, Y. Takeda, C.G. Lee, V.T. Gritsyna: Nanocrystal Growth in Crystalline Insulators Irradiated with High-Current Copper-Ions, Mat. Res. Soc. Symp. Proc., 540, 153 (1999)
- (22) K. Kono, H. Amekura, N. Kishimoto: Flux-dependent Generation Rate of Irradiation-Induced Defect Clustering in N-Si Under 17 MeV Proton Irradiation, Mat. Res. Soc. Symp. Proc., 540, 115 (1999)
- (23) H. Amekura, A. Eckau, R. Carius and Ch. Buchal: Visible Photoluminescence from Tb³⁺ Ions Implanted in a SiO₂ Film on Si at Room Temperature, Proc. 12th Int. Conf. on Ion Implantation Technology (1999), in press
- (24) N. Kishimoto, Y. Takeda, V.T. Gritsyna, E. Iwamoto, T. Saito: A High-current Negative-ion Implanter and Its Application for Nanocrystal Fabrication in Insulators, Proc. 12th Int. Conf. on Ion Implantation Technology (1999), in press

Oral Presentation : 48

- (1) International Conference: 16
- (2) Domestic Conference: 32

Registered Patent

- (1) Radiation Resistant Optical Sensor of Impurity Compensation Type, N. Kishimoto, H. Amekura, Pat. No.2884037, Feb. 12, 1999.

3.1.2.4 Neutral Beam Unit

Yasushi Yamauchi (Unit Leader), Mitsunori Kurahashi

3.1.2.4.1 Research work

Versatile technologies to generate and utilize excited neutral beams have been developed. An intense pulsed metastable helium atom beam, at the thermal energy level, was obtained by invention of the pulsed discharge method. The beam has proved to be a promising probe to get information from exactly the outmost surfaces by practical applications. The technologies have been extended to spin polarization.

<Research themes>

- (1) Development of Fundamental Technologies for Excited Neutral Beams
(Nuclear Energy Research: April 1997 to March 2002, Yasushi Yamauchi)
- (2) Creation of Advanced Materials with Atomic-Scale Structures Exhibiting Quantum Phenomena
(Special Coordination Funds for Promoting Science and Technology: April 1995 to March 2000, Kazuhiro Yoshihara)

3.1.2.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) Y. Yamauchi, M. Kurahashi, N. Kishimoto: Generation of Helium Metastable Atom Beams by Pulsed Discharge, Proc. 8th Symp. on Beam Engineering of Advanced Material Syntheses, 55 (1997)
- (2) M. Kurahashi, Y. Yamauchi: A Metastable-Atom Deexcitation Spectroscopy (MDS) Study on the First Atomic Layer of a Polycrystalline Titanium Surface, Mat. Res. Soc. Symp. Proc. 501, 67 (1998)
- (2) Y. Yamauchi, M. Kurahashi, N. Kishimoto: A Metastable Helium Atom Source Directly Pulsed by a Nozzle-Skimmer Discharge, Meas. Sci. Technol. **9**, 531 (1998)
- (3) Y. Yamauchi, M. Kurahashi, N. Kishimoto: A Pulsed Helium Metastable Atom Source Using Hollow Cathode Discharge, Proc. 9th Symp. on Beam Engineering of

Advanced Material Syntheses, 39 (1998)

- (4) M. Kurahashi, Y. Yamauchi: A Metastable De-excitation Spectroscopy (MDS) Study on Oxygen Adsorption on a Polycrystalline Zirconium Surface, Surf. Sci. 420, 259 (1999)
- (5) M. Kurahashi, Y. Yamauchi: Metastable Deexcitation Spectroscopy Study of Oxygen Adsorption on a Polycrystalline Titanium Surface, J. Vac. Sci. Technol. A 17, 1047 (1999)

Oral Presentation: 13

- (1) International Conference: 2
- (2) Domestic Conference: 11

Patent Application

- (1) Method of Generating Pulsed Metastable Atom Beam and Pulsed Ultraviolet Radiation and Its Device, Y. Yamauchi, M Kurahashi, N. Kishimoto, 6/30/1998, 1998-Patent-Application-019922.
- (2) Method of Generating Pulsed Metastable Atom Beam and Pulsed Ultraviolet Radiation and Its Device, Y. Yamauchi, M Kurahashi, N. Kishimoto, 9/29/1998, U.S.-Patent- Application- S.N. 09/161,718.

3.1.2.5 Advanced Photon Beam Unit

Kenji Sakurai (Unit Leader), Luc Ortega (STA fellow), Hiromi Eba, Krassimir Stoev (guest researcher), Sunil Deshpande (guest researcher), Weimin Chen (guest researcher), Dilip Parwate (guest researcher), Claude Landron (guest researcher), Masayuki Kawai (guest researcher), Masahiko Kurakado (guest researcher), Junji Ihara (guest researcher)

3.1.2.5.1 Research work

Synchrotron radiation is an extremely attractive tool for analysis, with super precision and high resolution, to evaluate the micro- and nano- meter scale structure of materials. The laboratory has been involved in the construction and commissioning of the beamline at the SPring-8, which is a new brilliant synchrotron facility just brought into service in October 1997. One of the major activities for these recent 2 years is development of a new grazing-incidence X-ray spectro-reflectometer for ultra trace

determination and surface/interface analysis of thin films using the total-reflection of X-rays. The instrument is now completed and is available for public use at the beamline. Another important work was X-ray absorption spectroscopic studies. The nano-meter scale structure of materials, especially, complex oxides has been investigated.

<Research themes>

- (1) Development of Analytical Techniques for Characterization of Nuclear Materials Using New Generation Synchrotron X-rays
(Nuclear Energy Research: April 1994 to March 1999, Kenji Sakurai)
- (2) Advanced Characterization of Micro and Nano Meter Scale Structure of Materials by Brilliant Synchrotron X-rays at the SPring-8
(Materials Research Using Synchrotron Radiation: April 1997 to March 2002, Kenji Sakurai)

3.1.2.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

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- (2) K.Sakurai: Total-reflection X-ray Fluorescence Imaging, *Spectrochim. Acta B*, in press
- (3) K.Sakurai, H.Eba, S.Goto: Grazing Incidence X-ray Fluorescence and Scattering Experiments at BL-39XU, SPring-8, *Jpn. J. Appl. Phys., Suppl.*38-1, 332(1999)
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 - (13) K.Hayashi, T.Yamamoto, J.Kawai, M.Suzuki, S.Goto, S.Hayakawa, K.Sakurai, Y.Gohshi, Atomic-Resolution X-ray Fluorescence Holography of Zn (0.02wt%) in a GaAs Wafer, Anal. Sci., **14**, 987 (1998)
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 - (20) M.Harada, K.Sakurai: Fast Detector Electronics for High-counting Rate X-ray Measurement, Adv. in Chem. X-ray Anal. in Japan, **28**, 277-288 (1997) (in Japanese)
 - (21) Kenji Sakurai, Krassimir Stoev: Review: Total-reflection X-Ray Fluorescence, Bunseki, No.7, 575(1997) (in Japanese)

Oral Presentation: 44

- (1) International Conference: 12

(2) Domestic Conference: 32

Patent Application

- (1) Synthesis Method for Yttrium Aluminum Complex Oxides, Kenji Sakurai, Xiaomei Guo, July, 29, 1998, H10-214778
- (2) X-ray Imaging Method and the Instruments, Kenji Sakurai, Hiromi Eba, August 13, 1998, H10-229180

Award

Ichimura Prize, Kenji Sakurai, April, 1997

3.1.2.6 Researchers in the High Resolution Beam Research Station

Age and research fields of the researchers in the High Resolution Beam Research Station

Age of Researchers	Number of Researchers	Materials Science	Physics	Chemistry
60 ~	1 (2)	1 (1)	(1)	
50 ~ 60	2 (10)	1 (7)	(3)	1
40 ~ 49	3 (10)	1 (5)	2 (4)	(1)
30 ~ 39	13 (9)	4 (2)	8 (6)	1 (1)
22 ~ 29	3	1	1	1
Total	22 (31)	8 (15)	11 (14)	3 (2)

(): short time visit

3.1.2.7.3 Research Budgets in the High Resolution Beam Research Station

Research budgets in the High Resolution Beam Research Station (yen)

Budgets	1997	1998
Materials Research under Advanced Physical Field	33,109,000	32,343,000
Nuclear Energy Research	181,171,000	189,937,000
Materials Research using Synchrotron Radiation	29,480,000	28,825,000
Special Coordination Funds for Promoting Science and Technology	138,896,000	116,079,000
Total	382,656,000	367,184,000

3.1.2.8 Near Future Scope of the High Resolution Beam Research Station

To extend the field of high-resolution beam research in the near future, it is necessary to continue the development of new and advanced equipments based on individual beam technology, such as high-resolution electrons, high-intensity ions, spin-polarized neutral beams, and high-speed and high-intensity photons. Especially, focusing on the nanometer scale structure, will become crucial to contribute to the discovery of new materials with reliable strength and a very stable, long life as well as unusual physical and/or chemical properties. The following research will be pursued in the next stage. The development of a fine electron probe less than 0.1 nm in diameter which can be used for annular-dark field scanning TEM and the fabrication of position and size controlled structures.

The non-equilibrium process under extreme particle fields is studied as the basis for the following, measurement of damage in extreme environments and the creation of nanostructured quantum materials,

Exploiting a new field of spin dependent surface phenomena by means of spin labeling techniques.

Advanced ultra fast time resolved and in-situ optical measurement including magneto-optic effect.

Brilliant synchrotron X-ray beamlines at the SPring-8 will make it possible to see what has been difficult to study so far because of very weak cross section imaging. Some specific challenges, such as ultra trace analysis in the order of sub-fg using total-reflection condition, chemical characterization by resonant X-ray fluorescence, and X-ray microscope with sub-micron resolution will be achieved in the next 5-7 years. Further development of the experimental techniques and instruments, including the construction of a new dedicated beamline will be required.

It is also very important to collaborate with researchers worldwide for increasing the impact of research and development at a high scientific level. As one of research groups which is opened towards the world, the production of new results and the discussion must be activated more through planning of, and participation in, scientific meetings.

3.1.3 Extreme High Vacuum Research Station

Nobuyuki Koguchi (Supervising Researcher)

3.1.3.1 Research work

Following the 5th Long-term Plan, R & D for an extremely high vacuum (XHV) integrated system in which several sample fabrication and surface analysis systems are combined through XHV transfer lines, have been carried out. The R & D for the improvement of surface analysis in XHV and the research for fabrication and characterization of novel nanostructures in XHV environments have been promoted at the Extreme High Vacuum Research Station. Examples of the consequences are shown next.

- (1) Surface modification techniques for vacuum motion material with a low friction coefficient were developed in the **Extreme High Vacuum and Its Application Unit**.
- (2) Novel techniques of making nano-scale metal wires by using STM or AFM were developed in the **Atomic-Scale Phenomena Unit**, and the electron transport in quantum wires with a tunneling barrier was studied.
- (3) Novel fabrication methods for semiconductor quantum dots was established in the **Surface New Material Unit**, and also a new system for real-time in-situ STM observation of adatoms on the MBE growth front was developed.
- (4) New phenomena of surface segregation for metal atoms on metal film were discovered in the **Surface and Interface Analysis Unit**, and work function change of metal by the surface segregation phenomena was studied.

The members of the Extreme High Vacuum Research Station, as at March 31, 1999, consist of 12 researchers, 7 COE fellows, 3 STA fellows, 2 domestic research fellows, 1 joint-doctrual course student, 1 graduate student and 3 secretaries.

The output of the research activities has been increased since the start of the 5th long-term plan. The number of publications, including submitted papers, is about 80, between April, 1997 to March, 1999. The details are shown in the research activities of each unit.

3.1.3.2 Extreme High Vacuum and Its Application Unit

Masahiro Tosa (Unit Leader), Akira Kasahara, Kim Young Sung (COE fellow),
Lee Kyung Sub (COE fellow)

3.1.3.2.1 Research work

The Extreme High Vacuum and Its Application Unit has built the stable generation system of extreme high vacuum, that is clean in an atomic scale and has developed advanced surface modification methods by means of XHV.

- (1) The obstacle of fabrication of nano-structures is atomic lattice matching between consistent materials. The lattice mismatching condition will be drastically relaxed in the crystal growth structure with van der Waals interactions. Boron nitride with a hexagonal structure (h-BN) has the c plane with van der Waals interaction. The c surface of h-BN basal plane layer parallel to the base substrate can offer van der Waals gap at the interface between grown material and the layer with little atomic mismatching. Research has been carried out on fabrication of the advanced substrate for an atomically controlled structure and the application of the advanced substrate to the fabrication of nano-materials, using the super clean environment by the XHV integrated process.
- (2) Hexagonal boron nitride (h-BN) has a hexagonal crystal plane with a little bonding force and displays a low friction coefficient at atmospheric pressure, but the friction coefficient increases as the pressure decreases and shows high value, in an ultra high vacuum. The unit has studied the control of the structure and concentration with the co-sputtering deposition method by making use of the self-organization of surface segregation from the mixture film of h-BN and copper and then developed the surface modification technology for vacuum motion materials with a low friction coefficient.
- (3) The unit has studied the development of the surface modification method for vacuum chamber materials that is suitable for the standard pressure field for the methodology of the vacuum gauge. The unit has also studied the interaction of hydrogen atoms and the chamber wall materials, i.e., hydrogen desorption, hydrogen atom penetration, coupling and so on.

<Research themes>

- (1) Advanced Substrate for Quantum Effect of Nanostructures (COE Project: April 1995 to March 2000, Masahiro Tosa)
- (2) Analysis and Control of Self Organization of Materials (Special Coordination Funds for Promoting Science and Technology: April 1999 to March 2001, Masahiro Tosa)
- (3) Standardization of Vacuum Pressure Measurement (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1999, Masahiro Tosa)

3.1.3.2.2 Research products, including papers submitted (April 1997 to March 1999)

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- (2) M.Tosa, A.Kasahara, K.Yoshihara: Substrate Transfer Operation with XHV Continuous Integrated Process Using Levitation Transport System, *J.Vacuum.Sci.Jpn*, **41**, 340 (1998) (in Japanese)
- (3) M.Tosa, A.Kasahara, K.Yoshihara: Tribological Properties of Vacuum Materials, *ibid.* **41**, 363 (1998) (in Japanese)
- (4) M.Tosa, A.Kasahara, A.Itakura, K.Yoshihara: Advanced Substrate for Fabrication of Nanostructures with XHV Integrated Process, *J.Surf. Anal.* **4**, 320 (1998)
- (5) M.Tosa, A.Kasahara, K.Yoshihara: XHV Integrated Process with Magnetic Levitation Transports, NASA/Cp-1998-20765, Proc. 4th Inter. Symp. on Magnetic Suspension Technology. 179 (1998)
- (6) K.S.Lee, M.Tosa, J.C.Ro, S.M.Ra, S.J.Suh: Improvement of Coercity and Microstructure of NdFeB Melt-spun Ribbons by Addition of Zn, *J. Magn. Soc. Jpn*, **23**, 288 (1999) (in Japanese)
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- (8) A.Kasahara, M.Tosa, K.Yoshihara: Friction Properties of Vacuum Materials. *ibid.* **42**, 447 (1999) (in Japanese)

Oral Presentation: 14

- (1) International Conference: 4
- (2) Domestic Conference: 10

Patent Application:

- (1) Surface Modified Vacuum Material and Its Modification Method, M.Tosa, A.Kasahara and K.Yoshihara, 9 Dec 1999, Patent No. 10-35033

3.1.3.3 Atomic-Scale Phenomena Unit

Hitoshi Nejo (Unit Leader), Daisuke Fujita, Dong Zhen-Chao, Takashi Uchihashi, Taro Yakabe, Taizo Ohgi (COE researcher), Yasushi Sozu (COE researcher), Pavel Dorozhkin (COE researcher), Wenli Deng (COE researcher) , Katsuki Amemiya (JST researcher), Norifumi Yamada (JST researcher) , Urs Ramsperger (STA fellow), Stephane Odasso (STA fellow)

3.1.3.3.1 Research work

(1) Surface Conductivity Measurements of Lead Nanostructures onto Ideal Si(111)-(1x1):H Templates

Our final objective is to perform surface conductance measurements at liquid helium temperature on Si(111) substrates supporting lead nanowires. A first concrete advance towards such a realization is the feasibility of forming depassivated regions onto a hydrogenated silicon surface by the application of high voltages and high demand tunneling currents between a scanning tunneling microscope tip and a silicon specimen during scanning various geometrical shapes. As a matter of fact, different patterns have been previously obtained with this method, the optimum efficiency for selective desorption of hydrogen being reached for: sample voltage = + 8V and $1\text{ nA} < \text{tunnel current} < 10\text{ nA}$. Our efforts were especially devoted to the formation of depassivated squares at each ends of a silicon nanoline.

From such reliable results, we could develop a novel nanostructuring process consisting of the condensation, in situ, of half a monolayer of Pb onto a clean Si(111) surface after selective desorption of passivated nanometer-sized areas exhibiting Pb clusters on top. Hydrogen extraction was engaged for $+1\text{V} < \text{sample voltage} < +10\text{V}$ and $1\text{ nA} < \text{tunnel current} < 10\text{ nA}$ with the feedback function still active.

Very recently, we focused our research activities on fabrication of novel depassivated areas and the improvement of depassivation conditions. The challenge is not only to minimize the presence of superficial clusters induced by extraction of hydrogen but also to increase laterally the extent of the depassivated regions. As a first consequence, depassivated areas can be confined to a single huge passivated terrace on Si(111) samples with high resistivity ($1000\text{ ohm.cm} < \text{resistivity} < 1500\text{ ohm.cm}$), opening a promising way towards further electron transport measurements in group IV nanostructures, and providing the possibility to find the structure by UHV SEM before performing surface conductivity measurements with our four-point probe machine.

Our next activities will concentrate on the fabrication of Pb nanostructures and on the measurements of their related electron transport characteristics onto highly

resistive Si(111) samples.

(2) Fabrication of Nano-scale Wires using Tip Contact

We have developed a novel technique of making nano-scale wires in UHV. A piezoresistive cantilever, whose tip was coated with gold beforehand, is brought into contact with a sample surface and is moved laterally. The gold is transferred from the tip onto the surface, forming a nano-wire. After wire fabrication, the sample surface is imaged by non-contact mode Atomic Force Microscope (AFM) using the same cantilever. The minimum line width of gold wire fabricated by this technique is 32 nm.

This technique has the following advantages: 1) Since an AFM cantilever is used instead of an STM tip, insulator can be adopted as a substrate. This is important because our target is to measure electric conductance through a nano-structure. 2) It is easy to realize and reliable. Since significant amount of Au is coated on the cantilever, it can be used for a relatively long time and then nano-wires can be fabricated over a long range. We do not need any other special device or techniques. 3) It can produce continuous metal wires with a μm -scale length, which would be hard to realize by other techniques. This helps to connect nanowires or other nanostructures to a macroscopic electric pad.

(3) Wire Connection to a Pad

We also succeeded in connecting a gold nano-wire to a silver pad on an Si(111) sample surface. The pad was fabricated by thermal evaporation through a through-hole mask prior to the wire fabrication. A straight line protruding from the surface is clearly seen in proximity of the pad. This result is quite important in terms of measuring electron conduction through nano-structures. The silver pad will be used as an electrode through which the conductance of a gold wire will be measured by the four point probe method.

(4) Electron Transport and Interference Phenomena in Quantum Wires with a Potential Barrier in Magnetic Fields

We have studied the electron transport in quantum wires with a tunneling barrier. The calculated results show that the conductance of these systems decreases to zero repeatedly with increasing magnetic fields. We found that this is caused by the interference of the evanescent modes with complex wavenumbers in the tunneling region. Although the behavior is aperiodic, this recalls the Aharonov-Bohm (AB) oscillation of conductance in magnetic fields. In order to clarify the relation between

these, we consider the electron transport in a quantum wire with an antidot.

We calculated the conductance of a wire with a square-shaped antidot at the center. The antidot is formed by the potential that is larger than the Fermi energy of electrons and the length of its side is a half of the width of the wire. The electron paths at the side of the antidot are so narrow that the electrons transport through them by tunneling. We found the repeated suppression of conductance also in this system. However, the intervals between the minima are much smaller than the period estimated from the magnetic flux in the antidot region. Since the magnetic field is applied to the whole system, as well as the electron path, the situation of this case is pretty different from a usual AB effect setup. Therefore the magnetic flux around the antidot may also contribute to the interference pattern found here. By studying these systems we expect that the relation between the quasi-periodic behavior of the conductance of tunneling barriers and the AB oscillation can be revealed.

<Research themes>

- (1) Evaluation of Super-structures on Ultra-clean Solid Surface and Research for Fabrication of Dimension-controlled Nano-material

(Special Research: April 1995 to March 2000, H.Nejo)

- (2) Fabrication of Nanostructures on Surface in Extremely High Vacuum Surface

(COE Project: April 1995 to March 2000, H.Nejo)

- (3) Construction of Molecule-Harmonic Structures and Development of Electro-magnetic Field Control Devices

(Organized Research Combination System: April 1998 to March 2003, H.Nejo)

3.1.3.3.2 Research products, including papers submitted (April 1997 to March 1999)

- (1) N.Yamada: "Is tunneling time definable?", *Cold Neutron Interferometry and Its Applications(III)*, Research Reactor Institute, Kyoto University, March (1999), P.1-7 (
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 - (14)T. Ohgi, H.-Y. Shend, H .Nejo: Au Particle Deposition onto Self-assembled Monolayers of Thiol and Dithiol Molecules, Appl. Surf. Sci. 130-132, 919 (1998)
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- (20) T.Yakabe, Z.C.Dong, H.Nejo: Observation of Negative Differential Resistance on Ag/Si(100) using STM, *Appl. Surf. Sci.* **121/122**, 187(1997)
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- (27) T.Yakabe Z.-C.Dong, H.Nejoh: Surface Structure of Initial Ag Deposition on Si(100) and its Local Density of State, *Appl. Surf. Sci.* , **121/122** ,187(1997)
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Oral Presentation: 32

- (1) International Conference: 15
- (2) Domestic Conference: 17

3.1.3.4 Surface New Material Unit

Nobuyuki Koguchi (Supervising Researcher), Toyohiro Chikyow, Shiro Tsukamoto, Keiko Ishige, Katsuyuki Watanabe (joint-doctral course student), Takaaki Mano (graduate student), Amane Shikanai (graduate student)

3.1.3.4.1 Research work

Predictions of an enhanced electron mobility device and advanced semiconductor laser with highly-monochromized and low threshold current density caused by the quantum size effect have been made for applications of semiconductor nm-structures, especially so-called quantum dots structures.

We have proposed a new method to fabricate compound semiconductor quantum dots directly without using any lithography. The process consists of forming numerous III-column element droplets, such as Ga or InGa, with homogeneous size of around 10 nm on the surface first by supplying their molecular beams, and then reacting the droplets with As molecular beam to produce GaAs or InGaAs epitaxial microcrystals. The quantum dots systems of InGaAs/GaAs and GaAs/AlGaAs showed strong photoluminescences caused by quantum size effect. This method, termed Droplet Epitaxy, is promising for the fabrication of compound semiconductor quantum dots, not only in a lattice-matched system but also in a lattice-mismatched system.

Surface treatments for III-V compound semiconductors increase their importance in practical applications because the surface treatments are crucial in increasing photoluminescence intensity, Schottky-barrier height controlling and reducing surface states density of semiconductor surfaces. The surface treatments are also applicable and effective in controlling crystal growth mode. Actually quantum dot structures by Droplet Epitaxy were demonstrated on treated semiconductor surfaces. However the surface structures, especially reconstructed structures, are not investigated sufficiently and the chemical bounds of the structures are not understood in detail.

Transition from GaAs(001)(2x6)-S to (2x3)-S surfaces was successfully observed by synchrotron radiation photoelectron spectroscopy (SRPES), X-ray absorption near edge structure (XANES), and X-ray standing waves (XSW). With increasing substrate temperature, a (2x6) structure turns into a (2x3) structure at around 520°C releasing about 20% of the surface sulfur atoms, observed by SRPES, which consisted of the central dimer pairs of the (2x6) structure. The E polarization dependence in the S K-edge XANES spectra suggests that interdiffusion of S atoms should not occur, and that S atoms are still in the top layer bridge site even after high temperature annealing. Moreover, by XSW, it was found that, after the transition from the (2x6) to the (2x3), Ga-S-Ga bridge-bond formation was almost same as that of the (2x6). Therefore, by the transition, only the central dimer pairs of the (2x6) structure were released without interdiffusion, while retaining the Ga-S-Ga bridge-bond formation.

We have also studied real-time in-situ STM observations of Ga adatoms on the MBE growth front, GaAs (001)(2x4)-As surface, with a system in which STM and MBE are completely combined. It is found that the Ga adatoms are relatively located near not the A- but the B-type step edges. Filled state STM images of Ga adatoms near the B-type stepped terrace were observed at 25 sec, 250 sec and 500 sec after the supply of 0.1 ML Ga at 200 °C. The Ga atoms form a dimer like surface structure with two Ga adatoms about one unit cell far from the B-step edge and on a missing dimer row. Then, with the addition of one of the Ga atoms, this dimer like structure changed into trigonal like surface structure. After 250 sec, this trigonal changed into a tetragonal-like structure with the addition of one more of the Ga atoms. It seems that this position attracts the Ga near the step edges. These calculations show that the lowest migration potential value on a stepped GaAs(001)(2x4) surface was located at the position of one unit cell far from the B-step edge and atoms. There are theoretical calculations which predict the migration potentials of Ga adatoms on a missing dimer row. The experiment agrees well with these theoretical results.

Some of this research was performed in collaboration with researchers of other

Divisions in NRIM (M. Shimoda of the Materials Physics Division, Y. Imanaka, T. Takamasu and G. Kido of the Physical Properties Division and T. Ohno of the Computational Materials Division), researchers of the NTT Basic Research Laboratories (M. Sugiyama, Y. Watanabe and S. Maeyama), researchers of the University of Tokyo (H. Fujioka and M. Oshima) and researchers of Korea Institute for Standards and Science (C. D. Lee, C. Park, H. J. Lee, K. S. Lee, S. J. Park, C. G. Park, S. K. Noh). Several papers were jointly published with them.

<Research themes>

- (1)Nanospace Lab. Project (Special Coordination Funds for Promoting Science and Technology: April 1994 to March 1999, N.Koguchi)
- (2)Fabrication and Characterization of Semiconductor Quantum Dots (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1998, N.Koguchi)

3.1.3.4.3 Research products, including papers submitted (April 1997 to March 1999)

- (1) S.Tsukamoto, T. Ohno, N. Koguchi: Scanning Tunneling Spectroscopy and First-principles Investigation on GaAs(001)(2x6)-S Surface Formed by Molecular Beam Epitaxy, , J. Crystal Growth, 175/176, 1303(1997)
- (3) S.Tsukamoto, N.Koguchi: Real-time in-situ Scanning Tunneling Microscopy Observation on GaAs(001) Surface during Molecular Beam Epitaxy Growth, Abstracts of 5th Int. Colloquium on Scanning Tunneling Microscopy, Kanazawa,(1997) 27.
- (4) T.Chikyow, N.Koguchi, A.Shikanai: Direct Ga Deposition by Low Eenergy Focused Ion Beam, Surf. Sci. 386, 254(1997)
- (5) T.Chikyow ,N.Koguchi, A.Shikanai: Direct formation of fine structure by low energy focused ion beam, Mat. Res. Soc. Symp. Proc. 448, 211(1997)
- (6) M.Shimoda,S.Tsukamoto,N.Koguchi: Photoelectron and Auger Electron Diffraction Studies of Sulfur-terminated GaAs(001)-(2x6) Surface, Sur. Sci., 395/1, 75(1998)
- (7) M.Sugiyama, S.Maeyama, Y.Watanabe, S.Tsukamoto, N.Koguchi: Ga-S-Ga Bridge Bond Formation in in-situ S-treated GaAs(001) Surface Observed by Synchrotron Radiation Photoemission Spectrtoscopy, Appl. Sur. Sci., 130-132, 436(1998)
- (8) K.Watanabe, N.Koguchi: Fabrication of Buried GaAlAs Microcrystal Structures by

- Droplet Epitaxy, *J Surf. Anal.* **4**, 316(1998)
- (9) T.Chikyow, N.Koguchi: GaAs Microcrystal Growth on Semiconductor Surfaces by Low Energy Focused Ion Beam, *J. Vac. Sci. and Technol.* **B16**, 2538(1998)
 - (10) M.Shimoda, S.Tsukamoto, N.Koguchi: Coverage Analysis of a Sulfur-terminated GaAs(001)-(2x6) surface: the effect of double sulfur-treatment, *Sur. Sci.* **402-404**, 669, (1998)
 - (11) S.Tsukamoto, M.Sugiyama, M.Shimoda, Y.Watanabe, S.Maeyama, T.Ohno, N.Koguchi: X-ray standing wave study of GaAs(001)(2x6)-S Surface, *Photon Factory Activity Report.* **15**, 243, (1998)
 - (12) M.Shimoda, Y.Watanabe, S.Tsukamoto, M.Sugiyama, S.Maeyama, T.Ohno, N.Koguchi: Synchrotron radiation photoelectron spectroscopy study of surfur-terminated GaAs(001)(2x6) Surface, *Photon Factory Activity Report.* **15**, 244, (1998)
 - (13) M.Sugiyama, S.Tsukamoto, M.Shimoda, S.Maeyama, Y.Watanabe, T.Ohno, N.Koguchi: S-treated GaAs(001) surface studied by X-ray absorption near edge structure, *Photon Factory Activity Report.* **15**, 245, (1998)
 - (14) C.D.Lee, C.Park, H.J.Lee, K.S.Lee, S.J.Park, C.G.Park, S.K.Noh, N.Koguchi: Fabrication of Self-assembled GaAs/AlGaAs Quantum Dots by Low Temperature Droplet Epitaxy, *Jpn. J. Appl.Phys.* **37**, 7158(1998)
 - (15) S.Tsukamoto, N.Koguchi: Realtime in-situ Scanning Tunneling Microscopy Observation of Ga Adatoms near Step Edges on GaAs (001)(2x4)-As Surface, *Proc. 17th Electronic Materials Symposium*, K2, pp.209. Izu Nagaoka (1998)
 - (16) S.Tsukamoto, N.Koguchi: Self-Organizing Process of Ga Adatoms on GaAs (001)(2x4)-As Surface, *Proc. 4th Inter. Conf. on Intelligent Materials*, pp.316-317, Tokyo, (1998)
 - (17) S. Tsukamoto, N. Koguchi: Dynamics of Ga adatoms near step edges on GaAs (001) surface, *Proc. 6th Inter. Colloquium on Scanning Tunneling Microscopy*, pp16, Kanazawa, (1998)
 - (18) S. Tsukamoto, N. Koguchi: Atomic-level in-situ real-space observation of Ga adatoms on GaAs(001)(2x4)-As surface during Molecular beam Epitaxy growth, *J. Crystal Growth.* **201/202**, 118, (1999)
 - (19) S.Tsukamoto, M.Shimoda, M.Sugiyama, Y.Watanabe, S.Maeyama, T.Ohno, N.Koguchi: Transition from GaAs(001)(2x6)-S to (2x3)-S Surfaces Observed by Synchrotron Radiation Photoelectron Spectroscopy, X-ray Absorption near Edge Structure, and X-ray Standing Waves, *Institute of Physics Conference Series*, **162**, 603,(1999)

- (20) T. Mano, K. Watanabe, S. Tsukamoto, H. Fujioka, M. Oshima, N. Koguchi: New Self-organized Growth Method for InGaAs Quantum Dots on GaAs(001) using Droplet epitaxy, Jpn. J. Appl. Phys. in press
- (21) T. Mano, K. Watanabe, H. Fujioka, M. Oshima, N. Koguchi: Fabrication of InGaAs Quantum Dots on GaAs (001) by Droplet Epitaxy, J. Crystal Growth, in press
- (22) T. Mano, K. Watanabe, S. Tsukamoto, Y. Imanaka, T. Takamasu, H. Fujioka, G. Kido, M. Oshima, N. Koguchi: Magneto-photoluminescence Study of InGaAs Quantum Dots Fabricated by Droplet Epitaxy, Physica E, in press.
- (23) M. Shimoda, S. Tsukamoto, T. Ohno, N. Koguchi, M. Sugiyama, S. Maeyama, Y. Watanabe: Stoichiometry Study of S-terminated GaAs(001)-(2x6) Surface with Synchrotron Radiation Photoelectron Spectroscopy, submitted to Phys. Rev. Lett.

Oral Presentation: 42

- (1) International Conference: 11
- (2) Domestic Conference: 31

Patent Application:

- (1) Crystal Growth Observing Apparatus: S. Tsukamoto and N. Koguchi, 1997.12.10, 9-340413 (Japan), 1998.12.9, 09/206,976 (U.S.A.), 1998.12.9, 98310092.6 (EC)

Award:

- (1) Fabrication of InGaAs QDs by Droplet Epitaxy, Award for the Excellent Student, 18th Annual Meeting of Surface Science Soc. in Japan, 1998 Dec. T. Mano,
- (2) Fabrication of InGaAs QDs by Droplet Epitaxy with Highly-dense Ga Droplets, JSAP Award for the Most Promising Young Scientist, 46th Annual Meeting of Jpn. Appl. Phys. Soc. 1999 March, T. Mano

5.3.5 Surface and Interface Analysis Unit

Nobuyuki Koguchi (Supervising Researcher), Michiko Yoshitake, Yarama-Reddy Aparna (STA fellow), Andrei Rar (COE fellow)

3.1.3.5.1 Research work

We found that when deposited metallic film on substrate is heated in a vacuum, the substrate element diffuses onto the surface of the film and makes a

segregation layer. The segregation layer has the thickness of one atom and is quite stable in structure and composition. Even after the removal of the surface layer, this atomic order structure is formed again easily by heating. We call the behavior the self-controlling property of surface composition. Surface with segregation has special characteristics due to the co-existence of segregant and film atoms. Some of such characteristics are useful for vacuum technology application. We have been studying Ti segregation on Nb film for getter material and Cu segregation on Ti film for electron field emitter. This self-controlling property is advantageous to vacuum application, in respect of fabrication and maintenance. We have established the general theory on the kinetics of the composition recovery rate under ion sputtering, segregation dependence on the elemental combination between a substrate and a film.

The relation between surface composition and work function has been studied in the case of Cu segregation on Ti film and Ti segregation on Cu film. It was found that the work function decreases by approximately 0.3 eV with the segregation of Cu on Ti film and increases about 0.2 eV when Ti segregates on Cu film. The chemical shift of Cu 2p_{3/2} suggests that the electron of Cu is partially transferred to Ti in both cases, Cu segregation on Ti film and Ti segregation on Cu film. It suggests that an electric dipole with a positive charge towards a vacuum exists on Cu-segregated Ti film and that with a negative charge towards a vacuum on Ti-segregated Cu film. This picture well coincides with the experimental work function changed by segregation. By heating the specimen after the surface layer was removed by ion sputtering, segregation of Cu occurred again and the same saturated concentration was obtained, as well as the same amount of work function decrease. As the work function varies with the amount of adsorbed (segregated) atoms, the advantage of using the self-controlling property to obtain a stable work function for field emitter fabrication is proved.

<Research theme>

- (1) Self-control of Surface Composition of Thin Ffilm and its Application to Field Emitter

(Research for Intelligent Materials: April 1995 to March 2000, Michiko Yoshitake)

3.1.3.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) M. Yoshitake, K. Yoshihara: Round Robin on Spectrometer Transmission Calibration for AES in the COMMON DATA PROCESSING SYSTEM: Surf.Interface Anal., 25, 209(1997)

- (2) M. Yoshitake, K.Yoshihara: Compositional and Chemical State Analysis of Layered Intermetallic Compounds by Line Scanning of an Etched Crater with Scanning XPS, *J.Surf.Anal.*, **3**, 478(1997)
- (3) M. Yoshitake, K.Yoshihara: Study on Smart Getter Film, *J.Vac.Soc.Jpn*, **40**, 141(1997). (in Japanese)
- (4) M. Yoshitake, K.Yoshihara: Self-controlled Composition of Ti-Nb Film with Getter Function, Text for Electronic Material Soc., FEM-97-7(1997)
- (5) M. Yoshitake, K.Yoshihara: Retrieval System of AES/XPS Spectra on World Wide Web and the Description of Specimen Information for the System, ECASIA'97 Proc. p.1117, (1997)
- (6) M. Yoshitake, K.Yoshihara: Measurement of Work Function Change with Surface Segregation of Substrate Element on a Deposited Film, *J.Vac.Soc.Jpn*, **41**, 230(1998) (in Japanese)
- (7) M. Yoshitake, K.Yoshihara: The Surface Segregation of Ti-Nb Ccomposite Film and its Application to a Smart Getter Material, *Vacuum*, **51**, 369(1998)
- (8) M. Yoshitake, K.Yoshihara: Effect of Temperature on Recovery of Surface Segregation of Ti on Nb film, *J.Japan Inst.Metals*, **63**, 252(1999) (in Japanese)
- (9) M. Yoshitake, K.Yoshihara: The Effect of X-ray Scanning on Intensity and Binding Energy of Photoelectron Peak and the Line Scanning Analysis of an Etched Crater, *J.Surf.Anal.*, **5**, 258(1999)
- (10) M. Yoshitake, K.Yoshihara: Relation between Change of Binding Energy and that of Work Function, *J.Vac.Soc.Jpn*, **42**, 290(1999) (in Japanese)
- (11) M. Yoshitake, K.Yoshihara: Work Function Control by Surface Segregation Phenomenon on Deposited Films, *Appl.Surf.Sci.* **146**, 97 (1999)
- (12) M. Yoshitake, K.Yoshihara: Interface modification of coated materials for substrate material recycle, Proc. 11th Inter. Conf. of Women Engineers and Scientists, in press

Oral Presentation: 18

- (1) International Conference: 9
- (2) Domestic Conference: 9

3.1.3.5 Researchers in the Extreme High Vacuum Station

Age and research fields of researchers in the Extreme High Vacuum Station (March 31, 1991)

Age	Number	Metal-lurgy	Physics	Chemistry	Electronics	Mechanical Engineering
60 ~	0	0	0	0	0	0
50 ~ 60	1	0	0	0	1	0
0 ~ 49	4	1	1	0	1	1
30 ~ 39	18	3	11	3	1	0
22 ~ 29	3	0	1	2	0	0
Summation	26	4	13	5	3	1

3.1.3.6 Research Budgets in the Extreme High Vacuum Station

Research budgets in the Extreme High Vacuum Station (yen)

Kinds of budgets	1997	1998
• Special Research	12,723,000	29,750,000
• COE Project	250,044,000	222,931,000
• Special Coordination Funds for Promoting Science and Technology	64,690,000	49,136,000
• Research for Intelligent Materials	25,202,000	44,625,000
• Organized research Combination System	0	25,421,000
• Supplementary Budgets	0	9,901,000
Summation	352,659,000	381,764,000

Chapter 4 Social-Needs-Oriented Research

4.1 Frontier Research Center for Structural Materials

Akira Sato (Director of Frontier Research Center for Structural Materials)

A large portion of the infrastructure that was built during the era of the great economic growth of Japan will be outdated and need renewal. To maintain a secure society in the forthcoming century, we need to guarantee the safety and prolonged service life of structural materials and infrastructure. Taking global economic issues into consideration, such concepts as low environmental load and conservation of resources have attained increased significance, whereby innovations in steels as the principal components of structures are believed to make a great impact on society.

The NRIM has founded the Frontier Research Center for Structural Materials with the goal to create non-traditional structural materials, in the form of Ultra-Steels, for the 21st century. At the Frontier Research Center, revolutionary concepts rather than modifications of conventional technology will be the main objective of talented staff collected from industries and universities, with the whole operation supported from a specific budget allocation.

The Frontier Research Center for Structural Materials is composed of 3 Research Stations, The Materials Creation Research Station, The Joining and Interface Research Station, and The Strength and Life Evaluation Research Station. The activities of these Stations will be described later.

In the Frontier Research Center for Structural Materials, the research project of Creation of Ultra-Steels for the 21st century is being carried out. The motto of the project is to decrease the environmental load and the total cost of structural materials, that is, steels, by doubling their strength and life. In pursuit of this objective, the following 4 research topics are launched: (1) Development of 800-MPa-class high strength, ferrite-matrix steels with improved weldability, (2) Advanced ultra-high strength steels (1500-MPa-plus class), (3) Advanced ferritic steels for 650 °C ultra-supercritical steam boilers, and (4) Development of steels resistant to marine corrosion. Tentatively, a ten-year research period is allotted, during the first 5 years of which, the focus is on basic studies seeking breakthroughs from traditional technology. In the later 5 years we are going to challenge the scale-up of the processes based on the principles

found in the first 5 years.

The Frontier Research Center for Structural Materials consists of a network structure based on taskforces, which facilitates each research topic. Researchers in 4 taskforces come from the 3 Research Stations mentioned above. This cross-linking is designed for effective and strengthened cooperation and competition among Stations and Taskforces. Such a network creates the Spiral Dynamism. The number of researchers working in the Frontier Research Center is about 110: about 30 from industries and universities, and the rest from the Institute. The Frontier Research Center for Structural Materials is expected to grow as a focal point of human resources by attracting more scientists both from industries and universities. The technical reputation as an R&D center of structural materials will increase by distributing achievements in the form of technical papers and other types of information.

Research products

Table 1 Patent

1997		1998	
Domestic	International	Domestic	International
23	0	40	29

Table 2 Research presentation

1997				1998			
Domestic		International		Domestic		International	
Oral	Written	Oral	Written	Oral	Written	Oral	Written
105	50	18	24	315	138	36	56

Table 3 Research budget for STX-21 (thousand Yen)

Theme Number	Topic	1997	1998	
			Original	Final
09-61-01	800-MPa- class steels	146,000	200,413	304,873
09-61-02	1500-MPa-class high strength steels	148,000	124,913	265,942
09-61-03	Heat-resistant steels	141,000	286,913	425,528
09-61-04	Corrosion-resistant steels	137,405	169,913	233,243
09-61-20	Common equipment	170,000	100,500	96,600
09-61-30	Adjusting appropriation	47,371	33,231	20,232
09-61-40	Common expenses	---	6,960	9,219
Total		789,776	922,843	1,355,637

4.1.1 Materials Creation Research Station

Akira Fukuzawa(Supervising Researcher)

4.1.1.1 Research work

Following the 5th Long-term Plan, the R&D aiming to create new structural materials, in other words, ultra steels, has been promoted at the Materials Creation Research Station. The efforts for creating new steels to sustain the coming new century and to continue the Iron Age in the following new century have been endeavored and are being carried out now. Examples of the outcomes are shown next.

- (1) Ultrafine grain steels have been created by advanced thermomechanical treatment, and their mechanical properties have almost reached the levels proposed in the project, that is, double the strength.
- (2) To produce high tensile strength steels with a high resistance to delayed fracture, a modified ausforming method based on a new concept has been established.
- (3) Leading principles to develop heat resistant steels for the ultra supercritical boiler of power plants have been proposed and new steels are becoming real in terms of creep rupture strength or oxidation resistance.
- (4) Stainless steel with ultra low P levels to obtain a high anti-stress corrosion properties has been obtained by the use of a cold crucible levitation melting method with the application of reduction refining.
- (5) Powder metallurgy with a new process flow concept has produced a plain oxygen steel with unexpectedly high strength, or a heat resistant steel with finely dispersed oxide.
- (6) New type Mn alloys for damping and recyclable aluminum casting alloys without grain refining elements have been developed.

The members of Materials Creation Research Station, as at March 31, 1999, consisted of 21 researchers, 5 STX-21 research fellows, 3 STA fellows, 2 domestic research fellows, 2 technicians, 2 special technical staffs, 5 graduate students, 2 under graduate students, and 11 guest researchers.

The output of the research activities has been increased since the start of the 5th Long-term Plan. The number of publications, including submitted papers, oral presentations and patents submitted is 71, 169 and 46, respectively, and the number of awards won is 8, between April 1, 1997 to March 31, 1999. The details are shown in the research activities of each research unit.

The supporting work in melting, forging, rolling and heat treatment for the requirements from researchers in the Institute is engaged in the 5th Laboratory. For the support of metal working, facilities such as vacuum induction melters, 300t press, rolls and heating furnaces are kept in good condition and operated with highly experienced skills, to produce samples to meet researchers demands, by 3 researchers, 2 technicians, and 2 special technical staff. A serious problem is the decrease in number of support members due to retirement.

4.1.1.2 1st Unit

Kazuyuki Sakuraya, Toshiaki Watanabe, Satoshi Iwasaki and Yoshinao Kobayashi

4.1.1.2.1 Research work

- (1) The development and improvement of a cold crucible type levitation melting method of a capacity more than 1kg-steel have been carried out successfully. It made possible to melt refractory metals or active metals without pollution.
- (2) Reduction of P content in stainless steel for marine use to as low as 2 ppm has been obtained by reduction refining methods in the cold crucible.
- (3) Study on deoxidization of Ti by the use of rare earth elements has proved the possibility to decrease the oxygen content of Ti, which contains over 1000ppm at the outset.

<Research themes>

- (1) Development of Structural Steels Durable to Marine Environments (Research on Structural Materials for 21st Century: April 1997 to March 2002, Toshiaki Kodama)
- (2) Melting of Refractory Metals by Cold Crucible Levitation Melting (General Research: April 1997 to March 1999, Akira Fukuzawa)
- (3) Properties of inclusion removal of molten metal by the cold crucible levitation method (Bilateral International Joint Research by Special Coordination Funds for Promoting Science and Technology: 1998 Fiscal Year, Akira Fukuzawa)

4.1.1.2.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) A.Fukuzawa, K.Skuraya, T.Watanabe, S.Iwasaki: Melting of Titanium with Cold

Crucible, in Progress Report on Heisei 9th, ed. by JST(Kawaguchi, Japan 1998) (in Japanese)

- (2) A.Fukuzawa, K.Skuraya, T.Watanabe, S.Iwasaki: Removal of Phosphorous in Stainless Steel by Cold Crucible, in Progress Report on Heisei 9th, ed. by JST(Kawaguchi, Japan 1998) (in Japanese)
- (3) M.Nagano, A.Akutsu, T.Takahashi, T.Watanabe, S.Iwasaki: Remelting of Nodular Cast Iron by Electromagnetic Levitation Melting System, Symposium on New Magnetic Science'97, Urawa Nov. 27-28,1997 (in Japanese)
- (4) A.Fukuzawa, K.Skuraya, T.Watanabe, S.Iwasaki, Y.Kobayashi : Dephosphorization of Molten Stainless Steel by Using a Cold Crucible, in *New Advancement in Electromagnetic Processing of Materials*, ed. by Electromagnetic Noble Processing Research Group in ISIJ(Tokyo, Japan 1999) (in Japanese)

• Oral Presentation: 4

- (1) International Conference: 0
- (2) Domestic Conference: 4

• Patent Application :

- (1) Refining of Molten Metals, A.Fukuzawa, K.Sakuraya, T.Watanabe, S.Iwasaki, 030398, 10-51112
- (2) Removal of P in Stainless Steel, A.Fukuzawa, K.Sakuraya, T.Watanabe, S.Iwasaki, 030398, 10-51113
- (3) Production Method of Anti-corrosion Stainless Steel, K.Sakuraya, T.Watanabe, S.Iwasaki, Y.Kobayashi, A. Fukuzawa, H.Uno, Y.Katada, 260299, 11-051031

• Award :

- (1) STA Award for Achievement, A. Fukuzawa, 1998.5.19

4.1.1.3 2nd Unit

Masaaki Igarashi (Unit Leader), Seiichi Muneki, Kazuhiro Kimura, Josef Nemec (STA fellow), Kenta Suzuki (joint-doctoral course student), Satoshi Itoh (undergraduate student)

4.1.1.3.1 Research work

- (1) Several fundamental guiding principles to improve creep rupture strength and steam oxidation resistance of ferritic steels at elevated temperatures have been established for the development of new materials to be used for 650°C USC boilers. Improvement of microstructural stability is found to be a prime candidate and useful approach for life extension of ferritic creep resistant steel based on the established inherent creep strength.
- (2) An automatically controlled constant stress creep test machine has been developed. A new guideline has been proposed to evaluate inherent creep strength from creep deformation under a constant stress creep test.
- (3) A new sophisticated evaluation method has been established to characterize the transformation and precipitation behavior of ferritic steels using a combination of differential scanning calorimetry and vibrating sample magnetic measurement. This allows one to measure A_{c1} , A_{c3} , M_s , M_f and A_2 of the ferritic steels and the nose temperatures of various precipitations in the steels with great accuracy and ease.

<Research themes>

- (1) Research and Development of Advanced Ferritic Steels for 650°C USC Boilers from the Standpoint of Long-term Phase Stability (Research on Structural Materials for 21st Century: April 1997 to March 2002, Fujio Abe)
- (2) Fundamental Research on Evaluation Method of Inherent Creep Strength (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1998, Kazuhiro Kimura)
- (3) A New Approach to Characterize Recovery and Softening Processes of Martensite (Special Coordination Funds for Promoting Science and Technology: April 1998 to March 1999, Masaaki Igarashi)

4.1.1.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) K.Kimura, H.Kushima, F.Abe, K.Yagi: Inherent Creep Strength and Long Term Creep Strength Properties of Ferritic Steels, Materials Science and Engineering, A234-236, 1079 (1997).
- (2) M. Igarashi, Y. Sawaragi: Development of 0.1C-11Cr-3W-3Co-V-Nb-Ta-Nd-N Ferritic Steel for USC Boilers, Inter. Conf. on Power Eng. '97, Vol.2, 107 (1997).

- (3) K.Kimura, H.Kushima, K.Yagi: Life Extension of Ferritic Creep Resistant Steels by a Stabilizing of Microstructure, Proc. 3rd Inter. Conf. ECOMATERIALS, 119 (1997).
- (4) K.Kimura, H.Kushima, F.Abe, K.Yagi, H. Irie: Assessment of Creep Strength Properties of 9 to 12% Cr Steels from a Viewpoint of Inherent Creep Strength, Proc. 4th Inter. Charles Parsons Turbine Conf., 257 (1997).
- (5) S. Muneki, F. Abe, H. Irie: Relationship between Hardness and Fracture Toughness of a 9.7mol% MgO Partially Stabilized Zirconia Alloy and High Specific Strength Metallic Materials, 5th Japan Inter. SAMPE Symposium, 547 (1997).
- (6) K.Kimura, H.Kushima, F.Abe, K.Yagi: Evaluation of the Creep Strength Property from a viewpoint of Inherent Creep Strength for Ferritic Creep Resistant Steels, *Microstructural Stability of Creep Resistant Alloys for High Temperature Plant Applications*, Edited by A.Strang, J.Cawley and G.W.Greenwood, Microstructure of High Temperature Materials, Number 2, The Institute of Materials, p.185 , (1998).
- (7) M. Igarashi, S. Muneki, F. Abe: Microstructure Control of Martensitic Phase Matrix in Advanced Ferritic Steels for USC Boilers to Achieve Long Term stability, 6th Liege Conf. on Materials for Advanced Power Engineering, I.637 (1998).
- (8) S. Muneki, M. Igarashi, F. Abe: Microstructure Control of Martensitic Phase in Advanced Ferritic Steels for USC Boilers to Achieve Long Term Stability at High Temperatures, 11th Congress of Inter. Fed. For Heat Treatment and Surface Engineering, 265 (1998).
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- (10) K. Kimura, F. Abe, H. Irie, K. Yagi: Long-Term Creep and Creep Rupture Properties and Microstructural Changes of Heat Resistant Steels, Materials for Advanced Power Engineering 1998, Vol.5, Part 1, 627 (1998).
- (11) M. Igarashi, S. Muneki: Homogeneous precipitation of L10 type ordered FePd in advanced heat-resistant martensitic steels, Proc. of Inter. Conf. On Solid-Solid Phase Transformations '99, May 24-28, Kyoto, 1999
- (12) M. Igarashi, S. Muneki, F. Abe: Homogeneous precipitation of L10 type ordered FePd in advanced heat-resistant martensitic steels, Proc. of Inter. Conf. On Case Histories on Integrity and Failures in Industry, Sept. 28-Oct.1, Milan, 1999, submitted
- (13) S. Muneki, M. Igarashi, F. Abe: Effect Of Thermomechanical Heat Treatment On Creepcharacteristics Of High Cr Heat Resistant Ferritic Steels, Proc. of Inter. Conf. On Case Histories on Integrity and Failures in Industry, Sept. 28-Oct.1, Milan,

1999, submitted

• **Oral Presentation: 38**

(1) International Conference: 12

(2) Domestic Conference: 26

Patent Application :

- (1) Heat Resistant Ferritic Steels: M. Igarashi, F. Abe, S. Muneki, K. Kimura, H. Kushima, N. Fujitsuna, 1997.9.24, Hei-09-256479, Japan, U.S., Germany, Denmark.
- (2) Heat Resistant Ferritic Steels: M. Igarashi, F. Abe, S. Muneki, K. Kimura, H. Kushima, N. Fujitsuna, 1997.9.24, Hei-09-256480, Japan, U.S., Germany, Denmark.
- (3) Heat Resistant Ferritic Steel with Lath-martensitic Microstructure and Method for Producing it: M. Igarashi, F. Abe, S. Muneki, 1997.9.24, Hei-09-256482, Japan, U.S., Germany, Denmark.
- (4) Precipitation Hardened Heat-resistant Ferritic: M. Igarashi, F. Abe, S. Muneki, 1999.8.31, Hei-10-260956, Japan, U.S., Germany.
- (5) Precipitation Hardened Heat-resistant Ferritic: M. Igarashi, S. Muneki, F. Abe, 1999.02.26, Hei-11-052005, Japan, (U.S., Germany, Denmark to be expected).
- (6) Precipitation Hardened Martensitic Heat-resistant Ferrous Alloys: M. Igarashi, S. Muneki, F. Abe, 1999.02.26, Hei-11-051844, Japan, (U.S., Germany, Denmark to be expected).

4.1.1.4 3rd Unit

Kotobu Nagai (Unit Leader), Shiro Torizuka, Osamu Umezawa, Tadanobu Inoue, Tetsuya Ohashi (STX-21 research fellow), Tohru Hayashi (STX-21 research fellow), Hiroshi Nakajima (STX-21 research fellow), Toshihiro Hanamura (domestic research fellow), Ilaria Salvatori (STA fellow), Naohisa Yokoyama (graduate student), Noriyuki Tsuchida (graduate student), Satoshi Genda (undergraduate student), Hideaki Moriya (guest researcher), Yo Tomota (guest researcher), Yoshiyuki Saito (guest researcher), Shiro Yoshimatsu (guest researcher)

4.1.1.4.1 Research Work

- (1) Ultrafine grain steels have been created through heavy deformation and heat treatments, and their mechanical deformation behaviors also studied with an aid of computer simulation.
- (2) Recyclable design of base metals was attempted from the perspectives of environmental load reduction.

<Research themes>

- (1) Study on Strengthening of Ferrite Matrix Steels for Welded Structures (Research on Structural Materials for 21st Century: April 1997 to March 2002, Kotobu Nagai)
- (2) Study on Design and Evaluation of ECOMATERIALS -Fine Microstructure Development in Plural Phases Alloy- (Special Coordination Funds for Promoting Science and Technology: April 1996 to March 1998, Kotobu Nagai)

4.1.1.4.2 Research products, including papers submitted(April 1, 1997 to March 31, 1999)

- (1) S.Torizuka, O.Umezawa, K.Tsuzaki, K.Nagai: Refinement of Ferrite-Pearlite Structures through Transformation from Heavily Deformed Austenite in a Low Carbon Si-Mn Steel, Materials Science Forum, 284-286, 225 (1998)
- (2) S.Torizuka, O.Umezawa, K.Tsuzaki, K.Nagai, S.Genda, Y.Kogo: Effect of Strain rate and Deformation Temperature on Fine Ferrite Grain Structure Formed from Heavily Deformed Austenite, Inter. Conf. on Solid-Solid Transformation '99, in press
- (3) O.Umezawa, K.Nagai: Microstructural Design and Processing of Plasticized Hyper-eutectic Al-Si-Fe Alloys, Proc. 3rd Inter. Conf. on ECOMATERIALS, 59, (1997)
- (4) O.Umezawa, K.Nagai: Microstructural Design of Hyper-eutectic Al-Si Alloy and Its Deformation Behavior, Proc. 3rd Inter. Conf. on ECOMATERIALS, 91, (1997)
- (5) C.-Y.Lim, O.Umezawa, K.Nagai: Microstructural Control of Recyclable Al-Si-Fe Alloys by Low Temperature Working Process, Proc. 3rd Inter. Conf. on ECOMATERIALS, 99, (1997)
- (6) O.Umezawa, K.Nagai: Subsurface Crack Generation in High-cycle Fatigue for High Strength Alloys, Iron Steel Inst. JPN. Inter., 37, 1170, (1997)
- (7) H.Yokoyama, O.Umezawa, K.Nagai, T. Suzuki: Distribution of Internal Crack Initiation Sites in High-cycle Fatigue for Titanium Alloys, Iron Steel Inst. JPN. Inter., 37, 1237, (1997)
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- Subsurface Crack Initiation of Ti-6Al-4V alloys, *High Cycle Fatigue of Structural Materials*, TMS, 287, (1997)
- (9) O.Umezawa, K.Nagai: Deformation Structure and Subsurface Fatigue Crack Generation in Austenitic Steels at Low Temperature, *Metall. Mater. Trans. A*, **29A**, 809, (1998)
 - (10) C.-Y.Lim, O.Umezawa, K.Nagai: Influence of Low Temperature Working on Microstructure and Mechanical Properties of Al-7Si-(Fe) Alloys, *Metals and Materials*, **4**, 1027, (1998)
 - (11) O.Umezawa, K.Nagai: Effects of Test Temperature on Internal Fatigue Crack Generation Associated with Non-metallic Particles in Austenitic Steels, *Metall. Mater. Trans. A*, **29A**, 3017, (1998)
 - (12) O.Umezawa, H.Yokoyama, K.Nagai, T.Suzuki, K.Kokubo: Electron Microscopy Study of Cyclic Deformation in Ti-Fe-O alloy at Low Temperature, *FATIGUE'99*, vol.1, High Edu. Press, 211, (1999)
 - (13) O.Umezawa, K.Nagai: Microstructural Refinement of As-cast Al-12.6wt%Si Alloy by Repeated Thermomechanical Treatment to Produce a Heavily Deformable Material, *Metall. Mater. Trans. A*, **30A**, (1999), in press
 - (14) H.Yokoyama, O.Umezawa, K.Nagai, T.Suzuki, K.Kokubo: Internal Crack Initiation in High-cycle Fatigue of Ti-Fe-O Alloy at Liquid Nitrogen Temperature, *Titanium'99*, in press
 - (15) T.Inoue, Y.Mutoh, M.Hojo, S.Ochiai: Conditions of Compressive Residual Stress at the Interface Edge of Dissimilar Materials, *J. Welding Society*, **17**, 120(1999)
 - (16) T.Inoue, S.Kubo: Thermal Stress Field at the Interface Edge in Dissimilar Materials, *J. Society Materials Science*, **48**, 365(1999)
 - (17) T.Inoue, M.Hojo, S.Ochiai: Disappearance Conditions of Thermal Stress Singularities Based on Stress Intensity in Two and Three-Phase Bonded Structures, *Inter. J. Fracture*, in press.
 - (18) T.Obashi, K.Hidaka, S.Imano: Elastic Stress in Single Crystal Ni-base Superalloys and the Driving Force For Their Microstructural Evolution under High Temperature Creep Conditions, *Acta Mater.* **45**, 1801(1997)
 - (19) T.Obashi, K.Miyake, K.Obashi: Molecular Dynamics Simulations of Low Energy Atomic Collisions Between an Atom and a Substrate: Effect of incident Angle and Energy, *Nuclear instruments and Methods in Physics Research B*, **121**, 40(1997).
 - (20) T.Obashi: Finite Element Analysis of the Plastic Slip and Evolution of the Geometrically Necessary Dislocations in F.C.C. Crystals, *Phil. Mag. Lett.*, **75**, 51(1997).

- (21) T.Ohashi, K.Hidaka, M.Saito: Quantitative Study of The Plastic Slip Deformation and Formation of Internal Stresses in Ni-base Superalloys, *Mat. Sci. Engi. A*, **238**, 42(1997)
- (22) T.Hayashi, M.Saito, K.Tsuzaki, K.Nagai: "Formation of Equiaxed Fine Ferrite Grain Structures through Warm Forging of Low Carbon Martensite", The 4th Inter. Conf. on Recrystallization and Related Phenomena, **13**, 333,(1999)
- (23) H.Moriya, K.Nagai, Y.Kawabe, A.Okada, Strain Rate Dependence of Stress-Strain Curves in a Ti-Fe-O Alloy, *ISIJ Inter.* **37**, 1016,(1997)
- (24) H.Moriya, K.Nagai, Y.Kawabe and A.Okada, Room-Temperature Stress Relaxation in a Ti-Fe-O Alloy, *Tetsu to Hagane* **83**, 599,(1997) (in Japanese)
- (25) H.Moriya, K.Nagai, O.Umezawa, Fracture Characteristics and Deformation Behavior of Solution-treated Ti-15V-3Cr-3Sn-3Al Alloy, *Int. J. Materials & Product Technology*, in press
- (26) K.Nagai, Y.Sakai, O.Umezawa, Trade-off-Balancing through Geometrical Control of Microstructure in Plural Phase Alloys, *Proc. of 28th FRP Symposium, JSMS*, (1999) (in Japanese)
- (27) K.Nagai, S.Matsuoka, R&D on High Strength Steels in 'Ultra-Steels Project', *J. Soc. Mater. Science. JPN*, **48**,(1999) in press. (in Japanese)

• **Oral Presentation: 83**

- (1) International Conference: 8
- (2) Domestic Conference: 75

• **Patent Application**

- (1) Ultra-fine Structure Steel and Method for Producing It, S.Torizuka, K.Tsuzaki, K.Nagai, 1997.9.22, Japan 09-256682
- (2) Ultra-fine Ferrite Grain Structure Steel, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.3.4, Japan 10-052545
- (3) Method for Producing Ultra-fine Structure Steel, S.Torizuka, O.Umezawa, K.Tsuzaki, K.Nagai, 1998.2.26, Japan 11-051799
- (4) Processing of Multi-phases Al-Si-Fe Alloys, O.Umezawa, K.Nagai, Japan, 1998. 2.27, Japan 10-048382
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- (6) Oxide Dispersed Steel and Its Production Method, H.Nakajima, S.Torizuka, K.Tsuzaki, K.Nagai, 1998.03.04, Japan 10-052556

- (7) High Strength P-added Low Carbon Steel with Fine Grain Structure, T.Hanamura, H.Nakajima, S.Torizuka, K.Tsuzaki, K.Nagai, 1998.08.31, Japan H10-260957
- (8) Oxide Dispersed Steel and Production Method, H.Nakajima, S.Torizuka, K.Tsuzaki, K.Nagai, 1998.09.02, Japan 10-248483
- (9) Thick Steel Product with High Weldability and Production Method, H.Nakajima, S.Torizuka, K.Tsuzaki, K.Nagai, 1999.02.25, Japan 11-048962
- (10) Ultra Fine Ferrite Steel, T.Hayashi, S.Torizuka, K.Tsuzaki, K.Nagai, 1998.09.22, Japan 09-256483
- (11) Ultra Fine Ferrite Steel and Its Production Method, T.Hayashi, O.Umezawa, S.Torizuka, K.Tsuzaki, K.Nagai, 1999.02.26, Japan 11-052004
- (12) Production Method of Thick Ultra Fine Ferrite Steel, T.Hayashi, M.Saito, S.Torizuka, K.Tsuzaki, K.Nagai, 1999.02.26, Japan 11-052006
- (13) High Strength and Toughness Steel Bar and Its Production Method, T.Hayashi, S.Torizuka, K.Tsuzaki, K.Nagai, 1999.02.26, Japan 11-052008
- (14) Production Process of High Strength P-added Low Carbon Steel with Fine Grain Structure, T.Hanamura, H.Nakajima, S.Torizuka, K.Tsuzaki, K.Nagai, 1999.02.26, Japan H11-052008
- (15) Ultra-fine Texture Steel and Method for Producing It, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.9.21, U.S.A 09/157,394
- (16) Ultra-fine Texture Steel and Method for Producing It, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.9.21, China 98120620.4
- (17) Ultra-fine Texture Steel and Method for Producing It, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.9.21, Korea 1998-38944
- (18) Ultra-fine Texture Steel and Method for Producing It, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.9.21, Taiwan 87115693
- (19) Ultra-fine Texture Steel and Method for Producing It, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.9.21, EPC U.K. 98307632.4
- (20) Ultra-fine Texture Steel and Method for Producing It, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.9.21, EPC France 98307632.4
- (21) Ultra-fine Texture Steel and Method for Producing It, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.9.21, Germany 98307632.4
- (22) Ultra-fine Texture Steel and Method for Producing It, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.9.21, Sweden 98307632.4
- (23) Fine Ferrite-based Structure Steel and Production Method Thereof, T.Hayashi, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.09.21, America 09/157.393
- (24) Fine Ferrite-based Structure Steel and Production Method Thereof, T.Hayashi,

- S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.09.21, Korea 98307638.1
- (25) Fine Ferrite-based Structure Steel and Production Method Thereof, T.Hayashi, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.09.21, Germany 98307638.1
- (26) Fine Ferrite-based Structure Steel and Production Method Thereof, T.Hayashi, S.Torizuka, K.Tsuzaki, K.Nagai, O.Umezawa, 1998.09.21, France 98307638.1

• **Awards:**

- (1) Best Paper Award in Kogakuin University, H.Yokoyama, 1998.3.20.
- (2) Nishiyama Memorial Award, ISIJ, K.Nagai, 1998.4.1
- (3) JSME Young Engineers Awards, JSME, T. Inoue, 1998.4.3.
- (4) Encourage Award of Presentation, The Japan Society for Heat Treatment, T.Hayashi, 1998.5.27
- (5) Best Poster Presentation Award for Student, ISIJ, H.Yokoyama, 1999. 3.30.

4.1.1.5 4th Unit

Kaneaki Tsuzaki (Unit Leader), Yoshikazu Sakai, Yoshiaki Osawa, Goro Arakane, Susumu Takamori, Toru Hara, Satoru Yusa (STX-21 research fellow), Shusaku Takagi (STX-21 research fellow), Yin Fuxing (domestic research fellow), Jiao Yuning (STA fellow), Toshihei Misawa (guest researcher), Masato Enomoto (guest researcher), Masaharu Kato (guest researcher), Setuo Takaki (guest researcher), Kohji Kawahara (guest researcher), Osamu Ohasi (guest researcher)

4.1.1.5.1 Research work

- (1) Ultra-high strength martensitic steels with high resistance to delayed fracture have been created through thermomechanical processing.
- (2) The ultra-fine grained ferrite steels containing fine oxide particles with a tensile strength over 1500MPa have been created by utilizing a mechanical milling process.
- (3) The morphology and crystallography of carbides in tempered martensite have been studied by means of TEM and AFM.
- (4) Estimation of hydrogen embrittlement sensitivity of high strength steels has been studied from the viewpoint of the diffusible hydrogen content for crack initiation.
- (5) Development of new types of damping alloys, to substitute for both damping steel

plates and higher recyclability aluminum casting alloys, without grain refining elements has been carried out.

- (6) Fabrication and characterization of ceramics particles dispersed cast iron by compo-casting process have been carried out.

<Research themes>

- (1) Study on Development of Advanced Ultra-High Strength Martensitic Steels
(Research on Structural Materials for 21st Century: April 1997 to March 2002, Saburo Matsuoka)
- (2) Research and Development of Recyclable Simple-System Alloys
(Pollution Prevention Research: April 1996 to March 2001, Akira Sato)
- (3) Fabrication of Highly Functional Composite Cast Irons
(Special Coordination Funds for Promoting Science and Technology: April 1996 to March 1999, Akira Sato)

4.1.1.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) K.Tsuzaki, N.R.Ikeda, T.Maki: Effect of Ferrite Morphology on the Cold-rolling and Recrystallization Textures in an Ultra-low Carbon Steel, *Scripta Mater.* **36**, 905-913, (1997).
- (2) K.Tsuzaki, E.Sato, S.Furimoto, T.Furuhara, T.Maki: Formation of an (α + γ) Microduplex Structure without Thermomechanical Processing in Superplastic Ultrahigh Carbon Steels, *Scripta Mater.* **40**, 675-681, (1999).
- (3) T.Maki, T.Furuhara, K.Tsuzaki: Microstructure Development by Thermomechanical Processing in Duplex Stainless Steel and Beta Titanium Alloy, *Inter. Conf. on Thermomechanical Processing of Steel & Other Materials (Thermec'97)*, 78-86, 1997.
- (4) E.Sato, S.Furimoto, T.Furuhara, K.Tsuzaki, T.Maki: Microstructure Control for Superplasticity of an Ultra-High Carbon Steel, *Mater. Sci. Forum*, **304-306**, 133-138, (1999).
- (5) K.Tsuzaki: Metallurgy of Grain Refinement through Ferrite Transformation from Work-hardened Austenite, *J. Jpn. Soc. for Heat Treatment*, **33**, 146-153, (1998). (in Japanese)
- (6) K.Tsuzaki, M.Mituzono, H.Kobayashi, T.Maki: Formation of {111} Recrystallized Grains at Grain Boundaries in Bicrystals of an Fe-19%Cr Ferritic Alloy, *4th Inter. Conf. on Recrystallization and Related Phenomena*, Tsukuba, July 13-16, 347-354,

1999.

- (7) S.Yusa, T.Hara, K.Tsuzaki, T.Takahashi.: Refinement of grain boundary cementite in medium-carbon tempered martensite by thermomechanical processing, *Mat. Sci. Eng.*, in press
- (8) T.Hara, T.Ohba, K.Otsuka: Twin Interface Structures in Martensitically Transformed γ 1'Cu-Al-Ni and γ 2'Au-Cd Alloys, *Proc. 3rd Pacific Rim Inter. Conf. on Advanced Materials and Processing (PRICM3)*, 1119-1204, 1998.
- (9) T.Hara, S.Yusa, K.Tsuzaki: Microstructural Characteristics of Cementite in Tempered Martensite Derived from Strain-Hardened Austenite, *Proc. Inter. Conf. on Solid-Solid Phase Transformations'99(PTM'99)*, (accepted)
- (10) T.Hara, T.Ohba and K.Otsuka Twin Interface Structures in γ 1'Cu-Al-Ni Martensite, *Materia*, 37, 384, (1998).
- (11) Y.Sakai, H.-J.Schneider-Muntau: Ultra-High Strength, High Conductivity Cu-Ag Alloy Wires, *Acta Mater.* 45, 1017-1023, (1997).
- (12) Y.Sakai, T.Asano, T.Kiyoshi, K.Inoue, H.Wada: Development of the Ultra-High Strength, High Conductivity Cu-Ag Alloy, *Proc. 15th Inter. Conf. on Magnet Technology*, 1222-1225, (1997).
- (13) Y.Sakai, T.Asano, K.Inoue, H.Maeda: Strength and Conductivity of Cu-Ag Microcomposites, High Magnetic Field, *World Scientific*, 477-488, (1997).
- (14) Y.Sakai: Strength of Havily Cold Worked Two-Phase Copper Alloys, *Materia*, 36, 692-696, (1997). (in Japanese)
- (15) Y.Sakai, K.Inoue, K.Tanaka, K.Moriyasu, T.Ohgaki: Development and Application of Cu-Ag Alloys with High Strength and High Conductivity, *Showa Electric Wire & Cable Review*, 48, 144(1998). (in Japanese)
- (16) F. Yin, Y. Ohsawa, A. Sato. Kohji Kawahara: Temperature Dependent Damping Behavior in a Mn-19Cu-6Ni-2Fe alloy Continuously Cooled in Different Rates from the Solid Solution Temperature, *Scripta Mater.* 38, 1341, (1998).
- (17) F. Yin, Y. Ohsawa, A. Sato, K. Kawahara: Solid Solution Treatment Improved Damping Behavior in an As-casted and Cold-rolled Mn-20Cu-5Ni-2Fe Alloy, *Zeitschrift für Metallkunde*, 89, 481(1998).
- (18) F.Yin, Y. Ohsawa, A. Sato, K. Kawahara: Effect of Solid Solution Treating on the Microstructure and Damping Behavior of MnCuNiFe Alloys, *J. Materials Science and Technology*, 14, 299(1998).
- (19) F. Yin, Y. Ohsawa, A. Sato, K. Kawahara: Decomposition of High Temperature Gamma-Mn Phase during Continuous Cooling and Resultant Damping Behavior in Mn_{74.8}Cu_{19.2}Ni_{4.0}Fe_{2.0} and Mn_{72.4}Cu_{20.0}Ni_{5.0}Fe_{2.0} Alloys, *Materials Transactions*,

JIM 39, 841(1998).

- (20) F. Yin, Y. Ohsawa, A. Sato, Kohji Kawahara: X-Ray Diffraction Characterization of the Decomposition Behavior of Gamma-Mn Phase in a Mn-30 at %Cu Alloy, Scripta Mater. 40, 992(1999).
- (21) F. Yin, Y. Ohsawa, Akira Sato, K. Kawahara: Modulated Microstructure Observation in a Slowly Cooled Mn-19.7Cu-7.9Ni-2Fe (at.%) Alloy, Zeit. Metallk., 90(6),(1999).
- (22) F. Yin, Y. Ohsawa, A. Sato, K. Kawahara: Magnetic Measurement Determination of the Decomposition Behavior of the Gamma-Mn Solid Solution in a Mn-19.7Cu-7.9Ni-2Fe (at.%) Alloy, Mater. Trans. JIM, 40, 451(1999).
- (23) K. Kawahara, F. Yin: A New-type Damping Materials, M2052 Alloy, J. Vac. Soc. Jpn., 42, 11(1999). (in Japanese)
- (24) F. Yin, Y. Ohsawa, A. Sato, K. Kawahara: Decomposition Behavior of the Gamma-Mn Phase in Mn-Cu and MnCuNiFe alloys during Aging within the Miscibility Gap, Proc. Solid- Solid Phase Transformation Conf., Kyoto, Japan, ed. by K.Otsuka, JIM, p148,1999.
- (25) Y. Osawa, G. Arakane, S.Takamori, A. Sato: Refining of Graphite Particles in Cast Iron by Applying Ultrasonic Vibration to Their Melts. Processing and Fabrication of Advanced Materials IV, The Institute of Materials 15-22, 1998 (in Japanese)
- (26) Y. Osawa, G. Arakane, S. Takamori, A. Sato, O. Ohashi : Effects of Ultrasonic Vibration on Refining of Crystal Structures of Al-Si Alloys During Solidification, J. Jpn. Found. Soc. 71, 98(1999). (in Japanese)

• **Oral Presentation:** 37

- (1) International Conference: 5
- (2) Domestic Conference: 32

• **Patent Application :**

- (1)Ultra-fine Multi-phase Structure Steel, K.Tsuzaki, S.Torizuka, K.Nagai, 1997.9.22, Japan 09-256802
- (2)High Toughness High Strength Steel and a Method for Manufacturing the Same, S.Yusa, K.Tsuzaki, T.Takahashi, 1998.3.4, Japan 10-52554
- (3)High Toughness Tempered Martensitic Steel and a Method for Manufacturing the Same, S.Yusa, K.Tsuzaki, T.Takahashi, 1998.3.4, Japan 10-52555
- (4)High Toughness Steel and a Method for Manufacturing the Same, S.Yusa,

K.Tsuzaki,T.Takahashi, 1999.3.2, U.S.A. 09/260,056

(5)High Toughness Steel and a Method for Manufacturing the Same, S.Yusa, K.Tsuzaki, T.Takahashi, 1999.3.2, Germany 19909324.5

(6)High Toughness Steel and a Method for Manufacturing the Same, S.Yusa, K.Tsuzaki, T.Takahashi, 1999.3.2, Korea 1999-0006811

(7) High Strength, High Conductivity Copper Alloy Sheets, Y. Sakai, K. Inoue and H. Maeda, 1997. 10. 31, Japan 2714555.

• **Award :**

(1) The Meritorious Honor Award, JIM, K.Tsuzaki, 1999.3.29

4.1.1.6 5th Unit

Tatsuro Mitsui (Unit Leader), Junji Takahashi, Nobuo Sakuma, Masashi Saito (technician), Tetsuo Shimizu (technician) , Takaaki Hibarui (special technical staff), Katsuya Onishi (special technical staff),

4.1.1.6.1 Support work

The 5th Unit has been in charge of the technical support work for researchers in melting, forging, rolling, processing and heat treatment of research materials.

For the melting, melting facilities such as high frequency vacuum induction, plasma, arc etc., have been operated by 2 researchers and 1 special technical staff, and the number of requested alloy ingots in 1997 and 1998 fiscal year were 460 and 532, respectively.

For the forging, rolling and processing, some facilities such as an oil hydraulic forging press, a hot rolling mill, a bar mill, a cold rolling mill and a swaging machine have been operated by 1 technician and 1 special technical staff, and the number of raw materials for test specimens in 1997 and 1998 fiscal year were 672 and 1117, respectively.

For the heat treatment, some facilities such as fluidized bed furnaces, high vacuum heat treatment furnaces, etc., have been operated by 1 technician for many test pieces for researchers.

• **Award**

(1) STA Award for Achievement, J. Takahashi, 1998.5.19

4.1.1.7 6th Unit

Minoru Otaguchi, Shuji Wanikawa, Yuji Muramatsu (guest researcher)

4.1.1.7.1 Research work

- (1) Ultra-steels with high strength and high toughness have been developed by using iron powder and fine oxide powder.
- (2) An invention of oxide-dispersion-strengthened alloys for high temperature use has been done by using a newly developed dispersion method.

<Research themes>

- (1) Study on Strengthening of Ferrite Matrix Steels for Welded Structures (Research on Structural Materials for 21st Century: April 1997 to March 2002, Kotobu Nagai)
- (2) Research and Development of Advanced Ferritic Steels for 650°C USC Boilers from the Standpoint of Long-term Phase Stability (Research on Structural Materials for 21st Century: April 1997 to March 2002, Fujio Abe)

4.1.1.7.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) Thermal Stability of the Ultrafine Grain Structure in a Consolidated of Mechanically Milled Powders.(Edited by T.Sakata. H.G.Suzuki. *Recrystallization and Related Phenomena*. JIM, Proc. Vol.13. July 13-16,1999. M.Otaguchi, K.Tsuzaki K.Nagai.p.495)

• Oral presentation : 7

- (1) International Conference: 0
- (2) Domestic Conference: 7

• Patent Application

- (1) Oxygen-steel(Oxsteel): M.Otaguchi. S.Wanikawa, Y.Muramatsu, K.Tsuzaki, K.Nagai, T.Hayashi. Japan.1999.3.26.No.83893.
- (2) High-strength Metallic Consolidates: M.Otaguchi, S.Wanikawa, Y.Muramatsu, K.Tsuzaki, K.Nagai,T.Hayashi.Japan.1999.3.26.No.83892.
- (3) Fabrication Methods for High-strength Metallic Consolidates and Oxygen-steel: M.Otaguchi, S.Wanikawa, Y.Muramatsu, K.Tsuzaki, K.Nagai, T.Hayashi. USA, EPC, China, Korean. 1999.3.26. PCT.No.PCT/JP99/01566.
- (4) The Manufacturing Method of Oxide Dispersion Strengthening Alloy Powder: Shyji Wanikawa, Minoru Ohtaguchi, Yuji Muramatsu, July 21 1998, No 205710

4.1.1.8 Researchers in the Materials Creation Research Station

Age and research fields of researchers in the Materials Creation Research Station.

(March 31, 1999)

Age	Num- ber	Tech- nicians	Melting & Refin- ing	Solidifi- cation	Working & Heat- treatment	Simu- lation	Metal- lurgy	Powder Metal- lurgy
60~	3			1	1			1
50~60	11	2	4	1		3	1	2
40~49	12			2	7	3		
30~39	16	1	2	4	4	3	2	
22~29	5	1			4	1		
Sum.	46	4	6	8	16	10	3	3

4.1.1.9 Research Budgets in the Materials Creation Research Station

Research budgets in the Materials Creation Research Station* (yen)

Kinds of budgets	1997	1998
STX-21 Budget	328,711,000	525,018,000
General Research	2,560,000	2,520,000
Pollution Prevention Research	19,978,000	32,095,000
Special Coordination Funds for Promoting Science and Technology	6,388,000	4,348,000
Supplementary Budgets		22,000,000
Summation	357,637,000	585,981,000

4.1.2 Joining and Interface Research Station

Chiaki Shiga (Supervising Researcher)

4.1.2.1 Research work

The most suitable welding methods, using arc, laser, brazing and other welding processes are studied to enhance the good properties to cover the weak ones in the welding joints, of the new steels which have been developed in the STX-21 projects. All of the physical phenomena in weldings are being fundamentally studied, especially to control the microstructure of the heat affected zones and reduce the residual stress. This study is linked to the improvement of welding joint properties.

The corrosion resistance steels, of which chemical elements determine fracture of the welding portion are investigated and spray coatings, such as HVOF, are also studied for their possibilities to resist corrosion in the severe sea environment.

The following subjects, in the frame work of the STX-21 projects, are promoted in the 6 Station Units:

- (1) Clarification of the mechanism on HAZ softening for ultra fine grain steels with low carbon equivalent.
- (2) Welding procedures to narrow the HAZ softening zone for ultra fine grain steels with low carbon equivalent.
- (3) Clarification of ductile-brittle fracture behavior of welding joints with HAZ softening.
- (4) Welding procedure to reduce residual stresses or to introduce compression stresses.
- (5) Welding procedure to increase the creep strength in the welding joint of heat resistant steels.
- (6) Development of corrosion resistant steels used for offshore and marine environments.
- (7) Development of suitable chemical compositions and welding procedures for high nitrogen bearing stainless steels.
- (8) Formation of coating films providing high resistance to corrosion on conventional steels with HVOF spray.
- (9) Investigation on non-destructive inspection to detect the microstructure, fault and residual stress-strain using ultrasonic, magnetic and laser speckle.

The members of the Joining and Interface Research Station, as at March 31, 1999, consist of 25 members, 8 domestic research fellows, and 2 STA fellows. The numbers of publications and patents are shown in the research activities of each Station Unit.

4.1.2.2 1st Unit

Akira Okada (ex-Unit Leader), Ken Sasabe (Unit Leader), Susumu Meguro, Tadayuki Otani (STX-21 research fellow)

4.1.2.2.1 Research work

- (1) Brazing experiments under microgravity were carried out using a sounding rocket. The experimental results showed that our prediction method for brazing phenomena under microgravity was reasonable.
- (2) The effect of surface compositions on solid state joining of some materials was studied. It was found that repetition of ion bombardment and heat treatment is effective in the joining of copper, and it was also found out that oxide on the surface improves the joint strength of FZ silicon.
- (3) The spot weldability of thin sheets of fine grain structure high tensile strength steel was investigated. It was made clear that this material shows very good spot weldability.
- (4) A computer simulation system for the selection of welding conditions were constructed and mounted in the on-line data base system of the NRIM.

<Research themes>

- (1) Brazing Experiment under Microgravity and Analyses of its Results (General Research: April 1996 to March 1998, Ken Sasabe)
- (2) Effects of Surface Elements on Diffusion Bonding of Silicon (General research: April 1995 to March 1998, Ken Sasabe)
- (3) Research of Lower Temperature Welding (Research on Structural Materials for 21st Century: April 1997 to March 2002, Ken Sasabe)

4.1.2.2.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) K. Sasabe, et al., Gap Penetration Phenomena of Molten Alloy under Microgravity, Final report of the experiment using TR-1A#5 small rocket, NASDA-TRM-970001, (1997) (in Japanese)
- (2) K. Sasabe, et al., Capillary Gap Penetration of Molten Alloy under Microgravity, J. JASMA, 14, 272(1997) (in Japanese)
- (3) K. Sasabe, et al., Brazing Experiment under Microgravity, Proc.JWS, IJ-31-97(1997) (in Japanese)
- (4) K. Sasabe, et al., Brazing under Microgravity, Proc.Japan-Canada Microgravity

Science Work Shop, Kyoto Mach 17-19, 1997

- (5) K. Sasabe, et al., Brazing Experiment using TR-1A Sounding Rocket, CAMP-
ISIJ .11, 970(1998) (in Japanese)

Oral Presentation: 10

- (1) International Conference: 1
(2) Domestic Conference: 9

Patent Application:

- (1)High Strength Steel and its Welding Method: T. Otani, K. Sasabe, 02/12/98,
10/343287(JAPAN)
(2)High Strength Steel and Welding Method: T. Otani, K. Sasabe, 02/12/98,
10/343385 (JAPAN)
(3)Method of High Frequency Resistance Welding and Shape Steel: T. Otani, K.
Sasabe, 02/12/98, 10/343386(JAPAN)
(4)Steel Pipes and Fabrication Method: T. Otani, K. Sasabe, 02/12/98,
10/343884(JAPAN)

4.1.2.3 2nd Unit

Kazuo Hiraoka (Unit Leader), Takeshi Fukushima, Seiji Kuroda, Terumi Nakamura, Yoshiaki Kawaguchi (domestic research fellow), Reisuke Ito (STX-21 research fellow), Philip Blazdell (STA fellow), Masao Ushio (guest researcher), Takayoshi Ohji (guest Researcher), Mikio Takemoto (guest researcher)

4.1.2.3.1 Research work

- (1) Both the ultra-narrow gap GMA welding process, which controls the arc heat input distribution, and the numerical simulation system of the GMA welding process have been developed in order to perform highly efficient welding, accompanied with obtaining high quality welded joints.
- (2) The HAZ softening behavior in the welding of ultra-fine grained high strength steels, being developed in the STX-21 project, has been evaluated by means of metallurgical analysis and fracture mechanics.
- (3) A high velocity oxy-fuel (HVOF) thermal spray technique has been employed to develop corrosion resistant coatings of various alloys, and their performance

evaluated by electrochemical methods.

- (4) TiC/Mo FGM coatings were carried out by plasma-spraying to fabricate a highly absorptive surface for solar radiation in space, and their thermo-physical properties were evaluated.
- (5) The surface strains at high temperatures during thermal shock tests of thermal barrier coatings (TBC), were measured in-situ using a the laser speckle strain meter and compared with the FEM calculation.

<Research themes>

- (1) Development of Arc Welding Process with Lower Heat Input Density for Ultra Fine Grained Microstructure Steel (Research on Structural Materials for 21st Century: April 1997 to March 2002, Kazuo Hiraoka)
- (2) Analysis of Softening and Fracture Processes in the Welded Joints of Ultra Fine Grained Microstructure Steel (Yoshiaki Kawaguchi, Research on Structural Materials for 21st Century: April 1997 to March 2002, Kazuo Hiraoka)
- (3) Development of Thermal Sprayed Corrosion Resistant Coatings for Steel Structures in Marine Environment (Research on Structural Materials for 21st Century: April 1997 to March 2002, Seiji Kuroda)
- (4) In-situ Evaluation of Degradation Process in Thermal Barrier Layered Structures (Special Coordination Funds for Promoting Science and Technology: April 1997 to March 1998, Seiji Kuroda)
- (5) Development of FGM Materials for Energy Conversion (Special Coordination Funds for Promoting Science and Technology: April 1996 to March 1998, Takeshi Fukushima)

4.1.2.3.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) K. Hiraoka: Plasma Structure of Ar-H₂ Gas Tungsten Arc by Some Spectroscopic Methods, Quarterly J. Japan Welding Society, 15, 259(1997) (in Japanese)
- (2) K. Hiraoka, Nobuo Sakuma, Johan Zipp: Energy Balance in Ar-He Mixed Gas Tungsten Arcs, Quarterly J. Japan Welding Society, 15,459(1997) (in Japanese)
- (3) K. Hiraoka: Emission Spectroscopic Measurement in Argon Arcs, J. Japan Welding Society, 67, 600(1998) (in Japanese)
- (4) K. Hiraoka: Plasma Structures of Ar-H₂ Mixed Gas Tungsten Arcs Determined by Spectroscopic Measurements, Welding Int., 12,186(1998) (Transaction of (1))

- (5) S. Kuroda, Y. Tashiro, H. Yumoto, S. Taira, T. Fukanuma: Measurement of Stress Development during HVOF Thermal Spray, Proc. 1st United Thermal Spray Conf., Indianapolis, USA, Sept. 805-811, 1997
- (6) S. Kuroda: Computer Simulation for Thermal Spray Technology, J. Surface Finishing Society of Japan, 48, 885(1997) (in Japanese)
- (7) S. Kuroda, Y. Tashiro, H. Yumoto, S. Taira, T. Fukanuma: Peening Action and Residual Stresses in HVOF Thermal Spraying of 316L Stainless Steel, Proc. 15th Int. Thermal Spray Conf., C. Coddet ed., Nice, France, May 569-574, 1998
- (8) S. Kuroda: Properties and Characterization of Thermal Sprayed Coatings –A Review of Recent Research Progress, Proc. 15th Int. Thermal Spray Conf., C. Coddet ed., Nice, France, May 539-550, 1998
- (9) S. Kuroda, Y. Tashiro, H. Yumoto, S. Taira, T. Fukanuma: Residual Stresses in HVOF Thermal Sprayed Coatings, Proc. Symp. for Welded Structures '97, 201-208, 1997 (in Japanese)
- (10) S. Kuroda, Y. Tashiro, H. Yumoto, S. Taira, T. Fukanuma: In-situ Measurement of Stress Generation in HVOF Thermal Sprayed Coatings, Quarterly J. Japan Welding Society, 17, 102(1999) (in Japanese)
- (11) T. Fukushima, S. Kuroda: High Temperature Properties of TiC/Mo Thermal Sprayed Coatings, Tokyo, Oct. 1998, submitted to Proc. FGM (in Japanese)
- (12) P. Blazdell, S. Kuroda: Thermal Spraying for the New Millennium, Materials World, April, 205(1999)
- (13) P. Blazdell, S. Kuroda: Plasma Spraying of Sub-micron Ceramic Suspensions by a Continuous Ink Jet Printer, submitted to Surface and Coatings Technology
- (14) C. Shiga: Progress in high strength structure steels in the past decade
Polish-Japanese Symp. on Environmental Effects on High Technology Materials, 1-11, 1997

• **Oral Presentation : 44**

- (1) International Conference: 3
- (2) Domestic Conference: 41

• **Patent Application :**

- (1) Welding Method: K. Hiraoka, T. Nakamura, 20/10/97, 09/287397 (JAPAN)
- (2) Welded Joint Structure, K. Hiraoka, T. Nakamura, 20/10/97, 09/287398, (JAPAN)
- (3) Arc Welding Method: K. Hiraoka, T. Nakamura, H. Yamamoto, 04/09/98, 10/251144 (JAPAN)

- (4) Arc Welding Method: K. Hiraoka, T. Nakamura, H. Yamamoto, 04/09/98, 10/251145 (JAPAN)
- (5) Welding Method and Welded Joint Structure: K. Hiraoka, T. Nakamura, 20/10/98, 09/175,563 (US)
- (6) Coating Method: S. Kuroda, P. Blazdell, 29/03/99, 11/086733 (JAPAN)
- (7) Corrosion Resistant Coating and its Fabrication Method: S. Kuroda, T. Fukushima, T. Kodama, 29/03/99, 11/086764, (JAPAN)

• **Awards :**

- (1) STA award, May 1997, K. Hiraoka
- (2) Best Paper Award of Symposium for Welded Structures'97, Nov. 1997, S. Kuroda,
- (3) Best Paper Award of Japan Welding Society, April 1998, K. Hiraoka
- (4) Best Paper Award of 15th International Thermal Spray Conference, May, 1998, Nice, France, S. Kuroda

4.1.2.4 3rd Unit

Susumu Tsukamoto (Unit Leader), Junichi Kinugawa, Yoshiki Muramatsu, Yoshikazu Asai, Fukuhisa Matsuda (guest researcher), Akira Matsunawa (guest researcher), Tomoyuki Kamata (graduate student)

4.1.2.4.1 Research work

- (1) Laser-plasma interaction has been investigated using a newly developed spectroscopic measurement procedure during CO₂ laser welding to elucidate the role of the plasma on the welding behavior and to establish a deep penetration defect-free weld using high power CO₂ laser.
- (2) The creep behavior of high-Cr ferritic heat resisting steel welded joints has been studied to clarify the mechanism of type IV cracking and to improve the creep lifetime of welded joints. It was found that low heat input welding, such as electron beam and laser welding, improved the creep lifetime of the joint due to a narrow heat affected zone.
- (3) A method for in-situ dynamic strain measurement has been developed using laser speckle interferometry to clarify the strain behavior of the weld metal and heat affected zone during welding.

<Research themes>

- (1) Development of Deep Penetration Welding Process using High Power CO₂ Laser (Research on Structural Materials for 21st Century: April 1997 to March 2002, Susumu Tsukamoto)
- (2) Non-contact Strain Measurement during Welding Procedure (Research on Structural Materials for 21st Century: April 1997 to March 2002, Yoshiki Muramatsu)
- (3) Creep Behavior of High Cr Ferritic Heat Resisting Steel Welded Joint (Research on Structural Materials for 21st Century: April 1997 to March 2002, Junichi Kinugawa)

4.1.2.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) S.Tsukamoto, K. Hiraoka, Y. Asai, H.Irie, M.Yoshino, T.Shida: Characteristics of Stably Induced Laser Plasma, Proc. Laser Materials Processing Conference, 81,B/77 (1997)
- (2) S.Tsukamoto, K.Hiraoka, Y.Asai, H.Irie : Characterization of Laser Induced Plasma in CO₂ Laser Welding, Proc. 5th Inter. Conf. on Trends in Welding Research, 431 (1998)
- (3) S.Tsukamoto: Introduction of Laser Group in NRIM —Fundamental Research on Laser-Plasma Interaction—, J. Japan Soc. of Laser Technology, 23, 53(1998) (in Japanese)
- (4) C.Shiga, A.Ohta, K.Hiraoka, S.Tsukamoto: Welding in Research Project on Frontier Structural Materials, J. Japan Welding Society, 66,43, (1997) (in Japanese)
- (5) T.Shida, T.Wakasa, S.Tsukamoto, K.Hiraoka: Measurement of Beam energy Absorption in CO₂ Laser Welding (Report II), Quarterly J. Japan Welding Society, in press (in Japanese)
- (6) J.Q.Guo, S.Tsukamoto, T.Kimura, H.Nakae: Nucleation Process Control of Undercooled Stainless Steel by External Nucleation Seed, Acta Mater. in press
- (7) X.Zhang, S.Tsukamoto:Theoretical Calculation of Nucleation Temperature and Undercooling Behaviors of Fe-Cr Alloys Studied with Electromagnetic Levitation Method, Metall. Mater. Trans., 30A, 1827, (1999)
- (8) H.Hongo, M.Yamazaki, T.Watanabe, J.Kinugawa, T.Tanabe, Y.Monma, T.Nakazawa : Creep Deformation Behavior of Weld Metal and Heat Affected Zones on 316 FR Steel Thick Plate Welded Joint, J. Soc. Mat. Sci., Japan, 48, 116, (1999) (in Japanese)

- (9) T. Watanabe, M. Yamazaki, H. Hongo, J. Kinugawa, T. Tanabe, Y. Monma: Long Term Creep-Rupture Properties and Microstructure of Weld Metal on 2.25Cr-1Mo Steel Thick Plate, J. Soc. Mat. Sci., Japan, **48**, 122, (1999) (in Japanese)
- (10) Y. Kaji, H. Tsuji, T. Sakino, M. Fujita, J. Kinugawa, Y. Tachi, J. Saito, S. Kano, K. Shimura R. Nakajima, S. Iwata: Retrieval Results on Various Properties of Superalloy Using Data-Free-Way (Joint Research) JAERI-Tech 99-007 (in Japanese)
- (11) Y.Muramatsu, S.Kuroda: Investigation on the Debonding of Thermal Sprayed Coatings under Thermal Shock Tests by using the Laser Speckle Strain Meter, Proc. Welded Structure Symp.'97, 209, (1997)
- (12) Y.Muramatsu, S.Kuroda: Monitoring Technique of Strains during Phase Transformation using Laser Speckle Method, J. Japan Welding Society, **67**,38, (1998) (in Japanese)

• **Oral Presentation: 26**

- (1)International Conference: 4
- (2)Domestic Conference: 22

• **Patent Application :**

- (1) Method and Equipment for Measuring Plasma Temperature: S.Tsukamoto, Y.Asai, Aug.31 1998, 10-246314
- (2) Method for Detection of Debonding and Adhesion of Surface Coatings, Y.Muramatsu, S.Kuroda, Nov.13, 1997, 09-312289
- (3) Method for Measuring Dynamic Strain in Welds, Y.Muramatsu, S.Kuroda, Jul.21,1998, 10-205702

4.1.2.5 4th Unit

Hiroaki Fukuhara, Ichizo Uetake, Hisashi Yamawaki

4.1.2.5.1 Research Work

- (1) A simplified ultrasonic computed-tomography (CT) system has been developed for detection of defects and structure change in the welded part of steel.
- (2) A magnetic flux leakage testing technique has been improved in sensitivity and accuracy, to detect smaller surface cracks in steels.

- (3) A newly developed computer simulation technique for ultrasonic propagation in solid has been utilized to clarify the mechanisms of various ultrasonic phenomena relating to material evaluation using ultrasound.

<Research theme>

- (1) Development of Nondestructive Measurement Technique for Fine Structure and Small Defects of Welding Steel (Research on Structural Materials for 21st century: April 1997 to March 2002, Kotobu Nagai)

4.1.2.5.2 Research Products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) I. Uetake, T. Saito: Magnetic Flux Leakage by Adjacent Parallel Surface Slots, NDT & International, 30, 371(1997)
(2) H. Yamawaki: Computer Simulation of Ultrasonic Propagation for Anisotropic Materials Evaluation, J. JSNDI, 47, 243(1998) (in Japanese)

• Oral Presentation: 8

- (1) International Conference: 0
(2) Domestic Conference: 8

4.1.2.6 5th Unit

Hiroyuki Masuda (Unit Leader), Hideshi Sumiyoshi, Naoki Washizu, Masahiro Yamamoto (STX-21 research fellow), Toshiya Nishimura (STX-21 research fellow), Kazuhiko Noda (domestic research fellow), T. Tsuru (guest researcher), T. Misawa (guest researcher), T. Shinohara (guest researcher)

4.1.2.6.1 Research work

Following research has been carried out.

- (1) Observation of adsorbed water was done by SPM to study the effect of humidity on the amount of adsorbed water.
(2) Observation of bacterial corrosion was done by AFM to study the mechanism of bacterial corrosion.
(3) Evaluation of surface oxide film was done by SPM with measuring I-V property to

seek the possibility of new method of evaluating corrosion resistance.

- (4) In-situ observation of atmospheric corrosion of iron was done by KFM to study the mechanism of atmospheric corrosion.
- (5) In-situ X-ray analysis of atmospheric corrosion was done to study the change in the structure of corrosion produced during the corrosion process.
- (6) Quantification of sea salt particles was done by QCM to standardize the amount of sea salt particles.

<Research themes>

- (1) Evaluation of Atomic Scale Damage of Materials in Aqueous Solution
(General Research: April 1995 to March 1998, Hiroyuki Masuda)
- (2) Development of Metals Having Cleaning Property of Environment by Photo-Catalysis
(Special Coordination Funds for Promoting Science and Technology: April 1995 to March 1998, Hiroyuki Masuda)
- (3) Improvement of Corrosion Resistance of Structural Steels in Marine Environment
(Research on Structural Materials for 21st Century: April 1996 to March 2001)

4.1.2.6.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) N. Washizu, H. Masuda: AFM Observation of Iron-oxidizing Bacteria on Surfaces of Corroded Metals, J. Japan Inst. Metals, **61**, 481 (1997) (in Japanese)
- (2) H. Masuda, S. Matsuoka: Effect of Young's Modulus on Fatigue Crack Propagation under Variable Loading, Proc. 9th Inter. Conf. Fracture, **3**, 1557 (1997)
- (3) H. Masuda: Observation of Water Droplet on Various Materials by Non-Contacting AFM, J. Japan Inst. Metals, **62**, 173 (1998) (in Japanese)
- (4) H. Masuda: Observation of Adsorbed Water on Various Materials by AFM and FT-IR, J. Japan Inst. Metals, **62**, 617 (1998) (in Japanese)
- (5) H. Masuda: Observation of Adsorbed Water Droplets on Gold Surface, J. Japan Inst. Metals, **62**, 961 (1998) (in Japanese)
- (6) H. Masuda: Possibility of Observation of Water Film by Measuring Surface Potential Distribution, J. Japan Inst. Metals, **62**, 1183 (1998) (in Japanese)
- (7) S. Matsuoka, H. Masuda: Damage Evaluation of Micromaterials, J. Soc. Mat. Sci. Japan, **47**, 534(1998) (in Japanese)
- (8) H. Masuda: Scanning Probe Microscope, Ferum, **3**, 884 (1998) (in Japanese)
- (9) H. Masuda: Recent Progress in the Study of Surface Observation Using Scanning

- Probe Microscopy, Material Transactions, JIM, 3, 161 (1999)
- (10) H. Masuda, M. Yamamoto: Observation of Sea Salt Particle by AFM, Zairyo-to-Kankyo, 48, 307 (1999) (in Japanese)

• **Oral Presentation : 18**

- (1) International Conference: 0
(2) Domestic Conference: 18

• **Patent Application :**

- (1) Measurement Device for Sea Salt Particles, M. Yamamoto, H. Masuda, 19/5/1998, 137219
(2) Quantification Method of Sea Salt Particles, M. Yamamoto, H. Masuda, 19/5/1998, 137218
(3) Evaluation Method of Corrosion Resistance for Metals, H. Masuda, 10/11/1998, 318929

• **Award :**

- (1) Award for Achievement in Material Chemistry (Award from Japan Institute for Metals in 1998), H. Masuda

4.1.2.7 6th Unit

Yasuyuki Katada (Unit Leader), Shigeo Ohashi, Katsutoshi Kurosawa, Hideki Uno (STX-21 research fellow), Yasushi Kikuchi (Guest researcher), Shinji Fujimoto (Guest researcher), Isao Sekine (Guest researcher), Kazuko Kamezaki (Technician), Atsuko Shimizu (Technician)

4.1.2.7.1 Research work

- (1) Forms of localized damage of structural materials in light water reactor environments, such as corrosion fatigue, stress corrosion cracking, etc., have been investigated in high-temperature water.
- (2) Mitigation of the enlargement of local damage by laser beam technique is one of the issues of interest.
- (3) Pitting corrosion and crevice corrosion in stainless steels have also been studied, associated with the development of anti-corrosion structural materials for marine use.

<Research themes>

- (1) Effect of Aging Degradation on Localized Corrosion of Structural Materials for Light Water Reactors (Nuclear Energy Research: April 1996 to March 2000, Yasuyuki Katada)
- (2) Development of anti-corrosion stainless steels for marine-use (Research on Structural Materials for 21th Century: April 1998 to March 2002, Yasuyuki Katada)

4.1.2.7.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) Y.Katada, S.Sato: Effect of Crevice on Low Cycle Fatigue Behavior of Pressure Vessel Steel in High Temperature Pressurized Water, Proc. of Symp. on Localised Corrosion and Environmental Cracking (SOLCEC), 1997 (Kalpakkam, India).
- (2) Y.Katada, S.Sato: Effect of Temperature on Corrosion Fatigue Behavior of Low Alloy Steels in High Temperature Water, Proc. of the 8th Inter. Symp. on Environmental Degradation of Materials in Nuclear Power System, 1997 (Florida, USA)
- (3) Y.Katada, S.Sato: The Role of MnS Inclusions on Environmentally Assisted Cracking in High Temperature Water, ASME Pressure Vessel and Piping, (1999).
- (4) Y.Katada, S.Ohashi: Interaction of CF/SCC of Structural Materials for LWR in High Temperature Water, J. Pressure Vessel and Piping (Accepted).
- (5) K. Ei, H. Irie, T. Kasugai: Corrosion Property for Solid State Bonded Joint of SUS304L Stainless Steel and Zr, Tetsu-to-Hagane, 85, 5, 53(1999). (in Japanese)

• Oral Presentation : 11

- (1) International Conference: 2
- (2) Domestic Conference: 9

• Patent Application :

- (1) Stainless Steels: H. Uno, Y. Katada, 25/2/1999, 049103
- (2) Acceleration Testing Method for Weathering Low Alloy Steels: K. Kurosawa, T. Kodama (1999)

4.1.2.8 Researchers in the Joining and Interface Research Station

Age and research fields of researchers in the Joining and Interface Research Station.

(March 31, 1999)

Age of Researcher	Number	Metal-lurgy	Welding /Joining	Chem-istry	Corro-sion	Cera-m-ics	Electrical Engineering	Mechanical Engineering
60 ~	4		2	1				1
50 ~ 60	15	3	6	1	2		1	2
40 ~ 49	11		2		6		1	2
30 ~ 39	5	2	2		1			
22 ~ 29	4				2	1		1
Summation	39	5	12	2	11	1	2	6

4.1.2.9 Research Budgets in the Joining and Interface Research Station

Research budgets in the Joining and Interface Research Station.(yen)

Kinds of budgets	1997	1998
Research on Structural Materials for 21 st Century	207,317,000	247,183,000
General Research	13,890,000	5,670,000
Special Coordination Funds for Promoting Science and Technology	28,325,000	12,874,000
Nuclear Energy Research	16,200,000	13,056,000
Supplementary Budgets	0	141,350,000
Summation	265,732,000	420,133,000

4.1.3 Strength and Life Evaluation Research Station

Toshihiko Takahashi (Supervising Researcher)

4.1.3.1 Research Work

The Strength and Life Evaluation Research Station has been conducting research on analyses and evaluation of newly developed steels and welded structures. These studies have been carried out in collaboration with the Materials Creation Research Station and the Joining and Interface Research Station. In order to accomplish the study's aims, the development of advanced facilities, in the form of nanoscopic and atom-scale analysis, and the establishment of standard evaluation methods and accelerated test methods have been achieved. Examples of outcomes are shown next.

(1) Research on 800MPa steel:

- 1) It has been made possible to increase the fatigue strength of welded joints of steel structures markedly by using newly developed weld metals.

(2) Research on 1500MPa steel:

- 1) The optimum microstructure of martensitic steels against delayed fracture have been revealed using nanoscale analysis instruments developed here.
- 2) A new strengthening mechanism of martensitic steel was presented by nanoscopic analysis.
- 3) An advanced concept on controlling of non-metallic inclusion for improving long term fatigue properties was established.

(3) Research on ferritic heat resistant steel:

- 1) An accelerated evaluation method for creep, based on the creep deformation rate, has been proposed.
- 2) The creep deformation characteristics of ferritic heat resistant steels can be related to in service microstructure changes in these steels.
- 3) Methods for improving the oxidation resistance of ferritic heat resistant steels have been developed.
- 4) Improvement in the accuracy of high temperature fatigue test has been achieved.

In addition to these studies, collaborative research on the Creep and Fatigue Data Sheet project, the VAMAS Project, and evaluation of nuclear energy plant materials have also been conducted at this Station. These studies are carried out in close relation to researches on the Materials for 21st Century.

The members of Strength and Life Evaluation station, as at March 31, 1999, consisted of 24 researchers, 1 STA fellow, 1 domestic research fellow, 2 STX-21 research fellows, 1 joint-doctoral course student, and 9 guest researchers.

The number of publications, including submitted papers, are about 60, patents submitted are 10 between April 1, 1997 to March 31, 1999. The details are shown in the research activities of each research unit.

4.1.3.2 1st Unit

Akihiko Ohta (Unit Leader), Naoyuki Suzuki, Yoshio Maeda, Ninh.T. Nguyen (STA Fellow)

4.1.3.2.1 Research work

- (1) Efforts were applied to change residual welding stress from tensile to compressive stress by using a low transformation temperature welding wire. The fatigue strength of welded joints can be increased up to about three times that of the conventional ones by using a newly developed wire.
- (2) A method evaluating the fatigue strength and yield strength magnitude, of welded components which have high tensile residual stress has been established. The method outline is contained in the draft ISO standard.
- (3) Evaluations of the fatigue strength of welded joints in synthetic sea water and of the random loading effect on the fatigue of notched members have been conducted.

<Research themes>

- (1) Development of 800-Mpa-Class High-Strength, Ferrite Matrix Steels with Improved Weldability (Research on Structural Materials for 21st Century: April 1997 to March 2002, Kotobu Nagai)
- (2) Fatigue Data Sheet Project- I (Materials Data Sheet Project: April 1997 to March 2002, Hirosada Irie)
- (3) Evaluation of Fatigue Strength of Welded Joints in Synthetic Sea Water (Special Coordination Funds for Promoting Science and Technology: April 1995 to March 1997, Akihiko Ohta)
- (4) Random Loading Fatigue of Notched Members (General Research: April 1997 to March 1998, Akihiko Ohta)

4.1.3.2.2 Research products, including papers submitted (April 1, 1997 to March 31,1999)

- (1) A. Ohta, N. Suzuki, Y. Maeda: Effect of Residual Stress on Fatigue of Weldment, Proc. Inter. Conf. Performance of Dynamically Loaded Welded Structures, IIW(1997)108-122.
- (2) A. Ohta, N. Suzuki, Y. Maeda: Unique Fatigue Threshold and Growth Properties of Welded Joints in Tensile Residual Stress Field, Inter. J. Fatigue, 19,S303-S310(1997).
- (3) N. T. Nguyen, A. Ohta, K. Matsuoka, N. Suzuki, Y. Maeda, Analytical Solutions for Transient Temperature of Semi-Infinite Body Subjected to 3D Moving Heat Sources, Welding Journal, August(1999), in press.
- (4) A. Ohta, N. Suzuki, Y. Maeda, K. Hiraoka, T. Nakamura, Superior Fatigue Crack Growth Properties in Newly Developed Weld Metal, Inter.J.Fatigue(1999), in press.
- (5) D. S. Tchankov, A. Ohta, N. Suzuki, Y. Maeda, Random Loading Life Assessments for Notched Plates, Fatigue & Fracture Engineer. Materials & Structures, submitted.
- (6) D. S. Tchankov, A. Ohta, N. Suzuki, Y. Maeda, Fractography of Random Fatigue Fractured Notched Specimens, Materials Characterisation, submitted. (in Japanese)

Oral Presentations: 24

- (1) International Conference: 5
- (2) Domestic Conference: 19

Patent Application

- (1) Welding Methods and Weld Metals: A.Ohta,T.Siga,S.Nishijima, O.Watanabe, Japan, U.S., Korea, U.K., Germany and France, 1997.12.25, 09-358023
- (2)Repair Methods and Weld Metal for Repair of Metal Parts: A.Ohta, N.Suzuki, Y.Maeda, K.Hiraoka, O.Watanabe, T.Kubo, Japan, 1998.2.25, 10-043864
- (3)Welding Methods: A.Ohta, N.Suzuki, Y.Maeda, Japan, 1998.9.8, 10-253903

4.1.3.3 2nd Unit

Saburo Matsuoka (Unit Leader), Takayuki Abe, Etsuo Takeuchi, Kensuke Miyahara, Nobuo Nagasima, Hisasi Hirukawa, Masao Hayakawa, Takahito Ohmura, Kohichi Tanaka (guest researcher), Toyonobu Yoshida (guest researcher), Hideo Kobayashi (guest researcher), Kenjiro Komai (guest researcher)

4.1.3.3.1 Research work

The fatigue and brittle fracture mechanisms of steels have been studied. The former study is related to the ultra-steels and fatigue data sheet projects. The latter was started since many steel structures, such as high-rise buildings, were brittle fractured during the great Hanshin-Awaji earthquake of January 17, 1995. Nanofractography by atomic microscope and nanoindentation by an ultra-micro hardness tester have been also studied in order to clarify the strength and fracture mechanisms of steels, thin films, and other materials at the nanoscopic level.

<Research themes>

- (1)Advanced Ultra-High-Strength Steels (1500-MPa-Plus Class) (Research on Structural Materials for 21st Century: April 1997 to March 2001, Saburo Matsuoka)
- (2)Ductile versus Brittle Behavior of Structural Steels (General Research, April 1992 to March 1998, Saburo Matsuoka)
- (3)Mechanical Properties of Thin Films and Coatings (Special Coordination Funds for Promoting Science and Technology(through the VAMAS collaborative research project): April 1997 to March 1999, Saburo Matsuoka)
- (4)NRIM Long-Term Fatigue Data Sheet Project I (Material Data Sheet: April 1997 to March 2002, Hirosada Irie)

4.1.3.3.2 Research products including submitted papers (April 1,1997 to March 31,1999)

- (1) K. Miyahara, S. Matsuoka, N. Nagashima: Nanoindentation Measurement for Tungsten (001) Single Crystal, Trans. JSME, 63A, 2220(1997), JSME Inter. J., 41, 562(1998)
- (2) K. Miyahara, N. Nagashima, S. Matsuoka, T. Ohmura: Evaluation of Vickers Hardness by AFM Nanoindentation Tester, Trans. JSME, 64A, 2567(1998) (in Japanese)
- (3) K. Miyahara, S. Masuoka, N. Nagashima: Nanoindentation Measurement for Tungsten (001) Single Crystal, Proc. Inter. Conf. on Advanced Technology in Experimental Mechanics (ATEM '97), 327 (1997)
- (4) K. Miyahara, N. Nagashima, T. Ohmura, S. Matsuoka: Evaluation of Mechanical Properties in Nanometer Scale Using AFM-based Nanoindentation Tester, NanoStruct. Mater., 12, 1049(1999)
- (5) H. Hirukawa, S. Matsuoka, E. Takeuchi, T. Omura, K. Yamaguchi, K. Tsuzaki: High Resistance of Fatigue Crack Growth for Austenitic Stainless steel Containing

- Nitrogen, Trans. JSME, 65A, 1343(1999) (in Japanese)
- (6) N.Nagashima, K.Miyahara, S.Matsuoka: Hardness Measurement of Small Cementite Particles Using an AFM Ultramicro Hardness Tester, Trans.JSME, 64A, 536(1998) (in Japanese)
 - (7) N.Nagashima, S.Matsuoka, K.Miyahara: AFM Ultra-Micro Hardness Test for Inclusions in High Strength Steel, Trans.JSME, 65A, 477(1999) (in Japanese)
 - (8) N.Nagashima, K.Miyahara, S.Matsuoka: Hardness Measurement of Small Cementite Particles in 0.25 Carbon Steel using an AFM Ultramicro Hardness Tester, JSME Inter. J. A42, 235(1999)
 - (9) T.Abe, T.Ohmura, S.Matsuoka: Surface and Internal Fatigue Fracture Mechanisms for High Strength Steels, submitted to Trans. JSME (in Japanese)
 - (10) M.Hayakawa, S.Mitsuno, C.Masuda, K.Kanazawa, H.Ichikawa: Bending Property at Elevated Temperature for Advanced Woven Cloth BN Coated SiC Fiber-Reinforced SiC Composite, Proc. JSME, Inter. Conf. on Materials and Mechanics '97, 357(1997)
 - (11) E. Takeuchi, H. Hirukawa, S. Matsuoka : Arrest Function in Elevated-temperature Fatigue Crack Growth of High Alloy with a Small Addition of Zirconium, Trans. JSME, 65A, 148(1999) (in Japanese)
 - (12) E. Takeuchi, H. Hirukawa, S. Matsuoka, S. Yamada, H. Akiyama, Y. Matsumoto: Fractography of Beam-to-Column Welded Connections by Full Scale Shaking Table Test, submitted to J. Struct. Constr. Eng. Architectural Institute of Japan (in Japanese)
 - (13) T. Ohmura, T. Suzuki: Ultra-microindentation testing at elevated temperature, Inter. Conf. on Advanced Technology in Experimental Mechanics. Wakayama July 25-26, 1997
 - (14) T. Ohmura, A. Shimamoto: Nanoindentation devices developed in Japan and its application to thin films, Hi-hakai-kensa. 47, 364(1998). (in Japanese)
 - (15) S.Matsuoka, H.Masuda: Micromaterials IV: Damage Evaluation of Micromaterials, J. SMSJ, 47, 534(1998) (in Japanese)
 - (16) K.Ito, A.Yamaguti, Y.Wada, K.Iwata, M.Morisita, O.Miyake, N.Aoto, K.Kobayashi, K.Shiba, Y.Yasumoda, S.Matsuoka: Technical Report on Monju's Sodium Leak Incident, J. At. Energy Soc. Japan, 39 , 704(1997) (in Japanese)
 - (17) H.Akiyama, S.Ymada, Y.Matsumoto, S.Matsuoka, F.Ohtake, H.Sugimoto: Transition from Ductile Fracture to Brittle Fracture of Full Scale Beam-to-Column Connections Caused by Temperature, J. Struct. Constr. Eng. Architectural Institute of Japan, to be published (in Japanese)

- (18) H.Akiyama, S.Yamada, Y.Matsumoto, S.Matsuoka, K.Ogura, H.Kitamura: Study on Fracture of Beam-to-Column Connections by Means of Full Scale Shaking Table Test, J. Struct. Constr. Eng. Architectural Institute of Japan, 512, 165(1998) (in Japanese)
- (19) S.Matsuoka: Ultra-Micro Hardness Tester, p262, “*Tribology of Materials for Electrical Engineering and Electronics*”, 1999.3, ed A. Kohno, Realize Co. LTD (in Japanese)

Oral Presentation: 48

- (1) International Conference : 3
- (2) Domestic Conference : 45

Patent Application

- (1)Hardness Testing Method in Micro-scale: K. Miyahara, S. Matsuoka, N. Nagashima, March 26, 1998, Heisei10-078169, Registration: January 29, 1999.1.29, Registration No.2879679
- (2)High-Fatigue-Strength Steel: T. Ohmura, H. Hirukawa, S. Matsuoka, M. Murayama, K. Hono, K. Tsuzaki, Feb. 27, 1999,11-0967665
- (3)Cantilever with a Long Plobe for Atomic Force Microscope: S.Matsuoka, M.Hayakawa, T.Takahashi , September 28, 1998, Application No.390002901,

Awards:

- (1) JSME Medal for Outstanding Paper (1998.3), S. Matuoka, K. Miyahara, N. Nagashima, K. Tanaka: Measurement of Elastic Modulus and Yield Stress by AFM Ultra-Micro-Hardness Tester
- (2) Commendation by the Minister of State for Science and Technology (1998.5), Saburo Matsuoka: Fatigue Damage Control

4.1.3.4 3rd Unit

Fujio Abe (Unit Leader), Hideo Tanaka, Kiyoshi Kubo, Masaharu Murata, Masaaki Tabuchi, Toshio Ohba, Hideaki Kushima, Osamu Kanemaru, Eiji Baba, Kenji Yokokawa, Masaru Shimizu, Hideko Miyazaki, Nobuyuki Fujitsuna (STX-21 research fellow), Toshiaki Horiuchi (STX-21 research fellow)

4.1.3.4.1 Research work

- (1) The creep rupture strength has been improved to develop advanced 9Cr steels for thick section boiler components of 650 °C ultra-supercritical (USC) plants.
- (2) The long-term, such as 10^5 h, creep and creep rupture behavior has been shown to be quite different from that of the short-term for many kinds of heat resistant steels and alloys, resulting from a complex microstructural evolution that occurs during creep.
- (3) A penning discharge micro-sputtering technique has been successfully applied for etching creep tested specimens as an advanced etching method, instead of the conventional chemical method.
- (4) The strain induced by the tetragonal to monoclinic phase transformation in partially stabilized zirconia ceramics has been shown to be accommodated by dislocations as well as microcracks.
- (5) A standard test method for creep crack growth of brittle materials has been established through the VAMAS collaborative research project.

<Research themes>

- (1) Strategic Research on Advanced Ferritic Steels for 650 °C USC Boilers (R & D of Structural Materials for 21st Century: April 1997 to March 2002, Fujio Abe)
- (2) Creep Data Sheet Project-V (Materials Strength Data Sheet Research: April 1996 to March 2000, Hirosada Irie)
- (3) Characterization of Creep-Damaged Microstructure of Stainless Steels by Computer Aided Quantitative Metallography (General Research: April 1995 to March 1998, Fujio Abe)
- (4) Stability of Tetragonal Phase and its Effect on High-Temperature Mechanical Properties of Zirconia (General Research: April 1996 to March 1999, Fujio Abe)
- (5) Evaluation Method of High Temperature Fracture Property for Creep Brittle Materials (Special Coordination Fund's Research for Promoting Science and Technology (through the VAMAS collaborative research project): April 1997 to March 2000, Masaaki Tabuchi)

4.1.3.4.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) F. Abe : Evolution of Microstructure and Acceleration of Creep Rate in Tempered Martensitic 9Cr-W Steels, Materials Science and Engineering, A234-236, 1045 (1997)

- (2) H. Tanaka, M. Murata, F. Abe, K.Yagi : The Effect of Carbide Distributions on Long-Term Creep Rupture Strength of SUS321H and SUS347H Stainless Steels, *Materials Science and Engineering*, **A234-236**, 1049(1997)
- (3) H. Tanaka, M. Murata, F. Abe, K.Yagi : Effect of Grain Boundary Precipitates on Long-Term Creep Rupture Properties for SUS 347H Steel, *Testu-to-Hagane*, **83**, 72 (1997) (in Japanese)
- (4) H. Tanaka, E. Nishikawa, F. Abe, K.Yagi, T. Sugita : A New Preparation Technique of Surface to Microstructural Examination of Heat Resisting Steels and Alloys by Using Penning Discharge Micro-Sputtering, *Testu-to-Hagane*, **83**, 263 (1997) (in Japanese)
- (5) T. Ohba, O. Kanemaru, K. Yagi, C. Tanaka : Long-Term Stress Relaxation Properties of NCF 800H alloy, *J.Soc. Mater. Sci.* **46**,19(1997) (in Japanese)
- (6) M. Murata, F. Abe, K. Yagi : Development of Monitoring System for Creep Tests, Report of the 123 Committee on Heat-Resisting Materials and Alloys, **38**, 87 (1997) (in Japanese)
- (7) O. Kanemaru, M. Shimizu, T. Ohba, H. Miyazaki, F. Abe, K. Yagi : Remaining Life Prediction by The Iso-stress Method of Boiler Tubes after Prolonged Service, *Mater. Sci. Res. Inter.* **3**, 31 (1997)
- (8) T. Ohba, O. Kanemaru, K. Yagi, C. Tanaka : Long-Term Stress Relaxation Properties and Microstructural Changes of NCF 800H, *Mater. Sci. Res. Inter.* **3**, 10 (1997)
- (9) F. Abe, K. Yagi: Evaluation of Long-Term Creep and Rupture Properties of Heat Resisting Steels, 4th Int. Charles Parsons Turbine Conf., 750, Newcastle, UK, November 4-6, 1997
- (10) M.Tabuchi, K.Kubo, K.Yagi : Evaluation of Creep Crack Growth Rate in terms of Creep Fracture Mechanism for 316 Stainless Steel, 9th Inter. Conf. on Fracture, 399, Sydney, April 1-5, 1997
- (11) K.Yagi, M.Tabuchi, K.Kubo, J.J.Kim, Y.H.Huh : The Influence of Fracture Mechanisms on the Creep Crack Growth Behavior of 316 Stainless Steel, *Engineering Fracture Mechanics*, **57**, 463-473(1997)
- (12) F. Abe : The Longest Creep Tests in the World, *Materia Japan*, **37**,7(1998) (in Japanese)
- (13) F. Abe : Kinetics of Carbide Precipitation during Creep and Its Effect on Creep Rate of 10Cr-30Mn Austenitic Steels, *Materials Trans., JIM*, **39**,211(1998)
- (14) H. Tanaka, F. Abe, K. Yagi, T. Sugita : Evaluation of Creep Damaged Microstructure in Austenitic Stainless Steels by Surface Observations, *Testu-to-*

- Hagane, 84, 303(1998) (in Japanese)
- (15) H. Tanaka, E. Nishikawa, F. Abe, K. Yagi, T. Sugita : Replication Technique for Microscopic Examination of Microstructures Etched by Penning Discharge Micro-Sputtering, Trans. JSME, 64, 795(1998) (in Japanese)
 - (16) H. Tanaka, E. Nishikawa, F. Abe, K. Yagi, T. Sugita : Non-Destructive Microstructure Examination of Boiler Tubes Using Penning Discharge Micro-sputtering Technique, Z. Metallkde, 89, 375 (1998)
 - (17) F. Abe, M. Igarashi, N. Fujitsuna, K. Kimura, S. Muneki : Alloy Design of Advanced Ferritic Steels for 650 °C USC Boilers, Inter. Conf. on Advanced Heat Resistant Steels for Power Generation, San Sebastian, Spain, April 20-25, (1998) 84.
 - (18) F. Abe, M. Igarashi, N. Fujitsuna, K. Kimura, S. Muneki : Research and Development of Advanced Ferritic Steels for 650 °C USC Boilers, 6th Liege Conf. on Materials for Advanced Power Engineering, Liege, Belgium, October 5-7, (1998) 259.
 - (19) K. Kubo, M. Tabuchi, K. Yagi, A.T. Yokobori, A. Fuji : Evaluation of Creep Crack Growth Prorate of High Temperature TiAl Intermetallic Compound, Report of the 123 Committee on Heat-Resisting Materials and Alloys, 39, 237 (1998) (in Japanese)
 - (20) H. Kushima, K. Kimura, F. Abe, K. Yagi, H. Irie, K. Maruyama : Effect of Microstructure on the Long-term Creep Deformation Properties of 2.25Cr-1Mo Steels, Report of the 123 Committee on Heat-Resisting Materials and Alloys, 39, 61 (1998) (in Japanese)
 - (21) H. Kushima, K. Kimura, F. Abe : Effect of Microstructural Change on the Long-term Creep Strength Properties of Modified 9Cr-1Mo Steel, Report of the 123 Committee on Heat-Resisting Materials and Alloys, 39, 335 (1998) (in Japanese)
 - (22) N. Fujitsuna, H. Kutsumi, T. Itagaki, M. Igarashi, F. Abe : Basically Investigation for Alloy Design of High Cr Ferritic Steels for Use at Higher Temperature, Report of the 123 Committee on Heat-Resisting Materials and Alloys, 39, 265 (1998) (in Japanese)
 - (23) M. Tabuchi, K. Kubo, K. Yagi, A.T. Yokobori, A. Fuji : Results of a Japanese Round Robin on Creep Crack Growth Evaluation Methods for Ni-base Superalloys, Engineering Fracture Mechanics, 62, 47(1999)
 - (24) A. Fuji, M. Tabuchi, A.T. Yokobori, Jr, T. Yokobori : Method of Creep Crack Growth Test with Improved Starter Notch for Brittle Materials, Engineering Fracture Mechanics, 62, 23(1999)

- (25) M. Tabuchi, A.T. Yokobori, A. Fuji, K.Kubo, K. Yagi, T. Yokobori : Creep Crack Growth Test Method and Creep Crack Growth Properties for TiAl Intermetallic Compound, J. Soc. Mater. Sci. (in press) (in Japanese)
- (26) H. Kushima, K. Kimura, F. Abe, K. Yagi, H. Irie, K. Maruyama : Evaluation of Long-Term Creep Strength Properties of Cr-Mo Type Heat Resistant Steels based on Creep Deformation Analysis, Report of the 123 Committee on Heat-Resisting Materials and Alloys, 40, 1(1999) (in Japanese)
- (27) F. Abe: Creep test method in Handbook of Metals, JIM, in press (in Japanese)

Oral presentation: 60

- (1) International Conference : 9
- (2) Domestic Conference : 51

Patent Application :

- (1) High Cr Ferritic Heat Resistant Steels with Sufficient Oxidation Resistance and Sufficient Steam-oxidation Resistance: Nobuyuki Fujitsuna, Fujio Abe, Takehiko Itagaki, Masaaki Igarashi, 9 September 1997, 09-256481
- (2) Ferritic Heat Resistant Steels and their Preparation Methods: Nobuyuki Fujitsuna, Fujio Abe, Takehiko Itagaki, Masaaki Igarashi, Seiichi Muneki, Kazuhiro Kimura, Hideaki Kushima, 21 September 1998, 09/157.392 (USA)
- (3) Ferritic Heat Resistant Steels and Their Preparation Methods : Nobuyuki Fujitsuna, Fujio Abe, Takehiko Itagaki, Masaaki Igarashi, Seiichi Muneki, Kazuhiro Kimura, Hideaki Kushima, 21 September 1998, 98307629.0 (Germany, Belgium)

Award :

- (1) Nishiyama Memorial Award, The Iron and Steels Institute of Japan, April 1998, Fujio Abe
- (2) Technical Skill Award, The Iron and Steels Institute of Japan, March 1999, Eiji Baba

4.1.3.5 4th Unit

Kohzo Nakazawa (Unit Leader), Takehiko Itagaki, Hiroyuki Kutsumi(domestic research fellow)

4.1.3.5.1 Research work

- (1) The oxidation properties of high Cr ferritic steels in high temperature steam have been studied as a function of small amounts of additional elements. It was found that the addition of Pd extremely improved the oxidation resistance of steels in high temperature steam. An addition of Si together with a small amount of active elements like as Ti or Y was also effective to decrease the oxidation of ferritic steels.
- (2) Coating of SiC on alumina powder was tried by fluidized bed CVD as to improve the wettability of ceramic particles with molten cast iron.

<Research themes>

- (1) Development of High Performance Ferritic Heat Resistant Steels for USC Boiler
(Research on Structural Material for 21st Century: April 1997 to March 2002, Fujio Abe)
- (2) Fabrication of Highly Functional Composite Cast Irons (Special Coordination Funds for Promoting Science and Technology: April 1996 to March 1999, Akira Sato)

4.1.3.5.2 Research Products (April 1, 1997 to March 31, 1999)

Oral Presentation: 3

- (1) International Conference: 0
- (2) Domestic Conference: 3

Patent Application:

- (1) A method to Improve the Oxidation Resistance of High Temperature Alloys and Steels, T. Itagaki, H. Kutsumi, N. Fujitsuna, M. Igarashi, M. Muneki, F. Abe, February 26, 1999, No.11-52009

4.1.3.6 5th Unit

Koji Yamaguchi (Unit Leader), Masuo Shimodaira, Kazuo Kobayashi and Megumi Kimura

4.1.3.6.1 Research work

- (1) Creep-fatigue interaction of newly developed ferritic heat resisting steels for fossil power plants have been evaluated by using tensile strain hold wave shapes as a

simulation of the daily start-up and shut-down operation.

- (2) The time-dependence of the low-cycle fatigue properties of 316FR austenitic stainless steel for fast breeder reactors have been studied as a parameter of strain rate, strain hold time, and testing temperature.
- (3) Tests on the NIRM Fatigue Data Sheet of high temperature fatigue have been carried out.

<Research themes>

- (1) Strategic Research on Advanced Ferritic Steels for 650°C USC Boilers (Research on Structural Materials for 21st Century: April 1997 to March 2002, Fujio Abe)
- (2) Long-term Creep-fatigue Properties of 316FR Stainless Steel for Fast Breeder Reactor (Elaluation of Technological Development for Diversification of Power Source: April 1995 to March 2001, Koji Yamaguchi)
- (3) NIRM Fatigue Data Sheet Project (Materials Strength Data Sheet: April 1997 to March 2002, Hirotsada Irie)

4.1.3.6.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) K.Yamaguchi, K.Kobayashi, S.Nishijima, K.Kanazawa: Fatigue Properties of High Temperature Materials, Inspection Engineering, 2, 1(1997) (in Japanese)
- (2) K.Kobayashi, K.Yamaguchi, S.Kato, T.Fujioka, T.Nakazawa, H.Koto, S.Date: High Temperature Fatigue Life of the 316 FR Steel, Report 123 Committee of Japan Soc. Promo. Sci., 38, 25(1997) (in Japanese)
- (3) K.Kobayashi, K.Yamaguchi: The Effect of Compressive Strain Hold on the Low Cycle Fatigue Life of Various Materials at Elevated Temperature, JHPI, 36, 27(1998) (in Japanese)
- (4) K.Kobayashi, K.Yamaguchi, S.Kato, S.Nishijima, T.Fujioka, T.Nakazawa, H.Koto, S.Date: High Temperature Fatigue Properties of the 316FR Steel, Trans. Japan Soc.Mech.Eng. 64, 545(1998) (in Japanese)
- (5) M.Kimura, K.Yamaguchi, S.Matuoka: Fatigue Fracture Mechanism Map of SUS 304 Steel, Report 123 Committee of Japan Soc. Promo. Sci., 39, 101(1998) (in Japanese)
- (6) M.Shimodaira, K.Yamaguchi: Fatigue Properties of TiAl Intermetallic Compounds and Dependences of the Microstructure, J. Soc.Mater.Sci. 48, 166(1999) (in Japanese)

Oral Presentation: 7

(1) International Conference: 1

(2) Domestic Conference: 6

4.1.3.7 Researchers in the Strength and Life Evaluation Research Station

Age and research fields of researchers in the Strength and Life Evaluation Research Station. (March 31, 1999)

Age of Researchers	Number	Metallurgy		Mechanics		Nanoscale analysis
		Structural Steel	Heat Resistant Steel	Fatigue	Creep	
60 ~	1	1	0	0	0	0
50 ~ 60	12	5	4	3	0	0
40 ~ 49	14	1	3	7	3	0
30 ~ 39	8	0	2	2	0	4
22 ~ 29	2	0	2	0	0	0
Summation	37	7	11	12	3	4

4.1.3.8 Research Budgets in the Strength and Life Evaluation Research Station

Research budgets in the Strength and Life Evaluation Research Station (Yen)

Kinds of budgets	1997	1998
Research on Structural Materials for 21 st Century		
General Research	13,660,000	5,040,000
Special Coordination Funds for Promoting Science and Technology	32,047,000	22,369,000
Evaluation of Technological Development for diversification of Power Source	44,471,000	43,854,000
Summation	90,178,000	71,236,000

CHAPTER 5 Innovative & Evolutional Research

Innovative and evolutional research for materials science has been promoted by the four Research Teams. The first research team was established April 1, 1993. The innovative and evolutional research is performed by the Biomaterials Research Team, the Ecomaterials Research Team, the Aperiodic Materials Research Team, and the Combustion Synthesis Research Team. Other research teams may be established on the emergence of new research fields, and social demand while the current research teams are successively evaluated.

5.1 Biomaterials Research Team

Takao Hanawa (Team Leader)

5.1.1 Staff

Takao Hanawa (Team Leader), Kozo Nakazawa, Norio Maruyama,
Akiko Yamamoto, Sachiko Hiromoto, Masae Sumita (guest researcher)

5.1.2 Research Work

Following the 5th Long-term Plan, basic research to develop biomaterials for a high quality of life in an aging society has been promoted. Biomaterials are implanted into the human body to recover functions lost due to accidents, diseases, or the aging processes. The staff in the team are making every effort to create biocompatible and biointegrated materials. High safety, affinity, and durability are required of biomaterials to be used in the human body. Fundamental and systematic data are being accumulated in order to develop new materials with superior properties. For conventional and newly developed materials, safety and affinity are examined using cultured cells, corrosion resistance is estimated using electrochemical techniques, and mechanical durability is tested using a newly developed hip-joint simulator and a fretting fatigue tester. On the basis of the fundamental data obtained, amorphous and ion beam techniques are utilized for the development of high-performance biomaterials. The development of amorphous alloys as biomaterials is assisted by Aperiodic Materials Research Team.

Examples of the research outcomes are shown as follows.

- (1) An original hip joint simulator has been developed and a simulation corresponding to a 10-year walk estimated.
- (2) A fretting fatigue tester using artificial body fluids with an *in vivo* dissolved oxygen

concentration and temperature has been developed and the effect of the artificial body fluid on the fretting fatigue has been elucidated.

- (3) An advanced fretting fatigue tester for small size specimens has been developed.
- (4) Cytotoxicity of metal ions and particulate of metal oxides were quantitatively and systematically evaluated.
- (5) Part of the mechanisms of the cytotoxicity of metal ions is elucidated in respect of DNA damage and the cell cycle.
- (6) New systems to measure directly the detachment force of a single cell adhering to a material's surface by applying a lateral load by a cantilever has been developed and the cell adhesive shear strength and cell detachment surface energy measured.
- (7) The mechanical and biochemical causes of metal ion release from titanium implant materials were elucidated.
- (8) The effect of the component elements of Pd-based and Zr-based amorphous alloys on the corrosion resistance of alloys in artificial body fluids was investigated.
- (9) Titanium-based amorphous alloys for biomaterials have been developed.
- (10) Calcium-ion-mixing titanium has been developed.

The output of the research activities has been increased since the start of the 5th Long-term Plan. The number of publications, including submitted papers, are 36, patents submitted are 1, between April 1, 1977 to March 31, 1999.

<Research themes>

- (1) Research on Highly Biocompatible Structural Materials for Biomedical Use
(Special Research: April 1994 to March 1999, Takao Hanawa)
- (2) Research on Creation of Biointegrated Materials to Improve Physically Handicapped People's Quality of Life
(Special Coordination Funds for Promoting Science and Technology: April 1997 to March 2000, Takao Hanawa)

5.1.3 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) M. Sumita: Durability of Metallic Biomaterials, J. Jpn. Soc. Biomater., 15, 240 (1997) (in Japanese)
- (2) M. Sumita: Present Status and Future Trend of Metallic Materials Used in Orthopedics, Orthop. Surg., 48, 927 (1997) (in Japanese)
- (3) M. Sumita: "Chemical and Mechanical Stresses in Living Body Caused by Metallic Biomaterials", Biomedical Materials Research in the Far East (III), Chengdu,

China, July 15-17, 1997

- (3) A. Yamamoto, R. Honma, M. Sumita: Comparison of Metal Salt's Cytotoxicity between Cell Lines, Biomedical Materials Research in the Far East (III), Chengdu, China, July 15-17, 1997
- (4) A. Yamamoto, S. Mishima, N. Maruyama, M. Sumita: "Evaluation of Shearing Cell Adhesive Strengths of Murine Fibroblasts to Metal Films and Extra Cellular Matrices", Biomedical Materials Research in the Far East (III), Chengdu, China, July 15-17, 1997
- (5) M. Sumita: Trend of Research on Metallic Biomaterials, J. Jpn. Soc. Heat Treat., **38**, 36 (1998) (in Japanese)
- (6) A. Yamamoto: Damage of Metallic Biomaterials in a Human Body and Biological Response, J. Mater. Sci. Soc. Jpn, **35**, 264 (1998) (in Japanese)
- (7) A. Yamamoto, S. Mishima, N. Maruyama, M. Sumita: A New Technique for Direct Measurement of the Shear Force Necessary to Detach a Cell from a Material, Biomaterials, **19**, 871 (1998)
- (8) N. Maruyama, K. Nakazawa, M. Sumita, M. Sato: Effects of Heat Treatment and Amount of SiC Particles on Fatigue and Fretting Fatigue Strength of Particulate Reinforced A2024 Alloy Composite, J. Jpn. Inst. Met., **62**, 224 (1998) (in Japanese)
- (9) A. Yamamoto, R. Honma, M. Sumita: Cytotoxicity Evaluation of 43 Metal Salts Using Murine Fibroblasts and Osteoblastic Cells, J. Biomed. Mater. Res., **39**, 331 (1998)
- (10) T. Hanawa, K. Asami, K. Asaoka: Repassivation of Titanium and Surface Oxide Film Regenerated in Simulated Bioliquid, J. Biomed. Mater. Res., **40**, 530 (1998)
- (11) M. Sumita, A. Yamamoto: Measurement of Cell Adhesive Shear Force to Material's Surface, J. Phys. Med., **9**, 10 (1998).
- (12) T. Hanawa: Surface Modification of Metallic Biomaterials, Bull Jpn. Inst. Met., **37**, 853 (1998)
- (13) M. Sumita: Corrosion and Mechanical Properties of Metallic Biomaterials, Bull. Jpn. Inst. Met., **37**, 859 (1998) (in Japanese)
- (14) T. Hanawa: Behavior of Titanium in Biological Systems, Bull. Kanagawa Dent. College, **26**, 121 (1998) (in Japanese)
- (15) T. Hanawa: Surface Modification of Titanium Implants that Improve Bone Conduction, 4th Inter. Symp. on Titanium in Dentistry, Geneva, Switzerland, September 2-4, 1998
- (16) T. Hanawa and S. Hiromoto: Surface Properties and Modification of Material Surfaces that Influence Biocompatibility, Corros. Eng., **47**, 750 (1998). (in

Japanese)

- (17) T. Hanawa: Interface Design of Metallic Materials by Ion Beam Technology, in *Biocompatibility of Biomaterials*, eds. by A. Sato, T. Ishikawa, Y. Sakurai, (Nakayama Shoten, Tokyo 1998). (in Japanese)
- (18) T. Hanawa: Calcium Ion Implanted Titanium for Biomaterials, 5th Inter. Symp. on Electrochemical/Chemical Reactivity of Novel Materials, Sendai, October 5-6, 1998.
- (19) T. Hanawa: Surface of Metallic Biomaterials in vivo and its Modification, Corrosion and Protection Committee of Japanese Society for Materials 208th Meeting, Environmental Resistance of Bio-Medical Materials, Osaka, January 22, 1999 (in Japanese)
- (20) T. Hanawa: Surface of Metallic Biomaterials and their Modification, Inorg. Mater. 6, 136 (1999) (in Japanese)
- (21) S. Hiromoto, H. Numata, A.-P. Tsai, K. Nakazawa, T. Hanawa, M. Sumita: Polarization Behavior of Pd₇₈Si₁₆Cu_{6-x}Cr_x Amorphous Alloys in an Artificial Body Fluid, J. Jpn Inst. Met., 63, 352 (1998) (in Japanese)
- (22) T. Hanawa: Surface Modification of Metallic Biomaterials Improving Hard Tissue Compatibility, Inter. Symp. on Advanced Biomaterials & Tissue Engineering, Tokyo, February 22-23, 1999
- (23) S. Hiromoto, A.-P. Tsai, M. Sumita, T. Hanawa: Application of Zirconium-based Amorphous Alloys to Biomaterials, Inter. Symp. on Advanced Biomaterials & Tissue Engineering, Tokyo, February 22-23, 1999
- (24) T. Hanawa: Properties of Metallic Materials for Artificial Organs, Artificial Organs, 23, 685 (1999) (in Japanese)
- (25) K. Nakazawa, M. Sumita, N. Maruyama: Fatigue and Fretting Fatigue of Austenitic and Ferritic Stainless Steels in Pseudo-Body Fluid, 7th Inter. Fatigue Conf., FATIGUE'99, Beijing, China, June 8-12, 1999
- (26) T. Hanawa: Surface Modification of Metallic Biomaterials with Ion Beam Technology, Ionics, 25, 3 (1999) (in Japanese)
- (27) T. Hanawa: In vivo Metallic Materials and Surface Modification, Mater. Sci. Eng. A 267, 260(1999)
- (28) T. Hanawa: Surface Modification of Metallic Biomaterials, J. Jpn. Soc. Heat Treat., (in press) (in Japanese)
- (29) T. Hanawa: Surface Phenomena of Metallic Materials *in vivo* and Surface Modification, J. Surf. Sci. Soc. Jpn., 20, 607 (1999) (in Japanese)
- (30) Y. Mu, T. Kobayashi, M. Sumita, A. Yamamoto, T. Hanawa: Metal Ion Release from

- Titanium with Active Oxygen Species Generated by Rat Macrophages *in vitro*, J. Biomed Mater. Res. (in press)
- (31) A.Yamamoto, R.Honma, A.Tanaka, M.Sumita: Generic Tendency of Metal Salt Cytotoxicity for Six Cell Lines, J. Biomed. Mater. Res., 47, 396 (1999)
 - (32) S.-K. Lee, K. Nakazawa, M. Sumita, N. Maruyama: Effects of Contact Load and Contact Curvature Radius of Cylinder Pad on Fretting Fatigue in High Strength Steel, in *Fretting Fatigue: Current Technology and Practices, ASTM STP 1367*, eds. by D. W. Hoepfner, V. Chandrasekaran, C. B. Elliott, (ASTM, West Conshohocken, PA 1999)
 - (33) T. Hanawa: "Other Relevant Data", in *Foreign Bodies, Surgical Implants and Prosthetic Devices, IARC Monograph Vol. 74*, (International Agency for Research on Cancer, World Health Organization, Lyon 1999) (in press)
 - (34) A. Yamamoto, S. Mishima, N. Maruyama, M. Sumita: Quantitative Evaluation of Cell Attachment to Glass, Polystyrene, Fibronectin- or Collagen-coated Polystyrene by Measuring Cell Adhesive Shear Force and Cell Detachment Energy, J. Biomed. Mater. Res. (in press)
 - (34) Y. Mu, T. Kobayashi, M. Sumita, K. Tsuhi, T. Hanawa: J. Biomed. Mater. Res. (Submitted)

• **Oral Presentation:** 66

- (1) International Conference: 6
- (2) Domestic Conference: 60

• **Patent Application:**

- (1) Bone-compatible Titanium Materials: T. Hanawa, K. Asami, September 28, 1998, 10-KN-53
- (2) Structure of Specimen Chamber and Fretting Pads for Fatigue Test in Liquids: N.Maruyama, K.Nakazawa, March 10, 1999, No.3058177

• **Awards:**

- (1) Award for Scientific Research Merits, Japanese Society for Biomaterials, October 16, 1998 M., M. Sumita
- (2) Intelligent Materials Forum TAKAGI AWARD '99, March 16, 1999 A. Yamamoto, S. Mishima, M. Sumita, T. Hanawa

5.1.4 Researchers in the Biomaterials Research Team

Age and research fields of researchers in the Biomaterials Research Team

(March 31, 1999)

Age of Researchers	Number of researchers	Mechanical Properties	Surface and Corrosion	Cell Biology
60 ~	1	1	0	0
50 ~ 60	1	1	0	0
40 ~ 49	2	1	1	0
30 ~ 39	1	0	0	1
22 ~ 29	1	0	1	0
Summation	6	3	2	1

5.1.5 Research Budgets in the Biomaterials Research Team

Research budgets in the Biomaterials Research Team (yen)

Kinds of budgets	1997	1998
Special Research	17,978,000	11,036,000
Special Coordination Funds for Promoting Science and Technology	23,017,000	22,771,000
Supplementary Budgets	15,000,000	0
Summation	55,995,000	33,807,000

5.2 Ecomaterials Research Team

Kohmei Halada (Team Leader)

5.2.1 Staff

Kohmei Halada (Team Leader), Hiroshi Yoshizu, Kazumi Minagawa, Kiyoshi Ijima, Tatsuhiko Aizawa (guest researcher), Shushi Miura (guest researcher), Ryoichi Yamamoto (guest researcher -1997)

5.2.2 Research work

"Ecomaterial" is a new concept of material's R&D which considers the global environmental issue. This concept and the technical approaches to realize it were proposed and developed in Japan, one year before the "United Nations Conference on Environment and Development (Rio de Janeiro)". The NRIM has given attention to the importance of the environmental performance of metals and materials, and established the Ecomaterials research team in the 5th Long-term Plan, going ahead of any of the other materials research institutes around the world.

Ecomaterials research prospects the future course of design and the development of materials which harmonize with the environment or minimize the life-cycle environmental load without deteriorating their properties. Recyclable materials, pollutant-free materials, materials with a lower consumption of energy and resources, and materials adaptable to DfE (Design for Environment) are the main targets of ecomaterials. The Ecomaterials Research Team in the NRIM has been working as the center of research on ecomaterials in Japan.

Between 1993 and 1998 the STA research project "Study on Design and Assessment of Ecomaterials" was carried out. The team played an important role in the project by developing the assessment methods of ecomaterials and preparing the materials-data required for assessment. In 1998 the team conducted the STA feasibility study "Life-cycle Design and Processing of Materials to Reduce the Environmental Load", and in result, a new STA research project, "Barrier-free Processing to Reduce the Life-cycle Environmental Load of Materials" was started in 1999.

The concrete activities of the team cover two areas. One is the assessment technology concerning ecomaterials. Methodological research to develop LCA (environmental life-cycle assessment) to a method which can be used in materials design and selection from an environmental viewpoint. Inventory data of metals for the assessment are prepared. The emissions of CO₂, NO_x, SO_x in the metallurgical stage of steel alloys and non-ferrous alloys were calculated and presented as databases with an interactive Web

interface. Not only the inventory data of metals but also that of parts and products are estimated and listed in the database “environmental load of 4000 social stocks”.

The second activity is the processing for ecomaterials, where typical attempts at recycling materials and the materials processing of recycled raw materials are subjected of research. Preparation of fiber reinforced porous Fe-Fe composite, composed from SCIFER (highly drawn iron fiber) and Fe powder, was investigated by using P/M technology. This Fe-Fe composite is expected to have high recyclability because the microstructure of the SCIFER reinforces the matrix without using any alien substance nor any alloying elements. From an opposite view point, positive utilization of contaminants which comes from recycling is investigated. Cu in Fe recycled scrap is known to obstruct the sound processing in hot-rolling. A new warm powder processing method where Cu is finely dispersed in the matrix as a strengthening element is investigated.

<Research themes>

- (1) Fundamental Study on the Processing for Ecomaterials (General Research: April 1996 to March 1998, Kohmei Halada)
- (2) Study on Design and Assessment of Ecomaterials (Special Coordination Funds for Promoting Science and Technology: April 1993 to March 1998, Kohmei Halada)
- (3) Life-cycle Design and Processing Materials to Reduce the Environmental Load (Special Coordination Funds for Promoting Science and Technology, April 1998 to March 1999, Kohmei Halada)

5.2.3 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) K.Halada, K.Ijima, K.Yagi: Estimation of the Emissions of CO₂, SO_x, NO_x of Steel Alloys, J. Mater. Res., 13, 2514(1998)
- (2) K.Halada, K.Ijima, K.Yagi: Estimation of the Emissions of CO₂, SO_x, NO_x of Non-ferrous Metals and Alloys in Japan, Int. J. of Lifecycle Assessment, in press
- (3) K.Halada: Problems and Possibilities of Powder Metallurgy from the Viewpoint of Environmental Issue, Materia Japan, 37, 42(1998) (in Japanese)
- (4) K.Halada: Modern Approach toward the Sustainable Society – Industrial Ecology-, Powder Science & Engineering, 30, 23(1998)
- (5) K.Halada: How should Ecomaterials be?, Boundary, 15, 14(1999) (in Japanese)
- (6) K.Halada: Current Status of Recycling Metals, J. of the Japan Welding Society, 67, 20(1998) (in Japanese)

- (7) K.Halada: Current and Future of Ecomaterials, *Handbook of environmental and recycling technology*, Kogyochosakai, pp.27-33(1999) (in Japanese)
- (8) K.Halada: Ecomaterials, *Kogyozairyo*, 47, 22(1999) (in Japanese)
- (9) K.Halada: Which material can reduce CO₂ emission? *Kogyozairyo*, 46, 106(1998) (in Japanese)
- (10) K.Halada: Realization of Ecomaterials which Reduce the Environmental Load, *M&E*, No.10,200(1998)
- (11) K.Halada: LCA of Metals, *Wrought Copper*, in press
- (12) K.Halada: Development of MLCA to an Assessment Tool in Coming Dematerialization Era, *Proc. 3rd Inter. Conf. on Ecomaterials*, pp.299-304(1997)
- (13) K.Halada: The Requirements for LCI Data Model as Tool of Life Cycle Engineering, *Proc. 3rd Inter. Conf. on EcoBalance*, pp.67-72(1998)
- (14) S.Sakai, K.Halada, Y.Yokoyama: Sensitivity Analysis in LCA Using Perturbation Method, *Proc. the 3rd Inter. Conf. on EcoBalance*, pp.55-58(1998)
- (15) K.Halada: Progress of Ecomaterials Research towards Sustainable Society, *Proc. Inter. Conf. of Ecodesign 99*, pp.212-215(1999)
- (16) K.Halada, K.Ijima, K.Minagawa: Recyclability and Life-cycle Environmental Loadings of PM Products, *Proc. of 1998 Powdermetallurgy World Congress*, pp.58-62(1998)
- (17) K.Ijima, K.Halada, K.Yagi: Environment Load Database System of Alloys on the Network, *Proc. 3rd Inter. Conf. on Ecomaterials*, 333(1997)
- (18) T.Abe, H.Onodera, K.Kimura, K.Halada, K.Ijima: Improvement of the Ecobalance of Ferritic Steels by the Minimum Alloying, *Proc. 3rd Inter. Conf. on Ecomaterials*, 47(1997)
- (19) K.Ijima, K.Halada, K.Yagi: Database of Environment Load of 4000 Social Stocks, *Proc. 3rd Inter. Conf. on Ecobalance*, 531(1998)
- (20) H.Yoshizu, K.Ijima, K.Halada, K.Yagi: Environmental Load Data as Part of Materials Data Base, *Proc. 3rd Inter. Conf. on Ecobalance*, 575(1998)
- (21) H. Yoshizu, K. Halada: Construction of Open System for Materials Database (I) - Development of Creep Rupture Database -, *Proc. 35th Symp. on Strength of Materials at High Temperatures*, 144 - 148 (1997) (in Japanese)
- (22) H. Yoshizu, K. Halada: Construction of Open System for Materials Database (II)- Link of the Variety Database -, *Proc. 36th Symp. on Strength of Materials at High Temperatures*, pp.133 - 137(1998) (in Japanese)
- (23) K.Minagawa, K.Halada, H.Okuyama, S.Ohno, N.Itsubo: Production of Recyclable Fe-Fe Composite Materials, *Proc. 3rd Inter. Conf. on Ecomaterials*, 351(1997)

• **Oral Presentation:** 30

(1) International Conference: 10

(2) Domestic Conference: 20

• **Patent Application**

(1) Fine Pulverization and Decoration Method of Powder Particle, K.Minagawa, K.Halada, S.Ohno, H.Okuyama, (1997), Patent No.2916611

(2) Preparing Method of Fine Powder of Metal, K.Minagawa, K.Halada, (1998). No.09-256972

• **Award :**

(1) JIM Award for Achievements: 1998, K.Halada

5.2.4 Researchers in the Ecomaterials Research Team

Age and research fields of researchers in the Ecomaterials Research Team.

(March 31, 1999)

Age of Researchers	Number of researchers	Metallurgy	Mechanical Engineering	Electric Engineering
60 ~	0	0	0	0
50 ~ 60	0	0	0	0
40 ~ 49	3	1	2	0
30 ~ 39	1	0	0	1
22 ~ 29	0	0	0	0
Summation	4	1	2	1

5.2.5 Research Budgets in the Ecomaterials Research Team

Research budgets in the Ecomaterials Research Team (yen)

Kind of Budgets	1997	1998
General Research	7,500,000	7,500,000
Special Coordination Funds for Promoting Science & Technology	15,373,000	10,012,000
Summation	22,873,000	17,512,000

5.3 Aperiodic Materials Research Team

An Pang Tsai (Team Leader)

5.3.1 Staff

An Pang Tsai (Team Leader), Taku J. Sato, Hiroyuki Takakura, Jing Xing Guo, Donna Perera (STA fellow), Fabrice Dugain (STA fellow), Shin Takeuchi (guest researcher), Kiyoshi Aoki (guest researcher)

5.3.2 Research work

The main research is focussed on the production of new materials with quasicrystalline and amorphous structures; preparation of new quasicrystalline alloys, growing single quasicrystals, production of bulk amorphous alloys, and the preparation of nanocomposites. The materials are studied by X-ray diffraction, neutron scattering, transmission electron microscopy (TEM), differential scanning calorimetry (DSC) and solid analyzer.

<Research theme>

- (1) Fabrication of Quasicrystals and Investigations of Physical Properties (Core Research for Evolutional Science and Technology, Japan Science and Technology Corporation: from October 15, 1996 to November 31, 2001, An Pang Tsai)

5.3.3 Research Products, including papers submitted(April 1, 1997 to March 31,1999)

- (1) A. P. Tsai, A. Niikura, A. Inoue, T. Masumoto: Stoichiometric Icosahedral Phase in the Zn-Mg-Y System, *J. Mater. Res.* **12**, 1468(1997).
- (2) T.J. Sato, E. Abe, A.P. Tsai: A Novel Decagonal Quasicrystal in Zn-Mg-Dy System, *Jpn. J. Appl. Phys.*, **36**, L1038(1997).
- (3) A.P. Tsai, A. Inoue, T. Masumoto: Phason Strains on Growth, Stability, and Structure of Icosahedral Phases, *Prog. Crystal Growth and Charact.* **34**, 221(1997).
- (4) H. Saito, K. Fukamichi, T. Goto A.P. Tsai, A. Inoue, T. Masumoto: Concentration Dependence of the Magnetic Properties of Melt-Quenched P-Type $Mg_{30}Gd_xZn_{70-x}$ Quasicrystals, *J. Alloys and Compounds*, **252**,6(1997).
- (5) A.P. Tsai, A. Niikura, K. Aoki, T. Masumoto: Hydrogen Absorption in an Icosahedral Zn-Mg-Y Alloy, *J. Alloys and Compounds* **253-254**, 90(1997).
- (6) K. Saitoh, K. Tsuda, M. Tanaka, A. P. Tsai: Structural Study of an $Al_{72}Ni_{20}Co_8$

- Decagonal Quasicrystal Using the High- Angle Annular Dark-Field Method, *Jpn.J. Appl. Phys.*, **36**, L1400(1997).
- (7) Z.M.Stadnik, D.Purdie, M.Garnier, Y.Baer, A.P.Tsai, A.Inoue, K.Edagawa, S.Takeuchi, K.H.Buschow: Electrical Structure of Quasicrystals Studied by Ultrahigh-Energy-Resolution Photoemission Spectroscopy, *Phys. Rev. B* **55**, 10938(1997).
 - (8) A. Singh, A.P. Tsai: Heterogeneous Nucleation of Lead on Quasicrystals, *Philo.Mag. Lett.*, **77**, 89(1998).
 - (9) Alok Singh, E.Abe, A.P.Tsai: A Hexagonal Phase Related to Quasicrystalline Phases in Zn-Mg-Rare-Earth System, *Philo.Mag. Lett.* **77**, 95(1998).
 - (10) T.J.Sato, T.Hirano, A.P.Tsai: Single Crystal Growth of the Decagonal Al-Ni-Co Quasicrystal, *J. Cryst. Growth*, **191**, 545(1998).
 - (11) E. Abe, T.J. Sato, A.P. Tsai: The Structure of Frank-Kasper Decagonal Quasicrystal in the Zn-Mg-Dy System: Compariosn with the Al-Ni-Co System, *Philo. Mag. Lett.*, **77**, 205(1998).
 - (12) T.J.Sato, E.Abe, A.P.Tsai: Composition and Stability of the Decagonal Quasicrystals in the Zn-Mg-RE Systems, *Philo. Mag. Lett.*, **77**, 213(1998).
 - (13) M.Terauchi, H.Ueda, M.Tanaka, A.P.Tsai, A.Inoue, T.Masumoto: Electron-Energy-Loss Spectroscopy Study of a Stable Decagonal Quasicrystal Al-Ni-Rh, *Philo. Mag. Lett.*, **77**, 1625(1998).
 - (14) T.J.Sato, H.Takakura, A.P.Tsai: Single Crystal Growth of the Icosahedral Zn-Mg-Ho Quasicrystal, *Jpn. J. Appl. Phys.*, **37**, L663(1998).
 - (15) A. Singh, A.P.Tsai: Crystallography and Solidifichation Behariour of Nanometric Pb Particles Embedded in Icosahedral and Decagonal Quasicrystalline Matrix, *Acta Mater.* **46**, 4641(1998).
 - (16) H.Takakura, A.Sato, A.Yamamoto, A.P.Tsai: Crystal Structure of a Hexagonal Phase Related to Zn-Mg-Y Quasicrystalline Phase, *Philo. Mag. Lett.* **78**, 263(1998).
 - (17) T.J. Sato, H. Takakura, A.P. Tsai K. Shibata: Anisotropic Spin Correlation in the Zn-Mg-Ho Quasicrystal, *Phys. Rev. Lett.*, **81**, 2364(1998).
 - (18) P.J. Steinhardt, H.-C. Jeong, K. Saitoh, M. Tanaka, E. Abe, A.P. Tsai: *Nature* **365**, 55(1998).
 - (19) A.P. Tsai, Y. Kitazawa, A. Inoue, T. Masumoto: Ferromagnetic Glasses with Stable Supercooled Liquid in Gd-Al-(Cu,Ni,Co) Alloys, *High Temp. Mater. Proc.* **17**, 203(1998).
 - (20) Y. Yan, S.J. Pennycook and A.P. Tsai: Direct Imaging of Local Chemical Disorder and Columnar Vacancies in Ideal Decagonal Al-Ni-Co Quasicrystals, *Phys. Rev.*

Lett. **81**, 5145(1998).

- (21) F. Dugain, M.de Boissieu, K. Shibata, R. Currat, T.J. Sato, A.R. Kortan, J.-B. Such, K. Hradil, F. Frey, A.P. Tsai: Inelastic Neutron Scattering Study of the Dynamics of Decagonal Al-Ni-Co Phase, *Eur.Phys.J.B7*, 513(1999).
- (22) E. Abe, T.J. Sato, A.P. Tsai: Structure of a Quasicrystal without Atomic Clusters, *Phys. Rev. Lett.*, **82**, 5269(1999).
- (23) E. Abe, A.P. Tsai: Quasicrystal-Crystal Transformation in Zn-Mg-Rare Earth Alloys, *Phys. Rev. Lett.*, **83**, 753(1999).
- (24) E. Abe, H. Takakura, A. Singh, A.P. Tsai: Hexagonal Superstructure in the Zn-Mg-rare-earth Alloys, *J. Alloy, Compound*, **283**, 169(1999).
- (25) J.Q. Guo, T.J. Sato, T. Hirano, A.P. Tsai: Solid-liquid Interface in the Growth of a Decagonal $\text{Al}_{72}\text{Co}_{16}\text{Ni}_{12}$ Quasicrystal, *J. Cryst. Growth*, **197**, 963(1999).
- (26) D.N. Perera, A.P. Tsai: Dynamics Tensile Measurements for $\text{Pt}_{60}\text{Ni}_{15}\text{P}_{25}$ below the Calorimetric Glass Transition Temperature, *J. Phys.; Condens. Matter* **11**, 3029(1999).
- (27) K. Saito, T. Yokozawa, M. Tanaka, A.P. Tsai: Structural Studies of Monoclinic Approximants of $\text{Al}_{13}\text{Fe}_4$ and τ^2 -inflated $\text{Al}_{13}\text{Co}_4$ by the High-Angle Annular Dark-field Method, *J. Electron Microscopy* **48**(2), 105(1999).
- (28) K. Saito, K. Tsuda, M. Tanaka, A.P. Tsai: Structural Study of an $\text{Al}_{70}\text{Ni}_{15}\text{Fe}_{15}$ Decagonal Quasicrystal Using High-Angle Annular Dark-Field Scanning Transmission Electron Microscopy, *Jpn. J. Appl. Phys.* **38**, L671(1999).
- (29) A.P. Tsai, T.J. Sato, J.Q. Guo, T. Hirano: Growing Perfect Quasicrystals, *J. Non-Cryst. Solids*, 1999, in press.
- (30) A. Singh, A.P. Tsai: Stability of Interface between Lead Particles and Quasicrystals and Its Effect on the Melting Temperature of the Lead Particles, *Philos. Mag. Lett.*, **79**, 561(1999).
- (31) A.P. Tsai: The Path to Discover the Quasicrystals, *Proc. New Horizons in Quasicrystals*, eds. A.I. Goldman, D.J. Srodelet, P.A. Thiel, J.M. Dubois (1997 World Scientific)pp.1-8.
- (32) K. Tsuda, K. Saitoh, M. Terauchi, M. Tanaka, A.P. Tsai, A. Inoue, T. Masumoto: Convergent-Beam Electron Diffraction and Electron Microscope Studies of Decagonal Quasicrystal, *Proc. 6th Int. Conf. on Quasicrystals*, eds. S. Takeuchi and T. Fujiwara (World Scientific Singapore 1998)pp11-18.
- (33) K. Saitoh, K. Tsuda, M. Tanaka, A.P. Tsai: Structure Analysis of Decagonal Quasicrystal by Convergent-Beam Electron Diffraction, *Proc. 6th Int. Conf. on Quasicrystals*, eds. S. Takeuchi and T. Fujiwara (World Scientific Singapore 1998)pp.

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- (34) A.P.Tsai: Developments in Quasicrystals: New Systems, Single Crystal and Better Structure Perfection, Proc. 6th Inter. Conf. on Quasicrystals, eds. S. Takeuchi and T. Fujiwara (World Scientific Singapore 1998) p253-260.
- (35) A.Fujiwara, A.Inoue, A.P.Tsai: Stability and Structural Variation of Decagonal Phase in Al-Ni-Co System, Proc. 6th Inter. Conf. on Quasicrystals, eds. S. Takeuchi and T. Fujiwara (World Scientific Singapore 1998) pp341-344.
- (36) A.Inaba, A.P.Tsai, K.Shibata: Vibrational Properties of Quasicrystals of Al-Cu-Ru, Al-Pd-Re and Al-Pd-Mn Deduced from Heat Capacities, Proc. 6th Inter Conf. on Quasicrystals eds. S. Takeuchi and T. Fujiwara (World Scientific Singapore (1998) pp.443-450.
- (37) Z.M.Stadnik, D.Purdie, M.Garnier, Y.Baer, A.P.Tsai, A.Inoue, K.Edagawa, S.Takeuchi: Electronic Structure of Quasicrystals Studied by Ultrahigh-Energy-Resolution Ultraviolet Photoemission Spectroscopy, Proc. 6th Inter. Conf. on Quasicrystals, eds. S.Takeuchi and T.Fujiwara (World Scientific Singapore(1998) pp.563-570.
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- (41) E. Abe, A.P. Tsai: High Resolution Electron Microscopy Study on Zn-Mg-Y Icosahedral Quasicrystal, Proc. Aperiodic '97 (World Scientific Singapore (1998) pp.235-239.
- (42) T.J. Sato, T. Hirano, A.P. Tsai: Single-Crystal Growth of the Decagonal Al-Ni-Co Quasicrystal, Proc. Aperiodic '97 (World Scientific Singapore (1998)pp.523-527.
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- (47) H. Takakura, T.J. Sato, A.P. Tsai, A. Sato, A. Yamamoto: Crystal Structure of Hexagonal Phases and its Relation to Icosahedral Quasicrystalline Phase in Zn-Mg-RE(RE=Rare Earth) System, MRS Symp. Proc. Vol. 553 (1999)pp.129-134.
- (48) K. Saito, K. Tsuda, M. Tanaka, A.P. Tsai: Structural Studies of Decagonal Quasicrystals and Approximants Using the High-Angle Annular Dark-Field Method, MRS Symp. Proc. Vol. 553(1999)pp.177-172.
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- (51) A.P. Tsai: Metallurgy of Quasicrystals: Alloys and Preparation, MRS Bulletin November, (1997)pp.43-47.
- (52) A.P. Tsai: Metallurgy of Quasicrystals, in *Physical Properties of Quasicrystals*, ed. Z. Stadnik, Springer Series in Solid-State Science(Springer-Verlag, Berlin, 1999)pp.5-50.

Oral Presentation: 66

- (1) International Conference: 19,
- (2) Domestic Conference : 47

Award

- (1) Japan Institute of Metals, Contribution Award (March, 1999), A.P. Tsai

5.3.4 Researchers in the Aperiodic Materials Research Team

Age and research fields of researchers in the Aperiodic Materials Research Team.

(March31,1999)

Age of Researchers	Number	Metallurgy	Crystal-lography	Physics	Chemistry
60 ~	0	0	0	0	0
50 ~ 60	0	0	0	0	0
40 ~ 49	1	1	0	0	0
30 ~ 39	4	1	1	2	0
20 ~ 29	1	0	0	0	1
Summation	6	2	1	2	1

5.3.5 Research Budgets in the Aperiodic Materials Research Team

Research budgets in the Aperiodic Materials Research Team.(yen)

Kinds of budgets	1997	1998
Core Research for Evolutional Science and Technology, Japan Science and Technology Co.	125,000,000	122,000,000
General Resear	1,850,000	1,850,000
Summation	126,850,000	123,850,000

5.4 Combustion Synthesis Research Team

Yoshinari Kaieda (Team Leader)

5.4.1 Staff

Yoshinari Kaieda (Team Leader), Nobutaka Oguro

5.4.2 Research work

Following the 5th Long-term Plan, the study on combustion synthesis has been promoted by the Combustion Synthesis Research Team. Fundamental study to reveal the reactions in combustion synthesis is carried out. The propagation of the reaction front and the synthesis process of the materials synthesized through the reaction are also studied. Investigation by thermal analysis with rising temperature, at a constant speed and/or in alternating speed, is carried out to reveal the conditions for the initiation, propagation and synthesis of the reaction. The influence of pressure and convection on the phenomena in the reaction process of the system containing gaseous phase or liquid phase is studied using a high gaseous pressure apparatus.

The selection of the combination of elements, which is the focused of the present study, will be investigated. The system of the combination that might exhibit the effect of convection and pressure during the reaction and synthesis process is selected, considering the system that performs the effect of the liquid and gaseous phase. The system of elements is selected, in which safety during the experiment is assured.

The processes, including high frequency induction vacuum melting and casting, conventionally produce most intermetallic compounds. It is difficult to control accurately the chemical components of intermetallic compounds produced by the conventional process. The industrial process, including a combustion synthesis method, which is a newly developed manufacturing process in this institute, produces a homogeneous intermetallic compound. The chemical components and impurities in intermetallic compounds produced industrially by the process are revealed. These properties are vitally important when the combustion synthesis method is applied to an industrial mass production process for producing intermetallic compounds.

<Research theme>

- (1) Study on Combustion Synthesis (General Research: April 1996 to March 1999, Yoshinari Kaieda)

5.4.3 Research Products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) Y.Kaieda, M.Otaguchi, N.Oguro: Study on Solid State Chemical Reaction, its Propagation and Materials Synthesis, NRIM Reports, **19**, 103(1997). (in Japanese)
- (2) Y.Kaieda: Microstructure of Titanium by Nitriding Reaction Under Microgravity, J. Jpn. Soc. Powder and Powder Met., **46**, 9(1999). (in Japanese)
- (3) Y.Kaieda: Morphology Change in Optical Microscopic Microstructure of Titanium by Nitriding Reaction and Combustion Synthesis Under Normal and Microgravity, J. of Materials Synthesis and Processing, **7**, 67(1999).

Patent Application

- (1) Production Method of Shape Memory Materials, Y.Kaieda, May 19, 1997, H9-128488.
- (2) Making of Ceramic Tube, Y.Kaieda, January 22, 1998, US.Patent 09/010,791.
- (3) Production Method of Intermetallic Compounds, Y.Kaieda, July 8, 1998, H10-193478.
- (4) Production Method of Intermetallic Compounds, Y.Kaieda, July 8, 1998, H10-193479

5.4.4 Researchers in the Combustion Synthesis Research Team

Age and research fields of researchers in the Combustion Synthesis Research Team

(March 31, 1999)

Age of Researchers	Number of Researchers	Metallurgy	Mechanical Engineering
60 ~	0	0	0
50 ~ 60	2	1	1
40 ~ 49	0	0	0
30 ~ 39	0	0	0
20 ~ 29	0	0	0
Summation	2	1	1

5.4.4 Research Budgets in the Combustion Synthesis Research Team

Research budgets in the Combustion Synthesis Research Team (yen)

Kinds of budgets	1997	1998
General Research	1,260,000	1,260,000
Summation	1,260,000	1,260,000

Chapter 6 Research for Fundamental Materials Information

Hirosada Irie (Director) and Toshiaki Kodama (Special Research Officer)

In the NRIM a number of information oriented research projects have been conducted independently at multiple divisions and sections. Some work has placed stress on data generation and others on the data collection from external resources or software construction.

The datasheet projects, categorized as data production work, have been carried out at Strength and Life Evaluation Research Station, and have the longest history among data related works in the NRIM. The creep Data Sheet Project was started in 1966 and the Fatigue Datasheet Project was started 1975. With the Datasheet Projects the materials for evaluation have been selected from commercial alloys to meet the needs of industries. In the High Resolution Beam Research Station and the Extreme High Vacuum Research Station, a spectrum data study has been carried out for surface analyses of AES and XPS. The database has been compiled in conjunction with the Surface Analysis Society of Japan (SASJ), and is open through Internet. In the Computational Materials Science Division, data collection work for superconducting materials is in progress, where the data retrieved from literature are compiled in a numerical database. An Internet-oriented database called Data-Free-Way (DFW) has been constructed in the Second Research Group. The DFW program, launched in 1990, is a joint project of the NRIM, the Japan Atomic Energy Research Institute (JAERI), the Japan Nuclear Cycle Development Institute (JNC) and the Japan Science and Technology Cooperation (JST). The DFW was designed basically for application to nuclear materials but it can also be applied to materials designing. In the Ecomaterial Research Team the main concern is to provide a web-site system with an interactive user interface for the Life-Cycle Assessment (LCA) of metals and materials. A new database project has started on the corrosion of low-alloy steels in the team of the Special Research Officer with the aims of systematic data generation of atmospheric corrosion and the construction of a database accessible on the web-site according to the 5th Long-term Plan.

Individual datasheets and databases have already gained reputation in particular fields, although these research projects have been conducted seemingly with no interaction among them. In the future it is expected that these research projects be united under a plan of Materials Information Center to meet the increasing public demands for materials data.

6.1 Creep Data Sheets

Fujio Abe (Unit Leader)

6.1.1 Staff

Hirosada Irie (Director), Fujio Abe (Unit Leader), Eiji Baba, Masaru Shimizu, Hideo Tanaka, Kenji Yokokawa, Kiyoshi Kubo, Masaharu Murata, Osamu Kanemaru, Toshio Ohba, Masaaki Tabuchi, Hideaki Kushima, Hideko Miyazaki, Seiichi Muneki, Kazuhiro Kimura, Masayoshi Yamazaki, Takashi Watanabe

6.1.2 Research work

High-temperature components used under high-temperature creep conditions are designed on the basis of a 100,000 h creep rupture strength in Japan. On the other hand, the establishment of reliable methods for determining the remaining life has earnestly been wished for high-temperature components being operated for a long duration. Therefore, an understanding of long-term creep and creep rupture behavior is important for the safe design and reliable life assessment of structural components of high-temperature plants. The National Research Institute for Metals (NRIM) has been conducting a Creep Data Sheet Project since 1966, in order to obtain the 100,000 h-creep rupture strength for many kinds of heat resistant steels and alloys which were produced in Japan.

The materials being now examined in the Creep Data Sheet Project are listed in Table 1; 47 kinds of materials in total. The materials consist of new steels, such as 9Cr-0.5Mo-1.8W-VNb steel (ASME SA335 P92 and SA213 T92) and 11Cr-0.4Mo-2W-CuVNb steel (ASME SA335 P122 and SA213 T122), which have been recently developed by Japanese steel-making companies for ultra-supercritical (USC) power plants, as well as conventional heat resistant steels and alloys, and including welded joints. Most materials are subjected to several to twenty heat treatments. The creep specimens were sampled at random from commercial stocks.

The fact data of long-term creep and creep rupture tests have been published as a series of NRIM Creep Data Sheets. Up to now we have published 114 sheets, in sequence of the first, second and third editions for the individual materials. The first edition contains the creep rupture data up to about 10,000 h. The second edition is published when the creep rupture data up to about 30,000 to 50,000 h, and it also includes the data of the first edition. The third edition contains the data up to 100,000 h for the materials, except for the superalloys, and it also includes the data of the first and second editions. For the superalloys, the third edition contains the data

for up to about 70,000 h. The third edition is the final edition and contains a full set of data; creep rupture data, minimum creep rates, short-time tensile data, evaluation of short-time tensile strength and long-term creep rupture strength by curvilinear regression analysis and optical micrographs. The Creep Data Sheets for stress relaxation have been published since 1996; for the 18Cr-12Ni-Mo steel (SUS316) in 1996, the bolting materials in 1997 and the Fe-21Cr-32Ni-Ti-Al alloy in 1999.

In 1999, we published a first edition of the Microstructure Data Sheets named as "Metallographic Atlas of Long-Term Creep Materials" for the 18Cr-8Ni steel (SUS304), as another series of the NRIM Creep Data Sheets. The Metallographic Atlas contains not only series micrographs showing the microstructural evolution during creep for up to 100,000 h but also related data such as time-temperature-precipitation diagrams, histograms describing the distributions of precipitates and creep-voids, and creep damage parameters, using specimens tested in the Creep Data Sheet Project. The Creep Data Sheets and the Metallographic Atlas have been distributed to about 230 sites in Japan, and also about 200 sites in foreign countries.

In addition to the Creep Data Sheets and the Metallographic Atlas, we have published a technical document, which describes comprehensively the overall scheme of the project, materials details, testing machines and testing procedures, calibration of thermocouples, etching and microstructural examination, evaluation and analysis of creep data and publication procedures of the Creep Data Sheets, in 1996 as shown in Table 2.

The long-term creep rupture tests over 100,000 h have already been completed on 247 specimens and are now still continuing on 130 further specimens, while they have been interrupted for 147 specimen. The long-term creep rupture tests over 200,000 h have already been completed on the 6 specimens and are now still continuing on 19 specimens.

In parallel with the testing and publishing program in the Creep Data Sheet Project, we have made research for understanding long-term creep and the creep rupture properties of the materials. It is emphasized that long-term creep and creep rupture behavior is quite different from the short-term one, resulting from complicated microstructural evolution during creep. The main results in the recent research are summarized as follows.

- (1) The creep rupture strength of Cr-Mo ferritic heat resistant steels shows a tendency of convergence to a common level in the long term. The ferrite matrix containing small amounts of dissolved carbon and Mo is responsible for the fundamental creep

strength, named 'Inherent Creep Strength', of the steels after completion of their microstructural evolution.

- (2) The heat-to-heat variation in the long-term creep rupture strength of the Cr-Mo ferritic heat resistant steels is caused by the difference in microstructural stability during creep and can be expressed by the rate constant α of the constitutional equations based on a modified θ -projection concept.
- (3) During creep, fine TiC and NbC precipitate in the matrix of the 18Cr-10Ni-Ti (SUS321H) and 18Cr-12Ni-Nb (SUS347H) austenitic heat resistant steels, respectively, accompanying the re-dissolution of previously precipitated $M_{23}C_6$ at the grain boundaries. This accelerates grain boundary sliding and surface cracking. The formation and propagation of surface cracks result from grain boundary displacement on the specimen surface. The addition of small amounts of boron stabilizes the fine $M_{23}C_6$ along the grain boundaries and suppresses grain boundary sliding, increasing creep rupture strength.

We are now planning a new publication of Creep Strain Data Sheets in addition to the present Creep Data Sheets and the Metallographic Atlas. Although the most common and direct format is to describe creep strain data as a function of time, the description of creep curves using appropriate constitutional equations will be needed. Combination of the Creep Data Sheets, the Metallographic Atlas and the Creep Strain Data Sheets will provide more reliable creep life prediction.

6.1.3 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

<Papers>

- (1) F. Abe : Evolution of Microstructure and Acceleration of Creep Rate in Tempered Martensitic 9Cr-W Steels, Mater. Sci. and Engi., A234-236, 1045 (1997)
- (2) H. Tanaka, M. Murata, F. Abe, K. Yagi : The Effect of Carbide Distributions on Long-term Creep Rupture Strength of SUS321H and SUS347H Stainless Steels, Mater. Sci. and Engi. A234-236, 1049 (1997)
- (3) K. Kimura, H. Kushima, F. Abe, K. Yagi : Inherent Creep Strength and Long Term Creep Strength Properties of Ferritic Steels, Mater. Sci. and Engi., A234-236, 1079 (1997)
- (4) H. Tanaka, M. Murata, F. Abe, K. Yagi : Effect of Grain Boundary Precipitates on Long-Term Creep Rupture Properties for SUS 347H steel, Testu-to-Hagane, 83,

72 (1997)

- (5) T. Ohba, O. Kanemaru, K. Yagi, C. Tanaka : Long-Term Stress Relaxation Properties of NCF 800H alloy, J. Society Mater. Sci., **46**,19 (1997) (in Japanese)
- (6) M. Murata, F. Abe, K. Yagi : Development of Monitoring System for Creep Tests, Report of the 123 Committee on Heat-Resisting Materials and Alloys, **38**, 87 (1997) (in Japanese)
- (7) O. Kanemaru, M. Shimizu, T. Ohba, H. Miyazaki, F. Abe, K. Yagi : Remaining Life Prediction by The Iso-stress Method of Boiler Tubes after Prolonged Service, Mater. Sci. Res. Inter., **3**, 31 (1997)
- (8) T. Ohba, O. Kanemaru, K. Yagi, C. Tanaka : Long-Term Stress Relaxation Properties and Microstructural Changes of NCF 800H, Mater. Sci. Res. Inter. **3**, 10(1997)
- (9) K. Kimura, H. Kushima, F. Abe, K. Yagi : Inherent Creep Strength and A New Approach to The Evaluation of Long-Term Creep Strength Properties, 7th Inter. Conf. on Creep and Fracture of Engineering Materials and Structures,89,Irvine, USA, August 10-15, 1997
- (10) K. Yagi, F. Abe : Long-term Creep and Rupture Properties and Microstructural Changes of Heat Resisting Steels, 7th Inter. Conf. on Creep and Fracture of Engineering Materials and Structures, 427, Irvine, USA, August 10-15, 1997
- (11) K. Kimura, H. Kushima, F. Abe, K. Yagi, H. Irie : Assessment of Creep Strength Properties of 9 to 12% Cr Steels from a Viewpoint of Inherent Creep Strength, 4th Inter. Charles Parsons Turbine Conf.,257, Newcastle, UK, November 4-6, 1997
- (12) F. Abe, K. Yagi : Evaluation of Long-Term Creep and Rupture Properties of Heat Resisting Steels, 4th Inter. Charles Parsons Turbine Conf., 750, Newcastle, UK, November 4-6,1997
- (13) K. Yagi, F. Abe : Microstructure Changes and Creep Life Prediction of Heat Resisting Steels, Report of the 123 Committee on Heat-Resisting Materials and Alloys, **38**, 197 (1997) (in Japanese)
- (14) F. Abe : The Longest Creep Tests in the World, Materia Japan, **37**,7(1998) (in Japanese)
- (15) F. Abe : Kinetics of Carbide Precipitation during Creep and its Effect on Creep Rate of 10Cr-30Mn Austenitic Steels, Mater. Trans., JIM, **39**,211(1998)
- (16) H. Tanaka, E. Nishikawa, F. Abe, K. Yagi, T. Sugita : Non-Destructive Microstructure Examination of Boiler Tubes using Penning Discharge Micro-sputtering Technique, Z. Metallkde, **89**,375 (1998)
- (17) K. Kimura, H. Kushima, F. Abe, K. Yagi : Evaluation of Creep Strength Property

from a Viewpoint of Inherent Creep Strength for Ferritic Heat Resistant Steels, Second Microstructural Stability of Creep Resistant Alloys for High Temperature Plant Applications, 185, Sheffield, UK, March 24-26, 1998

- (18) K. Kimura, F. Abe, H. Irie, K. Yagi : Long-Term Creep and Creep rupture Properties for Advanced Power Engineering, 6th Liege Conf. on Materials for Advanced Power Engineering, 627, Liege, Belgium, October 5-7(1998)
- (19) H. Kushima, K. Kimura, F. Abe, K. Yagi, H. Irie, K. Maruyama : Effect of Microstructure on the Long-term Creep Deformation Properties of 2.25Cr-1Mo Steels, Report of the 123 Committee on Heat-Resisting Materials and Alloys, 39, 61 (1998) (in Japanese)
- (20) K. Yagi, F. Abe : Long-Term Creep and Rupture Properties of Heat Resisting Steels and Alloys, Acta Metallurgica Sinica, 11, 391(1998)
- (21) H. Kushima, K. Kimura, F. Abe, K. Yagi, H. Irie, K. Maruyama : Evaluation of Long-Term Creep Strength Properties of Cr-Mo Type Heat Resistant Steels based on Creep Deformation Analysis, Report of the 123 Committee on Heat-Resisting Materials and Alloys, 40, 1(1999) (in Japanese)
- (22) F. Abe : Creep test method in *Handbook of Metals*, JIM, in press (in Japanese)

<Creep Data Sheets> April 1, 1997 to March 31, 1999

- (23) Creep Data Sheet No.19B, 9Cr-1Mo Steel (JIS STBA 26), NRIM, 30 September(1997)
- (24) Creep Data Sheet No.45, 18Cr-12Ni-Mo-middle N-low C Steel (JIS SUS 316-HP), NRIM, 30 September(1997)
- (25) Creep Data Sheet No.10B, 12Cr-1Mo-1W-0.3V Steel (JIS SUH 616-B), NRIM, 31 March(1998)
- (26) Creep Data Sheet No.26B, Fe-based 21Cr-32Ni-Ti-Al Alloy (JIS NCF 800H TB), NRIM, 30 September(1998)
- (27) Creep Data Sheet No.46, 9Cr-2Mo Steel(STBA27, SFVAF27), NRIM, 30 September(1998)
- (28) Creep Data Sheet No.41A, Ni-based 15.5Cr-8Fe Superalloy(JIS NCF 600), NRIM, 31 March(1999)
- (29) Creep Data Sheet No.47, Fe-based 21Cr-32Ni-Ti-Al Alloy, Stress Relaxation(JIS NCF 800H-B), NRIM, 31 March(1999)
- (30) Metallographic Atlas of Long-Term Crept Materials No.M-1, 18Cr-8Ni Steel (JIS SUS 304H TB), NRIM, 31 March(1999)

Oral presentation: 25

- (1) International Conference : 8
- (2) Domestic Conference : 17

Award

- (1) Nishiyama Memorial Prize, The Iron and Steels Institute of Japan,
April 1998, Fujio Abe
- (2) Technical Skill Prize, The Iron and Steels Institute of Japan,
March 1999, Eiji Baba

6.1.4 Research Budgets

- 32,300,000 yen (1997)
- 29,384,000 yen (1998)

Table 1 Publication of Creep Data Sheets

	CDS No.	Materials	Year of Publication		
			First Edition	Second Edition	Third Edition
			< 10 ⁴ h	< 5 x 10 ⁴ h	10 ⁵ h
Carbon Steels	7	0.2C (tube)	1973	1979	1992
	17	0.3C (plate)	1980	1981	1994
	40	0.2C-1.3Mn (tube)	1989	-	-
Low Alloy Ferritic Steels	8	0.5Mo (tube)	1973	1979	1991
	25	High strength steel (plate)	1978	1983	1994
	18	1.3Mn-0.5Mo-0.5Ni (plate)	1985	1981	1987
	20	0.5Cr-0.5Mo (tube)	1976	1981	1994
	1	1Cr-0.5Mo (tube)	1972	1976	1996
	35	1Cr-0.5Mo (plate)	1985	1990	-
	9	1Cr-1Mo-0.25V (forging)	1973	1979	1990
	31	1Cr-1Mo-0.25V (cast)	1980	1984	1994
	2	1.25Cr-0.5Mo-Si (tube)	1972	1976	
	21	1.25Cr-0.5Mo-Si (plate)	1976	1981	1994
	3	2.25Cr-1Mo (tube)	1972	1976	1986
	11	2.25Cr-1Mo (plate)	1974	1980	1997
	36	2.25Cr-1Mo (plate)	1985	1991	-
	12	5Cr-0.5Mo (tube)	1974	1980	1992
High-Cr Ferritic and Austenitic Steels		9Cr-0.5Mo-1.8W-V-Nb (tube,	-	-	-
	19	9Cr-1Mo (tube)	1985	1981	1997
	43	Mod.9Cr-1Mo (tube & plate)	1996	-	-
	46	9Cr-2Mo (tube & plate)	1998	-	-
	13	12Cr (bar)	1974	1980	1994
		11Cr-0.4Mo-2W-Cu-V-Nb	-	-	-
	10	12Cr-1Mo-1W-0.3V (bar)	1973	1979	1998
	4	18Cr-8Ni (tube)	1972	1978	1986
	32	18Cr-8Ni (plate & welded	1982	1995	-
	6	18Cr-12Ni-Mo (tube)	1972	1978	-
	14	18Cr-12Ni-Mo (plate)	1974	1982	1988
	15	18Cr-12Ni-Mo (bar)	1974	1982	1988
	45	18Cr-12Ni-Mo-N (plate)	1997	-	-
	5	18Cr-10Ni-Ti (tube)	1972	1978	1987
	28	18Cr-12Ni-Nb (tube)	1979	1983	-
	26	Fe-21Cr-32Ni-Ti-Al (tube)	1978	1983	1998

CDS : Creep Data Sheet

Publication of Creep Data Sheets (Continued)

	CDS No.	Materials	Year of Publication		
			First Edition	Second Edition	Third Edition
			< 10 ⁴ h	< 5 x 10 ⁴ h	10 ⁵ h
	27	Fe-21Cr-32Ni-Ti-Al (plate)	1978	1983	-
	37	25Cr-12Ni-0.4C (cast)	1985	1992	-
	16	25Cr-20Ni-0.4C (cast)	1974	1980	1990
	38	25Cr-35Ni-0.4C (cast & tube)	1985	1991	-
Superalloys	22	Fe-15Cr-26Ni-Mo-Ti-V (disk)	1972	1982	1993
	23	Fe-20Cr-20Ni-20Co-W-Mo-(Nb+Ta)	1977	1982	1989
	33	Fe-21Cr-20Ni-20Co-Mo-W-(Nb+Ta)-N	1984	-	-
	41	Ni-15.5Cr-8Fe (bar, plate &	1991	1999	-
	39	Ni-15.5Cr-Ti-Al-Nb-7Fe (bar)	1988	1992	-
	29	Ni-13Cr-4.5Mo-Ti-Al-(Nb+Ta)-Zr-B	1979	1984	1990
	34	Ni-19Cr-18Co-4Mo-3Ti-3Al-B	1984	1989	1993
	24	Ni-15Cr-28Co-Mo-Ti-Al (bar)	1977	1992	1989
		Ni-16Cr-8.5Co-Mo-W-Ti-	-	-	-
Stress Relaxation	30	Co-25Cr-10Ni-7.5W-B (cast)	1979	1984	1988
	42	18Cr-12Ni-Mo (plate)	1996	-	-
	44	Bolting materials	1997	-	-
	47	Fe-21Cr-32Ni- (bar)	1999	-	-
		2.25Cr-1Mo (plate)	-	-	-
		18Cr-8Ni (plate)	-	-	-
Metallographic Atlas		1Cr-1Mo-0.25V (forging)	-	-	-
	M-1	18Cr-8Ni (tube)	1999	-	-
		18Cr-12Ni-Mo (tube)	-	-	-
		18Cr-10Ni-Ti (tube)	-	-	-
Creep Strain Data Sheets		18Cr-12Ni-Nb (tube)	-	-	-
		2.25Cr-1Mo (tube & plate)	-	-	-
		18Cr-8Ni (tube & plate)	-	-	-
		18Cr-12Ni-Mo (plate)	-	-	-
		18Cr-10Ni-Ti (plate)	-	-	-
		Fe-21Cr-32Ni-Ti-Al (bar &	-	-	-

Table 2 Publication of the NIRM Material Strength Data Sheet Technical Document

	No. 10	Testing Plan and Testing Procedures for NIRM Creep Data Sheet Project	1996	-	-
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6.2 Fatigue Data Sheets

Saburo Matuoka (Unit Leader)

6.2.1 Staff

Hirosada Irie (Director), Akihiko Ohta (Unit Leader), Noyuki Suzuki, Yoshio Maeda, Saburo Matsuoka (Unit Leader), Takayuki Abe, Etuo Takeuti, Hisashi Hirukawa, Nobuo Nagashima, Kohji Yamaguchi (Unit Leader), Kazuo Kobayshi, Megumi Kimura, Masuo Shimodaira

6.2.2 Research Work

As it is known that more than 80% of service failures occur due to fatigue, the fatigue strength of materials must be considered in designing machines and structures. The NRIM has been conducting the Fatigue Data Sheet Project since 1975 to provide standard reference data on the fundamental fatigue properties of typical materials used in Japan for machines and structures. The project has been directed and guided by advisory and technical advisory committees, which are composed of leading specialists from related academic and industrial societies in Japan as in case of the Creep Data Sheet.

Eighty-three Fatigue Data Sheets have been published, as shown in Table 1. Though the initial program was started for various steels, aluminum alloys were also employed as the first nonferrous materials. The Fatigue Data Sheets are classified into three subjects, entitled, "Basic Fatigue Properties of Materials for Machine Structural Use at Room Temperature", "Fatigue Properties of Welded Joints" and "Fatigue Properties at Elevated Temperatures". In the first subject, low- and high-cycle fatigue tests were conducted under rotating bending, reversed torsion and uniaxial loading on standard smooth specimens. Several heats for each material were sampled from Japanese manufacturers to examine the scatter of fatigue properties. In the second subject, effects of specimen size, welding procedure, stress ratio and testing temperature on high-cycle fatigue and fatigue crack propagation properties of welded joints were studied under uniaxial loading on large-scale specimens. These studies revealed that the tensile residual stress was the dominating parameter for fatigue failures of welded structures. In the third subject, low- and high-cycle fatigue tests were carried out under rotating bending and uniaxial loading on standard specimens at elevated temperatures between 673 and 1073K. In particular, low-cycle fatigue focused on creep-fatigue interaction by using various strain waveforms.

The Fatigue Data Sheets have been exchanged worldwide with scientific and

technical organizations, and have contributed to the safe and effective use of engineering materials. A typical example is the new standard "Fatigue Testing of Welded Joints-ISO Standard" established by the International Institute of Welding (IIW). The Fatigue Data Sheets of Welded Joints were adopted as the fundamental data the standard.

Sixteen Fatigue Data Sheet Technical Documents have been published, as shown in Table 2. In the Documents, selected data from the related Data Sheets are analyzed for fitting theoretical models and additional experiments are performed to verify the theories. As the results, the Documents offer guides for understanding the relation between fatigue strength and other mechanical properties and for applying these materials to industrial machines and structures. The Documents also provide useful information in failure analysis. For example, the NRIM deduced with help of the Document that a sheath thermometer, made of Type 304 stainless steel, was fractured by high-cycle fatigue in Monju's sodium leak incident in 1997.

Recently, the life extension of machines and structures has been desired from the ecological and economical points of view. In addition, much attention has been paid to giga-cycle fatigue. Fatigue limit is usually determined as the strength at 10^7 cycles. However, it has been pointed out that when more than 10^7 cycles loading is applied to high-strength steels, fatigue crack initiates from an inclusion even below the fatigue limit. The Long Term Fatigue Data Sheet Project has been pursued at NRIM according to the 5th Long-term Plan since 1997 in order to evaluate long-term fatigue strength. In the new project, the following subjects are studied; (1) high-cycle fatigue data up to 10^{10} cycles for high strength steels at room temperature, (2) low-cycle fatigue data up to 10^7 cycles at elevated temperatures, (3) fatigue data for titanium alloys, and (4) fatigue data of welded joints induced large tensile residual stress.

6.2.3 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) H. Hirukawa, S. Matsuoka, E. Takeuchi, T. Omura, K. Yamaguchi, K. Tsuzaki: HighResistance of Fatigue Crack Growth for Austenitic Stainless Steel Containing Nitrogen, Trans. Japan Society Mechan. Engi. 65A, 1343(1999) (in Japanese)
- (2) N.Nagashima, K.Miyahara, S.Matsuoka: Hardness Measurement of Small Cementite Particles using an AFM Ultramicro Hardness Tester, Trans. Japan Society Mechan. Engi. , 64A, 536(1998) (in Japanese)
- (3) T.Abe, T.Ohmura, S.Matsuoka: Surface and Internal Fatigue Fracture Mechanisms for High Strength Steels, submitted to Trans. Japan Society of

Mechan. Engi. (in Japanese)

- (4) E. Takeuchi, H. Hirukawa, S. Matsuoka : Arrest Function in Elevated-temperature Fatigue Crack Growth of High Alloy with a Small Addition of Zirconium, Trans. Japan Society of Mechan. Engi., **65A**, 148(1999) (in Japanese)
- (5) K. Ymaguti, K. Kobayashi, S. Nishijima, K. Kanazawa: Elevated Temperature Fatigue Properties of Various Steels, Kensa-gijutu, **5**, 1(1997) (in Japanese)
- (6) K. Kobayashi, K. Ymaguti: The Effect of Compressive Strain Hold on The Low Cycle Life of Various Materials at Elevated Temperature, J. High Pressure Institute Japan, **36**, 27(1998) (in Japanese)
- (7) K. Kobayashi, K. Yamaguti, S. Katoh, S. Nishijima, T. Fujioka, T. Nakazawa, H. Koto, S. Date: High Temperature Fatigue Properties of the 316FR Steel, Trans. Japan Society of Mechan. Engi., **64A**, 545(1998) (in Japanese)
- (8) M. Kimura, K. Yamaguti, S. Matsuoka: Fractography of Fatigue Surface for Type 304 stainless Steels, Report of the 123 Committee On Heat-Resisting Materials and Alloys, **39**(1998) (in Japanese)
- (9) M. Shimodaira, K. Yamaguti: Fatigue Properties of TiAl Intermetallic Compounds and Dependence of the Microstructure, J. Soc. Mater. Sci. Japan, **48**, 166(1999) (in Japanese)
- (10) Ohta, N. Suzuki, Y. Maeda: Effect of Residual Stress on Fatigue of Weldment. Pro. Inter. Conf. Performance of Dynamically Loaded Welded Structures, WRC, **108**(1997)
- (11) A. Ohta, N. Suzuki, Y. Maeda: Unique Fatigue Threshold and Crack Growth Properties of Welded Joints In Tensile Residual Stress Field, Inter. J. Fatigue, **19**, 303(1997)
- (12) N. Suzuki, A. Ohta, Y. Maeda: Elevated Temperature Fatigue Propagation Properties of Butt Welded Joints, Trans. Japan Institute of Welding, **15**, 365(1997) (in Japanese)
- (13) A. Ohta, Y. Maeda, N. Suzuki: Fatigue Strength of Transverse Butt Welded Joints under Random Loading, Trans. Japan Institute of Welding, **15**, 122(1997) (in Japanese)

• **Oral Presentation: 15**

- (1) International Conference: 1
- (2) Domestic Conference: 14

6.2.4 Research Budgets

1997: 24,303,000 Yen

1998: 24,303,000 Yen

Table 1 Publication of Fatigue Data Sheets

(1) Basic Fatigue Property at Room Temperature

(a) High-Cycle Fatigue

Specification	Nominal compositions(%)	FDS No.
(Carbon and Low Alloy Steel)		
JIS S25C	0.25C	1
JIS S35C	0.35C	2
JIS S45C	0.45C	3
JIS S55C	0.55C	4
JIS SMn438	0.38C-1.5Mn	16
JIS SMn443	0.43C-1.5Mn	17
JIS SCr440	0.40C-1Cr	8
JIS SCM435	0.35C-1Cr-0.2Mo	9
JIS SCM440	0.40C-1Cr-0.2Mo	10
JIS SNC631	0.31C-2.7Ni-0.8Cr	24
JIS SNCM439	0.39C-1.8Ni-0.8Cr-0.2Mo	25
JIS SNCM447	0.47C-1.8Ni-0.8Cr-0.2Mo	26
(Stainless Steel)		
JIS SUS403	12Cr	30
JIS SUS430	17Cr	29
JIS SUS304	18Cr-8Ni	33
(Carburizing Steel)		
JIS SCr420	0.20C-1Cr	37
JIS SCM420	0.20C-1Cr-0.2Mo	43
JIS SNCM220	0.20C-0.5Ni-0.5Cr-0.2Mo	50
JIS SNCM420	0.20C-1.8Ni-0.5Cr-0.2Mo	51
(Spring Steel)		
JIS SUP7	2.0Si-0.8Mn	59
JIS SUP9A	0.8Mn-0.8Cr	60
JIS SUP12	1.4Si-0.7Cr	63
(Tool Steel)		
JIS SKD61	0.36C-5Cr-1.25Mo-1V	69
JIS SKD11	1.5C-12Cr-1Mo-0.35V	73

(b) Low-Cycle Fatigue

Specification	Nominal composition (%)	FDS No.
(Carbon and Low Alloy Steel)		
JIS S25C	0.25C	38
JIS S35C	0.35C	39
JIS S45C	0.45C	44
JIS SCr440	0.40C-1Cr	45
JIS SCM435	0.35C-1Cr-0.2Mo	52
JIS SNCM439	0.39C-1.8Ni-0.8Cr-0.2Mo	56
(Aluminum Alloy)		
JIS A5083P-O	Al-4.5Mg-0.6Mn	61
JIS A7NOIS-T5	Al-4.6Zn-1.2Mg	70
JIS A7NOIP-T6	Al-4.5Zn1.5Mg	74

(2) Fatigue Property of Welded Joints

(a) High-Cycle Fatigue

Specification	Nominal composition (%)	FDS No.
Steel		
(Effect of Specimen Size)		
JIS SM490B	0.15C-1.4Mn-0.35Si	5
JIS SM570Q	0.13C-1.3Mn-0.27Si	11
HT80	0.12C-0.8Ni-Cr-Mo-V	12
JIS SM490B	0.15C-1.4Mn-0.35Si	13
JIS SM490B	0.15C-1.4Mn-0.35Si	18
(Effect of Welding Procedure)		
JIS SM490B	0.15C-1.4Mn-0.35Si	27
HT80	0.12C-0.8Ni-Cr-Mo-V	19
JIS SM490B	0.15C-1.4Mn-0.35Si	20
(Effect of Stress Ratio)		
JIS SB410	0.16C-0.80Mn	34
JIS SPV490	0.13C-1.2Mn-0.28Si	40
JIS SUS304-HP	18Cr-8Ni	53
(Effect of Temperature)		
JIS SB450	0.25C-0.85Mn	79
JIS SCMV2-2NT	1Cr-0.5Mo	75
Aluminum alloy		
JIS A5083P-O	Al-4.5Mg-0.6Mn	64
JIS A6N01S-T5	Al-0.6Mg-0.65Si	80
JIS A7N01S-T5	Al-4.6Zn-1.2Mg	71
JIS A7N01P-T6	Al-4.5Zn-1.5Mg	76

(b) Crack Propagation

Specification	Nominal composition (%)	FDS No.
(Effect of Welding Procedure)		
JIS SM490B	0.15C-1.4Mn-0.35Si	21
HT80	0.12C-0.8Ni-Cr-Mo-V	31
(Effect of Stress Ratio)		
JIS SB410	0.16C-0.80Mn	41
JIS SPV490	0.13C-1.2Mn-0.28Si	46
JIS SUS304-HP	18Cr-8Ni	54
(Effect of Temperature)		
JIS SB450	0.25C-0.85Mn	82
JIS SCMV2-2NT	1Cr-0.5Mo	81

(c) High- and Low-Cycle Fatigue of Weld and HAZ Material

Specification	Nominal composition (%)	FDS No.
JIS SB410	0.16C-0.80Mn	57
JIS SPV490	0.13C-1.2Mn-0.28Si	47
JIS SUS304-HP	18Cr-8Ni	65

(3) Fatigue Property at Elevated Temperature

(a) High-Cycle Fatigue

Specification	Nominal composition (%)	FDS No.
(Effect of Stress Concentration Factor)		
JIS S45C	0.45C	14
JIS SCM435	0.35C-1Cr-0.2Mo	23
ASTM A470-8	1Cr-1Mo-0.25V	55
JIS SCMV4	2.25Cr-Mo	48
JIS SUS403-B	12Cr	6
JIS SUH616-B	12Cr-1Mo-1W-0.3V	35
JIS SUS304-HP	18Cr-8Ni	42
JIS SUS316-HP	18Cr-12Ni-2Mo	15
JIS NCF800H-B	21Cr-32Ni-Ti-Al	32
(Effect of Frequency)		
JIS SB450	0.25C-0.85Mn	66
JIS SCMV2-2NT	1Cr-0.5Mo	72

(b) Low-Cycle Fatigue

Specification	Nominal composition (%)	FDS No.
JIS SB480	0.27C-0.78Mn	22
JIS SCMV3	1.25Cr-0.5Mo	28
JIS SCMV4	2.25Cr-1Mo	7
JIS SUS316-HP	18Cr-12Ni-2Mo	15
JIS SB450	0.25C-0.85Mn	67
JIS SCMV2-2NT	1Cr-0.5Mo	77

(c) Time Dependent Low-Cycle Fatigue

Specification	Nominal composition (%)	FDS No.
ASTM A470-8	1Cr-1Mo-0.25V	58
JIS SCMV4	2.25Cr-1Mo	62
ASTM A387G91	9Cr-1Mo	78
JIS SUH616-B	12Cr-1Mo-1W-0.3V	68
JIS SUS304-HP	18Cr-8Ni	49
JIS NCF800H-B	21Cr-32Ni-Ti-Al	36

Table 2 Fatigue Data Sheet Technical Documents

No.	Subjects	Issued
1	Fundamental Fatigue Property for Carbon, Cr and Cr-Mo Steels	1981
2	Fatigue Strength for Welded Joints of High Strength Steels	1983
3	Crack Growth in Arc-Welded Butt-Joints of High Strength Steels	1984
4	Strain Rate Effect in Low Cycle Fatigue of Alloy Steels at High Temps.	1985
5	Fundamental Fatigue Property of JIS Steels for Machine Structures	1989
6	High Cycle Fatigue of Alloy Steels at Elevated Temperatures	1990
7	High Cycle Fatigue Property of Carburized Steels	1992
8	Fatigue Crack Propagation for Welded Joints of Structural Steels	1995
9	High Cycle Fatigue Property of Hard Steels	1995
11	Time/Temp Effect in High Cycle Fatigue of Steels at Intermediate Temps.	1996
12	Time-Dependent High Temperature Fatigue of Steels and Alloys	1996
13	Fatigue Property of Aluminum Alloys for Welded Structures	1996
14	Intermediate Temperature Fatigue of Steels for Welded Structures	1997
15	Low/High Cycle Fatigue Properties of Steels and Aluminum Alloys	1997
16	Intermediate Temperature Fatigue of Steels for pressure vessels	1997
17	Elastic Moduli of Steels for Machine Structures	1997

6.3 Database for Corrosion for Low-Alloy Steels

Toshiaki Kodama(Special Research Officer)

6.3.1 Staff

Toshiaki Kodama(Special Research Officer), Akira Tahara, Hideki Katayama

6.3.2 Research work

Corrosion is not an intrinsic property of metal itself but is influenced by not only metal properties but also environmental parameters such as temperature, pressure and chemical species, since corrosion is materials degradation caused by the chemical reaction with environment. Only a small number of publicized corrosion database are available although the production of corrosion data has been carried out at many governmental and private institutions. This is as described above due to the complex data structure of corrosion data and is also due to the confidentiality of corrosion data in private sectors. In contrast with the activities of mechanical properties such as creep and fatigue, systematic data production work (data-sheet activity) has not been carried out for corrosion in NRIM. With the onset of the STX21 Project on structural steels we started systematic data collection works in the field of weathering of low alloy steels and constructing NRIM database of corrosion. The basic approach toward the corrosion database is to limit the area of coverage instead of comprehensiveness. In the data generation we use new NRIM facilities as well as those of external organizations for expanding environmental diversity. As for the data source to be included external data are stored after evaluation process.

• Atmospheric Corrosion Data of Low-Alloy Steels

As a part of alloy design program we started atmospheric corrosion tests in 1997 in NRIM campus in Tsukuba and exposure sites of Japan Weathering Test Center (JWTC) at Choshi and Miyako Island. The latter two sites were selected because they have established position as the standard sites of atmospheric exposure in Japan and are managed by a nonprofit organization. Miyako is southernmost exposure site in Japan classified as subtropical climate with very aggressive marine environment. Materials for exposure were selected in such manners as that the generated data may contribute to the elucidation of alloying elements and to alloy design of weathering steels in marine environments. Most of exposed alloys have very simple composition of binary Fe-X systems and are prepared high pure metals.

Since FY1998, we started constructing open database accessible over the Internet. In

the atmospheric corrosion database, the main source of data comes at the moment from Public Works Institute of Ministry of Construction, where nationwide corrosion tests had been carried out for low-alloy steels at selected steel bridges (41 sites in Japan). The database development includes programs to allow viewing and printing corrosion data and picture of corroded surfaces. In the future, the database will be expanded by adding updated data of NRIM exposure program, and by including data from external sources, such as Japan Weathering Test Center (JWTC). The Internet version of the atmospheric database is to be open to limited members in FY 2000 and will be accessible to unlimited members from FY 2001.

6.3.3 Research Products to be Presented

Oral Presentation :2

- (1) International Conference: 0
- (2) Domestic Conference: 2

<Contents>

- (1) M.Yamaoto, A.Tahara, T.Kodama, Development of Query System for Outdoor Exposure Tests over Internet. -Construction of Corrosion Database in NRIM.- , to be presented at 46th Japan Conf. on Materials and Environments, September (1999)
- (2) A.Tahara, M.Yamaoto, T.Kodama Development of Query System for Chemical Potential Diagrams over Internet. -Construction of Corrosion Database in NRIM.-, to be presented at 46th Japan Conf. on Materials and Environments, September (1999)

6.3.4 Research Budgets

This research has been conducted as a part of Study in Corrosion Resistant Steels in the Frontier Research Program of New Structural Steels

The total cost for software development of database is as follows:

1998: 4,756,000 yen

6.4 Assessment & Analysis of Environmental Effects of Steel Production

Komei Halada (Team Leader)

6.4.1 Staff

Kohmei Halada (Team Leader), Kiyoshi Ijima

6.4.2 Research Work

Environmental inventory data of steel alloys, for their quantitative assessment from the viewpoint of global environmental issues, are collected into a database and presented on the World Wide Web with an interactive user interface. Environmental inventory data is the data of emissions, such as the amount of CO₂, which was emitted from mining, metallurgical processes and the transportation of raw materials in processing a unit amount of a product. They are used in LCA (environmental Life-cycle assessment : ISO14040) to assess the ecological performance of materials or products.

Almost all of the stored data in this database were newly estimated ones, with process-models of the production systems of metals based on the investigations of practical metallurgical processes. The data can be assigned not only to the industrial standard code but also to the composition of alloys, the combination of processing, mechanical properties and the types of utilization. These data will be used in the life cycle engineering of products, such as Eco-design, in the same way as property data in the usual designing of products.

The database is composed of three units; 1) CO₂, SO_x, NO_x emissions of preceding alloying element processing and each process of steel making, 2) process flow in the production of steels, 3) the composition of steels in the JIS classification. System I is the primitive data-system which gives the emissions data corresponding to the input data of the composition of subjected steels. Four environmental loads (CO₂, SO_x, NO_x, energy consumption) are obtained for every composition with combinations of 12 alloying elements and 13 steelmaking processes. In System II, the retrieval system of alloys from the purpose of usage is modified. Another table of the purpose of usage and the JIS code (more than 1400) is prepared and related together, corresponding the input with the selection window of the purpose of usage. Product designers can use these data without knowledge of the composition of steels by System II, while System I is useful to employ these data in the field of materials design. Furthermore, System III is developed to design new products. In System III, the alloys are once selected corresponding to the

input data of the properties, such as yield strength or hardness. A designer can select or compare the materials from the environmental data of System III with the selected alloys.

Not only the environmental inventory data of steel alloys, but also that of 13 non-ferrous metals, Cu, Al, Pb, Zn, Sn, Ti, Mg, Ni, Cr, Si, Mn, Co are also arranged and presented in the interactive database on World Wide Web.

Enhancing the object, from the application of materials or product designer to the wide purpose practitioners of LCA, such as consumers, products assemblers and civil or social system designers, the "Database Environmental load of 4000 Social Stocks" is constructed. This database contains not only materials but also any product which is produced in Japan, including agricultural products, foods, electric parts, energy supplies, etc.. The number of products is nearly 4000 and the items of environmental stress are 19, which includes the inventory data of air-emissions, water-emissions, and heavy-metal or chemical consumption.

6.4.3 Database and access

(1) Database for Environmental Assessment of Materials

<http://www.nrim.go.jp:8080/ecomat/ecosheet/ecosheet.htm>

(2) Database of Environmental Load Data of 4000 social stocks

<http://www.nrim.go.jp:8080/ecomat/db/dai.htm>

<database access>

	total access	access from registered member
1996	46,405	90
1997	93,997	859
1998	104,633	1,532
1999(Jan.-Jun.)	62,684	796

6.4.4 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

(1) K.Halada: The Requirements for LCI Data Model as Tool of Life Cycle Engineering, Proc. 3rd Inter. Conf. on Ecobalance, 67-72(1998)

- (2) K.Ijima, K.Halada, K.Yagi: Environment Load Database System of Alloys on the Network, Proc. 3rd Inter. Conf. on Ecomaterials, 333(1997)
- (3) K.Ijima, K.Halada, K.Yagi: Database of Environment Load of 4000 Social Stocks, Proc. 3rd Inter. Conf. on Ecobalance, 531(1998)
- (4) H.Yoshizu, K.Ijima, K.Halada, K.Yagi: Environmental Load Data as Part of Materials Data Base, Proc. 3rd Inter. Conf. on Ecobalance, 575(1998)

• **Oral Presentation: 10**

- (1) International Conference: 5
- (2) Domestic Conference: 5

6.4.5 Budgets

Kind of Budgets	yen	
	1997	1998
General Research	7,500,000	7,500,000
Special Coordination Funds for Promoting Science & Technology	6,383,000	0
Summation	13,883,000	7,500,000

6.5 Data-Free-Way System for Materials Information

Mitsutane Fujita (Subgroup Leader)

6.5.1 Research work

A material information system, which has huge databases and effective computer aided tools, is required for alloy design or the selection of advanced nuclear materials. However there are limitations in storing numerous materials properties in databases and developing a variety of tools in one research institute. Thus, through the corporation of the National Research Institute for Metals (NRIM), the Japan Atomic Energy Research Institute (JAERI) and the Japan Nuclear Cycle Development Institute (JNC), each with different specialist fields, a pilot distributed database system for development of nuclear materials called the "Data-Free-Way (DFW)" has been under construction since 1990. The actual project in its second stage, was started in 1995 through the collaboration of the NRIM, the JAERI, the JNC and the Japan Science and Technology Corporation (JST). The aim of this project is to store the data in a database and to develop a useful computer utilizing tool for data analysis, and simulation codes for various phenomena under irradiated environments.

The method of connection among the databases of each organization in the DFW system changed from private line to the Internet. Using tools in the Internet, users can easily refer to any necessary information by accessing the database with a WWW browser without knowing in which database site the required data exists. The final target of the usage of the WWW for the present study will be the development of a data information system for designing and selecting nuclear materials. At present, the data of more than 15000 specimens in various kind of nuclear environments are stored in the database of the DFW. Moreover, some trails of the WWW server of each were done to supply the information on nuclear materials. The DFW can be easily accessed by engineers and scientists in the advanced power engineering field.

Several databases and tools for the system are provided in the WWW of the NRIM site (<http://inaba.nrim.go.jp/>), which has the main functions of material database, on-line simulators and remote experiment. The databases consist of the DFW, the nuclear data of transmutation under neutron irradiation and decay for nuclear materials, diffusion information in Iron or Aluminum matrix, Ni superalloy properties, a CCT diagram for welding, and SiCf/SiC composite materials. Using the simulators, users are able to obtain the products of prediction of the chemical changes and radioactivation under neutron transmutation, evaluation of mechanical properties at high temperatures in Ni-base super-alloys and calculation of the welding thermal cycle.

The trials of the remote experiment applying the techniques used in the "Data-Free-Way" system are carried out among the NRIM, the JST and the Michigan State University (MSU) on a test line with a 45MB wide band. We have succeeded in accessing from the NRIM server to the JST and the MSU clients and transferring real time movies for various microstructures in several alloys. The results suggest that both researches who join the experiment on a real time base can discuss the results simultaneously. Moreover, some scientists in the NRIM discussed with scientists in the State Materials Testing Institute(MPA) Stuttgart, to mutually exchange material information using the DFW system at the 1998 German-Japanese Workshop on Chemical Information at Karlsruhe on the programming of German-Japanese Meeting of the Panel for Information and Documentation.

The number of accessions to the WWW (<http://inaba.nrim.go.jp/>) is more than 65000 persons per year from the entire world. The users are about 22000 from the USA, about 15000 from Japan, and about 28000 from 79 other countries. The results show that the WWW of the NRIM site in the DFW greatly contributes to the materials science field.

<Research theme>

- (1) Research on Utilization Technique of "Data-Free-Way" system for Nuclear Materials
(Nuclear Energy Research: April 1995 to March 2000, Mitsutane Fujita)

6.5.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

Database:

- (1) Data-Free-Way System: 15000 specimens
- (2) Nuclear Data for Nuclear Materials: 7500 nuclear reactions
- (3) Diffusion Information in Fe and Al Matrix : 800 records and CR-ROM
- (4) Ni Superalloy : 200 alloy
- (5) CCT Diagrams for Welding: "Atlas of CCT Diagrams for Welding", Special report 99-02 in NRIM (1999)
- (6) SiCf/SiC Composites : 200 specimens

Papers:

- (1) T.Noda, M.Fujita, M.Okada: Transmutation and Induced Activity of W First Wall of Fusion Reactors, J. Nucl. Mater., 258-263, 934(1998).
- (2) M.Fujita, N.Yokoyama, Y.Tachi, R.Nakajima: Distributed Material Database on the

- Internet (Present Status of Data-Free-Way System), RIST News, No.24, 25 (1997) (in Japanese)
- (3) T.Atago, S.Kikuchi, J.Tateyama, M.Fujita, H.Harada, T.Yokokawa, S.Sugawara, T.Miyazaki: An Experiment of Research Collaboration between Japan and USA Using Broadband Research Network, J. Info. Processing & Management, 40, 404(1997) (in Japanese).
 - (4) M.Fujita, T.Yokokawa T.Noda: A Material Information System by Using the Internet, Proc. Post 14th SMiRT Seminar, Paris, Aug.23-26, 33, 1997
 - (5) M.Fujita, K.Kusunoki: Formation of ICOS Cluster at Chisel Point in Necking Process, Proc. 2nd Inter. Symp. on Adv. Physical Field Characterization of Nanostructures, Tsukuba, Feb. 19-21 521, 1997
 - (6) M.Fujita, Y.Kurihara, M.Shindo, N.Yokoyama, Y.Tachi, S.Kano, S.Iwata: A Distributed Database System for Mutual Usage of Materials Information (Data-Free-Way) , ed by S. Nishijima, S. Iwata, ASTM STP 1311, 249(1997).
 - (7) T.Yokokawa, H.Harada, M.Fujita: Open Laboratory for Material Design Using Internet, Proc. 33rd Ann. Meet. Info. Sci. Tech. Tokyo Oct. 14-15,79,1997 (in Japanese).
 - (8) R.Nakajima K, Shimura, M.Fujita, Y.Kurihara, H.Tsuji, N.Yokoyama, S.Kano, Y.Tachi, S.Iwata : A Distributed Material Database on Internet Recent Activity in Data-Free-Way), Proc. 6th Ann. Conf. JS of Information and Knowledge., Tokyo May 23, 43, 1998 (in Japanese).
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Oral Presentation: 27

- (1) International Conference: 8
- (2) Domestic Conference: 19

Patent Application:

- (1). Retrieval System of CCT Diagrams, M. Fujita, T. Kasugai, 1998, H10-217278.

Award:

- (1) M. Fujita: Persons of Scientific and Technological Information Research Merits, 1998 Oct.14 from Japan Science and Technology Corporation

6.5.3 Research Budgets

This research has been supported by Nuclear Energy Research from April 1996 to March 2000.

1997: 15,507,000yen

1998: 17,223,000yen

6.6 Database for Superconducting Materials

Yuji Asada (Senior Researcher)

6.6.1 Research work

We have developed a numerical database for high-Tc oxide superconductors. All the data in it have been extracted from the papers reported in journals. Data are updated every month. The database is open to the public via the Internet: URL= asagiri.nrim.go.jp. Data are extracted partially by graduate students and Russians partners (for Russian papers).

A database of standardized data for typical high-Tc oxide superconductors is also being constructed and is partially opened via the Internet. Data in this database are produced by our working group. Details of sample characterization and measuring conditions of physical properties can be used for research work.

We have constructed a Tc-prediction system using the neural network method. The data set of the training of neural network was retrieved from our database.

<Research theme>

- (1) Development of Knowledge Database for High-Tc Superconducting Materials
(Multi-core project: April 1995 to March 2000, Yuji Asada)

6.6.2 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) Main products: Numerical Database for Superconducting Materials
- (2) Y.Asada, E.Nakada, S.Matsumoto, H.Uesaka: Prediction of Tc for $\text{YBa}_2\text{Cu}_3\text{O}_x$ Doped with Ca Using Neural Network, J.Superconductivity 10, 23-26(1997).

6.6.3 Budget

The funding for this research is provided from the Multi-core Research Project on Superconducting materials which has been set up by the Science and Technology Agency in 1988.

1997: 20,432,000 yen

1998: 20,060,000 yen

6.7 Surface Analysis Database for Characterizing Unknown Materials

Kazuhiro Yoshihara (Director of Center for Advanced Physical Fields)

6.7.1 Research staff

Kazuhiro Yoshihara (Director of Center for Advanced Field), Michiko Yoshitake (Unit Leader)

6.7.2 Research work

About 20 years has passed since commercial AES and XPS apparatuses appeared. Since that time, a number of spectral data for surfaces were obtained by many scientists and engineers. However, almost all of them were not shared and not stored as databases. Nowadays, it becomes important to stock is knowledge or data as databases, because we are aware that these data are very useful to characterize unknown surfaces. To create a spectral database, we have to establish a system to share spectral data taken on different machines.

NIST has published a database for XPS peak positions. AVS is publishing Surface Science Spectra. Manufacturers have their own spectral databases which they distribute to their customers. Usually, the objective of these databases is to provide users with spectral data of the clean surfaces of pure materials. However, the concept of this database is that "If we store the all spectral data of all surfaces taken on all machines, we can characterize any surfaces and calibrate any analyzers without difficulty". This concept is different from those of other existing databases, and we consider this database should be open to public. If the database is connected to the Internet, its usefulness will increase as will the number of scientists who can freely access the accumulated scientific property.

In 1994 the Science and Technology Agency (STA) of the Japanese Government launched a project to interconnect networks under various ministries and agencies. As a site of this network we are implementing a network-oriented database for surface chemical analysis, such as AES and XPS spectra. We asked the Surface Analysis Society of Japan (SASJ) to provide the spectral data, and to control its quality. Internet spectral data now has about 2,000 spectra of metals, semi-conductors and ceramics. The file structure of spectral data is based on ISO 14976 and ISO 14975, and is fully compatible with the VAMAS Standard Data Transfer Format. Because this file structure can carry the information on specimens, calibration and data-processing, we could construct a GUI searching system for the Internet database.

A workstation was installed in the National Research Institute for Metals to

collect the spectral data in ISO format from different analysis machines via computer networks. Collected spectra are stored in the database. If a personal computer is connected to the Internet, one can access the database, the address of which is <http://sekimori.nrim.go.jp>. When <http://sekimori.nrim.go.jp> is opened, one can select the database menu and retrieve a spectrum from the selection menu. The retrieved spectrum will be displayed and can be downloaded if a user is a member of the SASJ.

Spectral data have been collected through the voluntary work of the members of the SASJ using certified materials. The quality of spectra is checked by the committee of the SASJ. The committee checks the calibration procedures for energy and intensity scales of a supplier's analyzer, and rates a spectrum sent from a supplier. We are intending to construct an automatic spectra acquiring system for databases using Internet. If this system is established, one can easily send one's spectral data to the SASJ database through the Internet. The spectra will be used to identify the surface chemistry of new materials by comparing it with an observed spectrum.

Since 1989, we have been constructing the spectral data processing system under the VAMAS (Versailles Project on Advanced Materials and Standards) umbrella. This system is called Common Data Processing System (COMPRO). The COMPRO is designed to be a program to assess the data processing procedures provided by scientists, to check a spectrum, and to build both a spectra and a physical property databases. To achieve these objectives, the COMPRO provides a tool for converting spectral data taken on different instruments to a common one. The COMPRO (present version is 6.4) runs on Windows95/98/NT and can be downloaded from <http://sekimori.nrim.go.jp>.

In future we hope all computers of the surface analysis machines can be connected to the system so that every surface analyst worldwide can share the spectral data to characterize the surfaces of materials.

6.7.3 Research products, including papers submitted (April 1, 1997 to March 31, 1999)

- (1) H.Tokutaka, K.Yoshihara, K.Fujimura, K.Obu-Cann, K.Iwamoto, Application of self-organizing maps to chemical analysis, *Appl. Surf. Sci.*, **144/145**, 59-63(1999)
- (2) K.Obu-Cann, H.Tokutaka, K.Fujimura, K.Yoshihara: Chemical Analysis of AES, XPS and XRD Data using Self Organizing Maps, *J. Surf. Anal.*, **5**, 208(1999)
- (3) H.Tokutaka, K.Yoshihara, K.Fujimura, K.Iwamoto, K.Obu-Cann, T.Watanabe and S.Kishida: Application of Self-Organizing Maps(SOM) to Chemical Data Analysis, *J. Surf. Anal.*, **5**, 102(1999)

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- (4) S.Hofmann and K.Yoshihara: The MRI-model in COMPRO5:A new Data Processing Software for the Quantitative Evaluation of Sputter Depth Profiles, J. Surf. Anal., 5, 40(1999)
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- (6) M.Yoshitake and K.Yoshihara: Round Robin on Spectrometer Transmission Calibration for AES in the Common Data Processing System, Surf.Interface Anal., 25,209(1997)

Oral Presentation : 7

- (1) International Conference: 3
- (2) Domestic Conference: 4

6.7.4 Research Budgets

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1997: 12,569,000yen

1998: 10,897,700yen