Center of Excellence Development Project: 1997-2006
Supported through Special Coordination Funds from
Ministry of Education, Culture, Sports, Science and Technology

Photoreaction Control and Photofunctional Materials

Report for the 6th Year Evaluation

August 1st (Thu) and 2nd (Fri), 2002
National Institute of Advanced Industrial Science and Technology

Contents

I. Outline of Research Plans in the COE Development Area

One major problem mankind confronts today is the energy problem. In order to continue sustainable development, we need to establish technologies for securing clean, safe and inexpensive energy, without resort to fossil energy such as coal and petroleum. Solar energy is the only energy that may meet this criterion. Plants reproduce energy and resources by means of photosynthesis. The development of artificial photosynthetic systems which mimic photosynthesis is the ultimate solution to the energy problem.

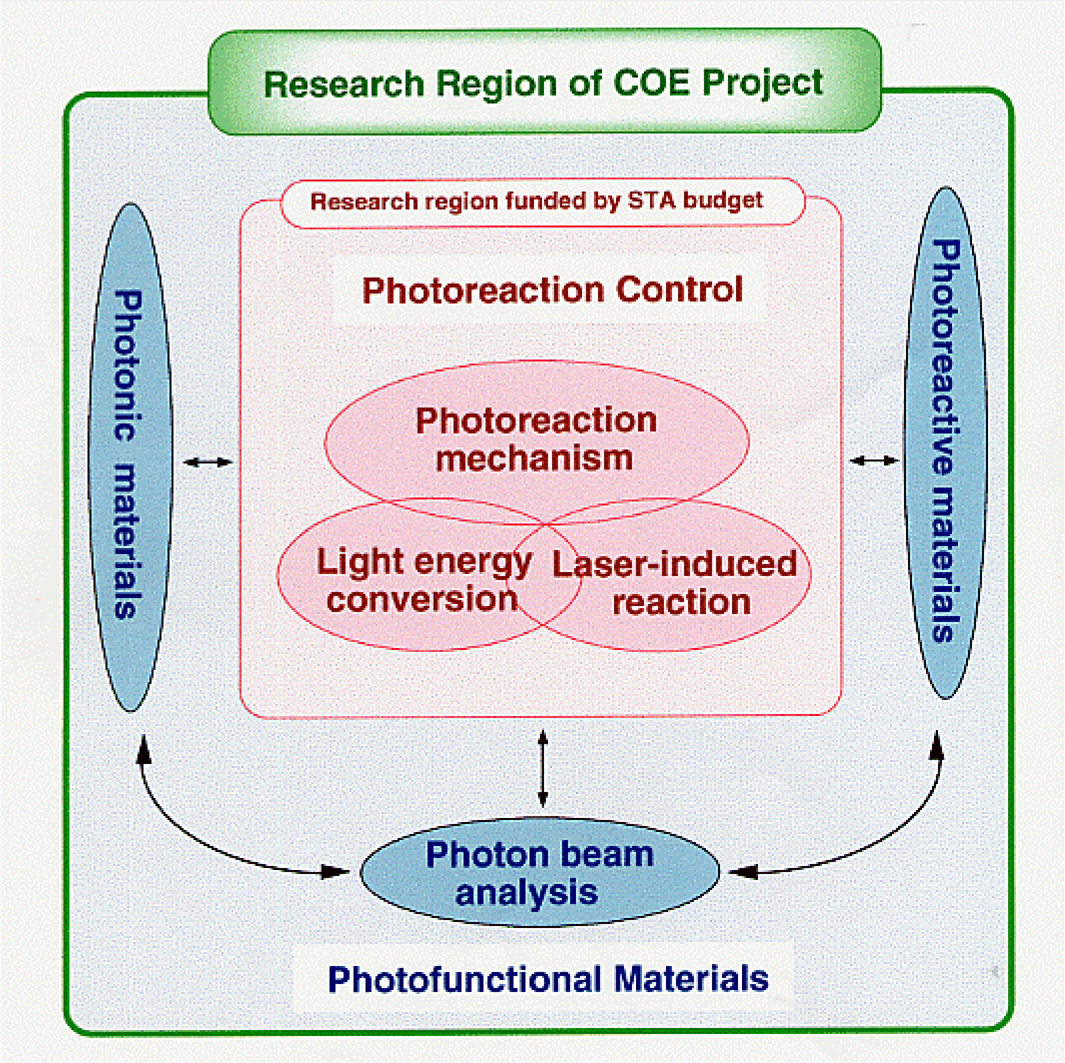
Another major problem is the environment problem. Chemical industries have made great contributions to mankind, but at the same time they have been imposing burdens on the environment by discharging unwanted by-products produced in chemical processes. A solution to this problem is the development of chemical processes without unwanted by-products.

Photoreaction control is expected to be a key technology to develop in solving both of these problems. Photoreaction control is, in our definition, a technology to control reactions at atomic and molecular levels by using photons. The success in developing artificial photosynthetic systems depends on how efficiently the visible region of solar light can be absorbed and used for energy conversion and how effectively electron transfer reactions that follow the absorption of solar light can be controlled. On the other hand, according to recent theoretical studies, it is possible to allow specific reactions to occur selectively with lasers by taking advantage of the coherent property of lasers. The discovery of this principle is expected to lead to the development of chemical processes free of unwanted by-products.

Light is drawing attention not only in connection with reaction control but also in connection with materials. With the information-intensive society close at hand, the needs for high-speed and high capacity information processing and transmission are intensified. Optical information processing is expected as a means to solve this problem, and the development of photofunctional materials is vital to the realization of optical information processing.

In this project we first explore fundamental principles for photoreaction control. By applying these principles we develop highly efficient artificial photosynthetic systems, photocatalysts, and solar cells for the effective use of solar energy. We also develop technologies for synthesizing materials selectively and processing materials on nano scales by using lasers. Furthermore, we develop photofunctional materials that are based on new principles, and contribute to the realization of optical information processing from the side of materials.

The Center of Excellence (COE) development area consists of Photoreaction Control part and Photofunctional Materials part, as shown in Fig. 1. The Photoreaction Control part constitutes the core of the COE development area, and is chosen as the area supported by Special Coordination Funds. The Photoreaction Control part is composed of three sub-themes, Photoreaction Mechanism, Light Energy Conversion, and Laser-Induced Reaction. In Photoreaction Mechanism, the interactions of photons with matter are elucidated by digging down to the levels of principles and phenomena, and on the basis of these studies guiding principles are explored for photoreaction control. By applying these principles, in Light Energy Conversion highly efficient artificial photosynthetic systems and dye-sensitized solar cells are developed for the effective use of inexhaustible solar energy. In Laser-Induced Reaction, technologies for synthesizing materials selectively and processing materials on nano scales are developed by using lasers. The Photofunctional Materials part also consists of three sub-themes, Photonic Materials, Photoreactive Materials, and Photon-Beam Analysis. In Photonic Materials we develop materials that fulfill their functions on the basis of their physical response to light. In Photoreactive Materials we develop materials that fulfill their functions on the basis of light-induced chemical changes. In Photon-Beam Analysis a variety of methods for analyzing materials microscopically are developed by using photons and lasers.



The research plans of each sub-theme are outlined below.

(1) Photoreaction mechanism (Research supported by Special Coordination Funds)

Elementary photoreaction processes such as energy transfer and electron transfer will be analyzed by spectroscopic methods using an ultrashort pulse lasers and by computer simulation. Guidelines on molecular design for efficiently converting solar energy to chemical energy will be prepared. Fundamental technologies for coherent reaction control, a new reaction control method applying the coherent characteristics of lasers, will also be developed.

(2) Light energy conversion (Research Supported by Special Coordination Funds)

Fundamental studies are conducted for the development of high performance solar cells mimicking photosynthesis mechanism of plant, and oxide semiconductor photocatalysts as well as supramolecular photocatalysts for water splitting under solar light irradiation.

Development of materials and reaction processes for enhancing overall energy conversion efficiency is another objective of this research.

(3) Laser-induced reaction (Research supported by Special Coordination Funds)

This research aims to study the interaction between ultraviolet laser and organic compounds at various forms and conditions of reactions, and to clarify the mechanisms of chemical and physical conversion caused by photo-irradiation. Based on the findings obtained, methods of synthesizing highly valuable chemical products will be developed. Also this theme will include fundamental, technological developments related to the production of highly functional micro-particles and thin films, and material surface processing.

(4) Photonic materials

To accomplish optical control by light, organic materials with high performance in optoelectronic properties such as photoconductivity, electroluminescence, and photoisomerization, will be developed. The substances are focussed into s and p conjugated materials, silicon-based polymer (polysilane) and poly-para-phenylene (PPP), poly-para-phenylen-vinylene (PPV) and their oligomers and azo-benzene polymer. And for the optoelectronic device application the structure in thin solid film is controlled. We have established new methods for controlling molecular orientation in film, capillary filling and friction transfer, to examine the dependence of film structure on the carrier mobility and the anisotropic luminescence. The combining of photoconductive and electroluminescent compounds is applied to photo-responsive electroluminescent device with the layered structure. And the photoisomerization of azo-benzene moiety is applied to the formation of photo-induced surface relief grating. Thus fundamental and practical studies should open next electronics and photonics in future.

(5) Photoreactive materials

Signal transduction and transmission in the vision systems of living creatures are considered as switching and chemical amplification triggered by light-induced structural changes of organic materials. Using these vision systems as one of the models, molecular devices with the ability of signal transduction and amplification will be developed. Photochromic molecules will mainly be used as the photoresponsive materials that undergo light-induced structural changes. The transduction and amplification of signals by photochromic molecules in the LB films, bilayers, self-assembled monolayers and other molecular assemblies will be clarified using various techniques such as scanning probe microscopy.

(6) Photon-Beam analysis

The clarification of phenomena in micro sites is essential to research on advanced substances and materials. Technologies to detect and analyze micro amounts of substances that can be differentiated at the atomic and molecular levels using light, especially laser, will be developed to fulfill this requisite. Studies will also be conducted on the effects of surface properties on material functions, by measuring the surface of functional materials using spectrometers which adopt strong ultrashort wavelength (1 nm-10 nm) light as the excitation source.

II. Research Organization and Management

1. Research Groups in the COE Development Area

General Manager	
Dr. Masanori Tachiya	AIST Fellow
Vice General Manager	
Dr. Hironori Arakawa	Photoreaction Control Research Center, Director

(1) Photoreaction Mechanism Group

Dr. Masanori Tachiya	AIST Fellow
Dr. Osamu Kitao	Photoreaction Control Research Center, Senior Research Scientist
Dr. A.V. Barzykin	Photoreaction Control Research Center, Senior Research Scientist
Dr. Makoto Fushiki	Research Institute for Computational Sciences, Senior Research Scientist (April 1997 - March 2001)
Dr. Kazuhiko Seki	Photoreaction Control Research Center, Senior Research Scientist
Dr. Shigeo Murata	Photoreaction Control Research Center, Leader
Dr. Toshio Fukaya	Laboratory for Advanced Optical Technology, Deputy Director (April 1997 - March 1999)
Dr. Takashi Arimura	Photoreaction Control Research Center, Senior Research Scientist
Dr. Ryuzi Katoh	Photoreaction Control Research Center, Senior Research Scientist (April 1998 -)
Dr. Shinichiro Iwai	Correlated Electron Research Center, Senior Research Scientist (April 1997 - March 2001)
Dr. Akihiro Furube	Photoreaction Control Research Center, Research Scientist (April 2001 -)
Dr. Taisuke Nakanaga	Photoreaction Control Research Center, Leader
Dr. Hidekazu Nagai	Photoreaction Control Research Center, Senior Research Scientist
Dr. Fumiyuki Ito	Photoreaction Control Research Center, Senior Research Scientist
Dr. Jun Miyawaki	Nanotechnology Research Institute, Senior Research Scientist (April 1998 - March 1999)
Dr. Hideki Ohmura	Photoreaction Control Research Center, Research Scientist (April 1999 -)

(2) Light Energy Conversion Group

Dr. Hironori Arakawa	Photoreaction Control Research Center, Director
Dr. Hideki Sugihara	Photoreaction Control Research Center, Leader
Dr. Seiji Ide	Fukuoka Industrial Technology Center (April 1998 - March 2000)
Dr. Masahiro Kaise	Retired (April 1998 - March 2001)
Dr. Kazuhiro Sayama	Photoreaction Control Research Center, Senior Research Scientist
Dr. Yoshinari Konishi	Photoreaction Control Research Center, Senior Research Scientist (April 2001 -)
Dr. Hitoshi Kusama	Photoreaction Control Research Center, Senior Research Scientist
Dr. Kohjiro Hara	Photoreaction Control Research Center, Research Scientist (October 1998 -)
Dr. Masatoshi Yanagida	Photoreaction Control Research Center, Research Scientist (April 2000 -)
Dr. Ryu Abe	Photoreaction Control Research Center, Research Scientist (April 2002 -)
Dr. Kazuyuki Kasuga	Photoreaction Control Research Center, Deputy Director
Dr. Yuichiroh Himeda	Photoreaction Control Research Center, Senior Research Scientist
Dr. Nobuko Onozawa	Photoreaction Control Research Center, Research Scientist

(3) Laser-Induced Reaction Group

Dr. Akira Yabe	Retired (April 1997 - March 2002)
Dr. Hiroyuki Niino	Photoreaction Control Research Center, Leader
Dr. Yoshizo Kawaguchi	Photoreaction Control Research Center, Senior Research Scientist (April 2001 -)
Dr. Tadatake Sato	Photoreaction Control Research Center, Research Scientist (April 1999 -)
Dr. Aiko Narazaki	Photoreaction Control Research Center, Research Scientist (April 2000 -)
Dr. Naoto Koshizaki	Nanoarchitectonics Research Center, Leader
Dr. Akio Watanabe	Institute for Materials & Chemical Process, Senior Research Scientist (April 1997 - March 2001)

Dr. Takeshi Sasaki	Nanoarchitectonics Research Center, Senior Research Scientist
Dr. Tetsuo Tsuchiya	Institute for Materials & Chemical Process, Research Scientist (April 2001 -)

(4) Photonic Materials Group

Dr. Kiyoshi Yase	Photonics Research Institute, Deputy Director
Dr. Hiroshi Ushijima	Photonics Research Institute, Senior Research Scientist
Dr. Noriyuki Takada	Photonics Research Institute, Senior Research Scientist
Dr. Hiro Matsuda	Photonics Research Institute, Leader
Dr. Takashi Fukuda	Photonics Research Institute, Senior Research Scientist
Dr. Tatsumi Kimura	Photonics Research Institute, Research Scientist (April 2001 -)
Dr. Toshihide Kamata	Photonics Research Institute, Leader
Dr. Kenji Kozasa	Photonics Research Institute, Research Scientist (April 2001 -)
Dr. Manabu Yoshida	Photonics Research Institute, Research Scientist (April 2001 -)
Dr. Shinji Minami	Nanotechnology Research Institute, Deputy Director (April 1997 - March 2002)
Dr. Said Kazaoui	Nanotechnology Research Institute, Senior Research Scientist (April 1997 - March 2002)

(5) Photoreactive Materials Group

Dr. Mutsuyoshi Matsumoto	Nanotechnology Research Institute, Leader
Dr. Reiko Azumi	Nanotechnology Research Institute, Senior Research Scientist
Dr. Toru Nakamura	Nanotechnology Research Institute, Senior Research Scientist
Dr. Hiroaki Tachibana	Correlated Electron Research Center, Leader (April 1997-March 2001)
Dr. Yuji Kawanishi	Nanotechnology Research Institute, Leader
Dr. Masako Sakuragi	Evaluation Department, Senior Researcher
Dr. Yoriko Sonoda	Nanotechnology Research Institute, Senior Research Scientist
Dr. Norihiko Minoura	Laboratory of Advanced Bioelectronics, Deputy Director

Dr. Masahiro Higuchi	Nanoarchitectonics Research Center, Senior Research Scientist

(6) Photon-Beam Analysis Group

Dr. Isao Kojima	Metrology Institute of Japan, Head
Dr. Kenji Kato	Metrology Institute of Japan, Chief
Dr. Naoki Matsuda	Nanoarchitectonics Research Center, Senior Research Scientist (April 1999 -)
Dr. Hiromichi Shimada	Planning Headquarters, Senior Planning Officier
Dr. Nobuyuki Matsubayashi	Metrology Institute of Japan, Senior Research Scientist
Dr. Hisakazu Nozoye	Nanotechnology Research Institute, Leader
Dr. Hiroshi Kondo	University of Tokyo (April 1997 - March 1998)
Dr. Toshitaka Kubo	Nanotechnology Research Institute, Research Scientist (August 1998 -)
Dr. Takayuki Miyamae	Nanotechnology Research Institute, Research Scientist (April 1999 -)
Dr. Katsutoshi Aoki	Institute for Materials & Chemical Process, Deputy Director

2. Research Management in the COE Development Area

2-1. Securing and Nurturing Excellent Human Resources

(1) Public collection of applications in employment

Excellent researchers have been employed under very competitive circumstances by colleting applications publicly. The ratio of total applicants to employed ones has been more than ten during these several years. In the recruitment the priority has been placed on researchers in the COE development area. The number of new staff researchers employed in the COE development area is listed below.

Table 2-1-1. New Staff Researchers Employed

Fiscal Year	1997	1998	1999	2000	2001	2002
COE development area	0	3	3	2	4	1
Special Coordination Fund supported area	0	2	2	2	2	1

(2) Recruitment of capable postdoctoral fellows

In the COE development area, many postdoctoral fellows have been accepted from the inside and outside of the country by using the Special Coordination Funds and various fellowships. Among them exceptionally capable ones have been employed as staff researchers in competition with other applicants. As our COE project becomes widely known inside and outside the country, the applicants for postdoctoral fellows have increased, which helps to excavate capable human resources. The number of postdoctoral fellows accepted in the Special Coordination Fund supported area is listed below.

Table 2-1-2. Postdoctoral Fellows Accepted

Fiscal Year	1997	1998	1999	2000	2001	2002	Total
Total Number Accepted	28	29	31	29	21	10	148
Number from Overseas	20	18	19	17	13	8	95

(3) Recruitment of graduate students

The Institute has started the Cooperative Graduate School Program, first with University of Tsukuba, later on with many other universities such as Tokyo Institute of Technology, Science University of Tokyo and so on. Up to now 15 staff researchers in the COE development area have been jointly appointed as professors or associate professors at graduate schools. The Cooperative Graduate School Program allows us to accept many graduate students from national and private universities, supervise them in research, and contribute to the nurturing of excellent human resources. The number of graduate students accepted in the COE development area is listed below (The figures in Fiscal Years 2000-2002 include only those accepted in the Special Coordination Fund supported area)

Table 2-1-3. Graduate Students Accepted.

Fiscal Year	1997	1998	1999	2000	2001	2002	Total
Number Accepted	15	17	19	4	3	6	64

2-2. Furnishing the Research Platform

(1) Research equipments and apparatuses

Research equipments and apparatuses that are indispensable for Photoreaction Control research, but too expensive to buy using ordinary research expenditures have been introduced by using Special Coordination Funds. They have been effectively used to produce the world’s top-class results. Main research equipments and apparatuses introduced in the COE development area are listed below.

1997	High-power femtosecond laser system
	Laser-beam decomposition reaction control equipment
	X-ray diffraction equipment for photocatalyst crystal analysis
	High-energy laser reaction irradiation device
1998	Cavity ring down laser spectroscope
	High-speed metal dispersion measuring device
	Solar cell evaluation equipment
	Ultrahigh-vacuum laser ablation equipment
1999	Photoionization detection type reaction control equipment
	Transient absorption spectrum measuring device
	Time-resolved near-infrared fluorescence spectrum measuring device
	Excimer laser for the light energy conversion excitation

	Active species excitation equipment
	Nano fine particle dynamic shape evaluation equipment
2000	High-quality wavelength-variable laser system for the photoreaction control
	Time-resolved transient Raman spectroscope
	Semiconductor photoelectrode surface characteristics equipment
	Vacuum ultraviolet spectrophotometer
2001	Picosecond time-resolved transient absorption spectroscopy system
	Infrared OPO laser equipment

(2) Measurement and Analysis Research Center

Main research equipments and apparatuses that have been introduced in the Measurement and Analysis Research Center are listed below.

1997	Electrospray mass spectrometer
1998	Precision mass spectrometer
1999	Ionizer for the precision mass spectrometry equipment
2000	Superconductive nuclear magnetic resonance apparatus

(3) Photoreaction Control Research Building

The construction of “Photoreaction Control Research Building” was planned for the purpose of securing research spaces that are necessary for efficient running of equipments and apparatuses, organic collaboration between different research groups, and joint research with industries. However, this has not yet been realized. On the other hand, with the launch of the National Institute of Advanced Industrial Science and Technology (AIST) in 2001, the allotment and usages of research spaces within the Institute have been made more reasonable and more flexible. This has enabled us to centralize spaces for Photoreaction Control research to some extent and secure spaces which are necessary for organic collaboration between different research groups and joint research with industries.

2-3. Maintaining Openness and Mobility

(1) Recruitment and acceptance of diverse human resources

Staff researchers have been employed by collecting applications publicly. They have been recruited widely from overseas, universities, other national research institutes, companies, and so on. Employment on a fixed term basis is positively adopted. Concerning recruitment from overseas, currently we have two foreign staff researchers in the COE development area. We plan to increase foreign staff researchers to more than 10% of the total staff researchers in the COE development area within 10 years. We have accepted many researchers from outside by using the Special Coordination Funds, various fellowships, and the Cooperative Graduate School Program. The numbers of accepted postdoctoral fellows and graduate students are already listed in 2-1. The total number of postdoctoral fellows and graduate students is always comparable to that of staff researchers. The number of guest researchers invited in the COE development area, excluding postdoctoral fellows and graduate students is listed below. Postdoctoral fellows, graduate students, and guest researchers from different countries and organizations participate in our COE project by turns, thereby stimulating our research activities..

Table 2-3-1. Invited Researchers Other than Postdoctoral Fellows and Graduate Students

Fiscal Year	1997	1998	1999	2000	2001	2002	Total
Number Accepted	32	46	54	40	34	-	206

(2) Research collaboration

We have concluded agreements on research cooperation with several overseas universities and research institutes. In 1998 we started a research cooperation program with Université Louis Pasteur and have been exchanging 2 researchers every year with them. In 2001 we concluded an agreement on comprehensive research cooperation with Centre National de la Recherche Scientifique (CNRS). Various informal research collaborations are also in progress. For example, we are collaborating with several institutes of Russian Academy of Sciences and so on on electron transfer reactions, with Ecole Polytechnique Federale de Lausanne and so on on dye-sensitized solar cells, and with University of Linz and so on on laser processing.

(3) Research group system

In 1997 National Institute of Materials and Chemical Research (NIMC) abolished the rigid laboratory system and introduced the research group system to ensure open and mobile research environments. Although this system proved very effective for securing mobility in research environments, unfortunately it was discontinued in 2001 with the launch of the AIST.

2-4. Research Evaluation Function

(1) External evaluation committee for the COE project

An external evaluation committee has been set up to evaluate research achievements and research management in the COE development area. The members of the committee are listed below. The first meeting of the committee was held in the 3rd year of the project (August 26 and 27, 1999). The committee gave excellent ratings to the project. Several improvements were made in the research plans and the research management on the basis of the evaluation results. The second meeting will be held in the 6th year of the project (August 1 and 2, 2002). The final meeting will be held in the 10th year of the project.

Table 2-4-1. Members of the External Evaluation Committee for the COE Project

Name	Affiliation	Specialty
Katsumi Tokumaru	Professor Emeritus, University of Tsukuba	Photochemistry
Frans C. De Schryver	Professor, Department of Chemistry, Katholieke Universiteit Leuven, Belgium	Photolysis, Confocal scanning probe microscopy, Fluorescence spectroscopy
Kinichi Obi	Professor, Department of Chemical and Biological Sciences, Japan Women's University	Photoreaction dynamics
Peter A. Hackett	Vice-President for Research, National Research Council, Canada	Chemical dynamics, Cluster photophysics, Photochemistry
Shozo Yanagida	Professor, Graduate School of Engineering, Osaka University	Organic physical chemistry, Photocatalysis
Michael Graetzel	Professor, Institute de Chemie Physique II, Ecole Polytechnique Federale de Lausanne, Switzerland	Photoinduced charge separation, Nanocrystalline solar cells, Dye sensitization, Mesoporous electronic junctions, Hole conduction and hetero junctions
Hiroyuki Sasabe	Professor, Department of Photonic Materials Science, Chitose Institute of Science and Technology	Organic nonlinear optics, Molecular electronics
Arthur J. Nozik	Senior Research Fellow, National Renewable Energy Laboratory, USA	Photoelectrochemistry, Quantum dots, Electron transfer across semi conductor-molecule interfaces, Photocatalysis, Photon conversion of solar energy, Materials science
Tsuguo Sawada	Professor, Graduate School of Frontier Sciences, University of Tokyo	Laser analytical chemistry, Laser photoacoustic and photothermal spectroscopies, Laser ultrasonics, Microchemistry

(2) Internal hearing committee

NIMC held the internal hearing committee each year to evaluate each group's research achievements of that year and research plans of the next year. The committee was comprised of Director General, Deputy Director General, Director of Research Planning Office, Directors of Research Departments and so on. At AIST, Research Institutes and Research Centers have been installed as research units in place of Research Departments. The Photoreaction Control part of the COE development area has moved as a body to one of the Research Centers, Photoreaction Control Research Center. Unfortunately the Photofunctional materials part has been scattered among several research units. Each research unit is evaluated by several external reviewers selected for each research unit. Evaluation results are reflected in the allocations of research funds and achievement bonus funds to each research unit.

(3) Evaluation of individual researchers

At NIMC the evaluation of individual researchers was done by making each researcher present his research activity report each year. With the launch of AIST, a new evaluation system of individual researchers has been started. In this system, each researcher prepares his research and work plans at the beginning of each fiscal year on the basis of the research and work plans of the group to which he belongs, and obtains approval from his supervisor. At the end of the fiscal year, each researcher prepares a self-evaluation report, and his supervisor evaluates his achievements in reference to his self-evaluation report. The evaluation results are reflected in the achievement bonus of each researcher.

2-5 Flexible Research Management

(1) Research Center

Researchers engaged in the Photoreaction Control research that is supported by the Special Coordination Funds came originally from seven different Research Departments. In order to efficiently promote this research, Special Department for COE Project was set up in 1998 as a research unit that was independent of other Research Departments, and the management of the Department was entrusted to the head of the Department. With the launch of AIST, the Special Department for COE Project has evolved to one of the Research Centers, Photoreaction Control Research Center. In general the directors of Research Centers are given a greater discretional power to manage their Centers, compared with the head of the Special

Department for COE Project.

(2) Discretionary power of sub-leaders

A total of three sub-groups were formed in the Special Department for COE Project in accordance with sub-themes in the Photoreaction Control research. A sub-leader was appointed for each sub-group, and a considerable discretionary power was given to each sub-leader. When the Special Department for COE Project evolved to Photoreaction Control Research Center, the sub-groups were restructured into four teams.

(3) COE Promotion Committee

NIMC installed the COE promotion committee to support the COE project throughout the whole institute. The committee was comprised of Director General, Deputy Director General (General Manager of the COE project) , Head of the Special Department for COE Project, Director of Research Planning Office and Director of Department of General Affairs. Each fiscal year the Head of the Special Department for COE Project reported the research achievements and management of the previous fiscal year and the research and management plans of the current year. The committee provided necessary advices and supports on the basis of his report. With the launch of AIST, the COE promotion committee disappeared, and a system of supporting COE project has yet to be established in the new organization (AIST).

(4) Flexible management of usages of research budgets

In 2001, 15 research institutes including NIMC, which belonged to Ministry of Economy, Trade and Industry were united into an independent agency, AIST. In independent agencies, operating expenses are provided by the government as grants. Concerning the grant for operating expenses, which comprises about 80% of the total budget of the Institute, how it is spent can be determined by the Institute without any restriction, with the exception of personnel expenses. The new system allows researchers to determine the usages of their allocated research budgets at their discretion so as for their researches to proceed most efficiently.

(5) Flexible management of the number of employees

In independent agencies, the strict restriction on the number of employees was abolished. The number of employees can be determined by the Institute more flexibly, although there is some restriction on the total personnel expenses the Institute can spend from the grant for operating expenses.

2-6. Disseminating Research Results

(1) Publication of papers in first-class journals

The numbers of published papers in the COE development area are listed in IV. 1. The total number of papers published in international journals is 248 and accounts for the majority. The numbers of papers published in journals with high impact factors are listed in IV. 1. The impact factor is an index that indicates the significance of each journal in its research field. There are, of course, limitations for using it as an index to represent the importance of individual papers published in that journal. In our opinion, it is, however, meaningful to some extent to consider the impact factor not just for a single paper but for the whole ensemble of published papers in the project and use it as an index for evaluating the research achievements of the project. From this point of view, in this project we consider the impact factor calculated from the whole ensemble of published papers in the project and regard the increase of the impact factor as an index that indicates the development of COE. The impact factor calculated in such a way has been increasing steadily.

(2) Patents

The numbers of patents applied for in the COE development area are listed in IV. 1. The number of patents applied for to European and North American countries is 25. Researchers are encouraged to apply for patents, if they invent new technologies that may draw the interest of industries. Patent applications to foreign countries, however, cost considerable amount of money. Therefore, within the limited budget, only a part of patents that we wish to apply for are actually applied for.

(3) Symposia

Since the start of the COE project, we have held International Symposium on Photoreaction Control and Photofunctional Materials (PCPM) every year to disseminate our research achievements. Up to now we have organized the symposium five times. Each time many (300 or more) people have attended the symposium, and more than 40 are from overseas. Each time Extended Abstracts of all papers presented at the symposium have been published. At the 4th and 5th symposia, the Proceedings that include all invited and oral presentations have been published in *J. Photochem. Photobiol. A*. In addition, we have frequently (on the average more than one seminar per month) organized seminars by speakers invited from outside to promote exchange of ideas and information on research.

(4) Research achievement report

All published papers in the COE development area have been collected and published as two volumes of book each year.

(5) Paper presentations at research conferences

The number of papers presented by researchers in the COE development area at research conferences is listed in IV. 1. Many papers have been actively presented at international conferences.

3. Role of the Special Coordination Fund Supported Research in the COE Development Area

(1) Core of the COE development area

The COE development area consists of Photoreaction Control part and Photofunctional Materials part. Since the Photoreaction Control part constitutes the core of the COE development area, it has been chosen as the area supported by Special Coordination Funds. Although the Photofunctional Materials part has been scattered among several research units with the launch of AIST, the Photoreaction Control part has moved as one body to the Photoreaction Control Research Center and continues its role as the core of the COE development area.

(2) Collaboration within the core

In the Photoreaction Control part that is supported by Special Coordination Funds, organic collaboration between different sub-themes is carried out. For instance, the Photoreaction Mechanism group are theoretically and experimentally studying electron transfer processes in dye-sensitized semiconductors in collaboration with the Light Energy Conversion group, and supporting their work on the development of dye-sensitized solar cells from the basic side of research. The Laser-Induced Reaction group is developing technologies for producing semiconductor nanoparticles with lasers. They are important for the development of dye-sensitized solar cells, which is pursued by the Light Energy conversion group.

(3) Collaboration between the core and satellites

Concerning the relation between the Special Coordination Fund supported area “Photoreaction Control” and self-supported area “Photofunctional Materials”, collaboration between them are gong very well with the Special Coordination Fund supported area playing a role as the core. For example, the Photoreaction Mechanism group are analyzing ultrafast optical processes in nonlinear optical materials by femtosecond spectroscopy in collaboration with the Photonic Materials group and supporting their work on the development of nonlinear optical materials from the basic side of research. The Photoreaction Mechanism group is also collaborating with the Photonic Materials group in the study of charge transport materials and developing a theoretical model for charge transport in disordered systems. The Photon-Beam Analysis group is developing a unique method called Slab Optical Waveguide (SOWG) spectroscopy, which is believed to be useful for analyzing an ultra small amount of absorbed substances. The Light Energy Conversion group is trying to apply this method to the study of sensitizing dyes absorbed onto semiconductor surfaces in collaboration with the Photon-Beam Analysis group.

4. Impact of the COE Project on the Whole Institute

(1) Activation of the whole Institute

The COE Project has contributed to the leveling up and activation of the whole Institute by stimulating other research activities of the Institute. Since the start of the COE Project in 1997, the numbers of published papers, invited talks, awards received, and competitive research grants acquired have all been increasing steadily.

(2) Installation of Research Centers in the new Institute

Researchers engaged in the Photoreaction Control research that is supported by the Special Coordination Funds came originally from seven different Research Departments. In order to efficiently promote this research, Special Department for COE Project was set up in 1998 as a research unit that was independent of other Research Departments, and the management of the Department was entrusted to the head of the Department. Since this department worked very effectively, AIST has decided to install organizations called Research Centers, which are modeled on this Department. Currently, AIST has 26 Research Centers. The Special Department for COE Project has moved to one of Research Centers, Photoreaction Control Research Center.

(3) Flexible management of the usages of research budgets

Special Coordination Funds allotted to the COE project can be used flexibly at discretion of the General Manager of the project. It can be used even for employing postdoctoral fellows, which have had very good effects on promoting research. Based on this experience, AIST has introduced a flexible budget management system that allows researchers to determine the usages of their allocated research budgets at their discretion so as for their researches to proceed most efficiently.

(4) Introduction of a new evaluation system in the new Institute

The external evaluation system adopted for the COE Project has enhanced researchers' motivation toward their research and heightened their will to do good research. When AIST was established, it has introduced the external evaluation system for research units in reference to the evaluation system for the COE project. In the evaluation system for research units each research unit is evaluated by several external reviewers selected for each research unit. Evaluation results are reflected in the allocations of research funds and achievement bonus funds to each research unit.

5. Support of the Ministry Supervising the Institute

(1) Infrastructure

A supercomputer system “Advanced Parallel Computing System” that has a world's top class ability of fast computing has been installed.

(2) Research budgets

In 2001, 15 research institutes that belonged to Ministry of Economy, Trade and Industry were united into an independent agency, AIST. In independent agencies, operating expenses are provided by the government as grants. Concerning the grant for operating expenses, which comprises about 80% of the total budget of AIST, how it is spent can be determined by the Institute without any restriction, with the exception of personnel expenses. Efforts have been made to increase the total budget of AIST.

(3) Securing man power

Individual researchers have been given authority to accept postdoctoral fellows by using their allocated research budgets in addition to various fellowships. At the same time the Institute has been given authority to expand the Cooperative Graduate School Program at their own discretion.

(4) Organization for the COE project

When AIST was established in 2001, 23 Research Centers and 22 Research Institutes were installed as research units. To continue Photoreaction Control research in the COE development area, Photoreaction Control Research Center has been set up as one of Research

Centers. As for research units related with Photofunctional Materials research, several research units have been set up which include Photonics Research Institute, Nanotechnology Research Institute and so on.

(5) Financial support for the COE project

Concerning the allocation of research funds, Photoreaction Control Research Center has been given high priority as one of Research Centers, compared with Research Institutes.

(6) Administrative support

The administrative procedures have been rationalized and simplified by introducing an intranet system that computerizes the procedures.

(7) Accommodation for long-term visiting researchers

An accommodation facility "Keyakikan" was built in October 1997 for long-term visiting researchers from abroad.

(8) Support for acquisition of patents

In order to effectively manage patents owned by AIST, "AIST Innovations" has been established as a TLO organization that is responsible for patent strategies of AIST. "Regulations concerning Inventions by Employees" have been revised to effectively manage patents at AIST. It has been decided that all patents arising from inventions made by employees during their duty work belong to AIST. At the same time it has been decided for the purpose of promoting incentives of individual researchers that an individual researcher who made an invention is given one fourth of the royalty of the patent arising from that invention.

(9) Employment of researchers in the COE development area

New staff researchers have been employed on a fixed-term basis with the priority placed on the COE development area.

6. Measures Taken on the Basis of the Third Year Evaluation Results.

(1) Promoting discussions and collaborations

In the 3rd year evaluation report it is recommended to further promote discussions within each sub-theme and collaborations between sub-themes. Efforts have been made to enhance the quality of research by increasing the opportunities for discussions and having intensive discussions within each sub-theme. Concerning collaborations between sub-themes, the Photoreaction Mechanism group, for example, is theoretically and experimentally studying electron transfer processes in dye-sensitized semiconductors in collaboration with Light Energy Conversion group and has made significant contributions. The field of dye-sensitized solar cells is a field of globally keen competition. Fortunately, this project team has three strong research groups specialized in the development of dyes and semiconductors, in the observation of electron transfer processes by femtosecond spectroscopy, and in the theory of electron transfer processes. Therefore, we believe that in its integrated strength this project team occupies the world's top class position in the field of dye-sensitized solar cells.

(2) Photoreaction Control Research Building

The report has recommended that "the Photoreaction Control Research Building" should be constructed at the early stage to further enhance synergistic effects due to collaborations between project members. However, this has not yet been realized. On the other hand, with the launch of AIST, the allotment and usages of research spaces within the Institute have been made more reasonable and more flexible. This has enabled us to centralize spaces for Photoreaction Control research to some extent and secure spaces which are necessary for organic collaboration between different research groups and joint research with industries.

(3) Strategies for recruiting capable researchers

The report has recommended that strategies for recruiting capable researchers from overseas should be worked out. Efforts have been made to have the presence of AIST in this area recognized all over the world by organizing international symposia to disseminate the research achievements in the COE development area of AIST and publishing Proceedings in an international journal, *J. Photochem. Photobiol. A*. Close contact with many overseas research groups has also been kept to secure capable researchers through this route. A homepage has been built to introduce the project.

7. Outline of the Plans after the Sixth Year

(1) World's top class integrated team

In the field of dye-sensitized solar cells, we have established a research team which includes not only applied but also basic and theoretical research groups and is the world's top class in its integrated strength. We are also planning to form the world's top class team in the field of laser ablation, which includes both applied and basic research groups.

(2) AIST as COE in the field of Photoreaction Control and Photofunctional Materials

This project was started in 1997 with the aim of making NIMC a Center of Excellence in the field of Photoreaction Control and Photofunctional Materials. In 2001 NIMC was unified with 14 other research centers to establish AIST. In the new framework, the objective of the project has changed to making AIST a Center of Excellence in the field of Photoreaction Control and Photofunctional Materials. The number of researchers in the field of Photofunctional Materials has been increased remarkably by the unification, although that in the field of Photoreaction Control remains almost the same. Our future task is to work out strategies to make AIST a Center of Excellence in the field of Photoreaction Control and Photofunctional Materials, by including the researchers increased.

III. Outline of the Research Achievements

1. Study of Photoreaction Mechanism

1-1. Theoretical Study of Photoinduced Electron Transfer

M. Tachiya (Fellow)
A. V. Barzykin, K. Seki (Photoreaction Control Research Center)

Purpose

In the conversion of light energy it is extremely important that charges are separated efficiently after the light is absorbed and that recombination of separated charges is suppressed. The purpose of this theoretical study is to elucidate physical factors that determine the rate of electron transfer and develop physical methods to control the rate and yield of electron transfer. Up to the third year of the project we studied electron transfer in liquids. We elucidated the relation between the rate of electron transfer and physical factors such as the donor-acceptor distance, the polarity of the solvent, the free energy change of reaction and so on, and generalized the Marcus theory of electron transfer [1]. We also studied electron transfer in photosynthetic reaction centers. We introduced two dimensional free energy surfaces for the first time to treat electron transfer in these systems, and showed that the superexchange mechanism is more important than the sequential mechanism. Since the fourth year of the project we have been studying the effect of an applied electric field on electron transfer theoretically for the purpose of controlling the rate and yield of electron transfer by means of an applied electric field. Electron transport process in doped polymers is important in the field of electrophotography. We have extended our work on the applied electric field and studied electron transport process in doped polymers to contribute to the improvement of the performance of hole transport materials. We have also studied electron transfer in dye-sensitized nanocrystalline semiconductors for the purpose of the enhancement of the efficiency of dye-sensitized solar cells. Electron transfer in liquids has already been studied extensively and its basic physical picture has been well established. However, in dye-sensitized nanocrystalline semiconductors and doped polymers described above, electron transfer at interfaces or in disordered systems is important. Elucidation of electron transfer in these complex systems is a very challenging problem.

Results and Future Problems

According to the Marcus theory of electron transfer, the rate of electron transfer depends on the free energy change of reaction. Since electron transfer changes the charges of reactants, the free energy change of reaction is affected by an applied electric field. Therefore, the rate of electron transfer is also affected by the applied electric field. The luminescence of the excited donor will be decreased by electron transfer from the excited donor to acceptors. If an electric field is applied to this system, the rate of electron transfer will be changed, and consequently the luminescence intensity of the excited donor will also be changed. We have theoretically studied the effect of an applied electric field on electron transfer from the excited donor to acceptors in the polymer matrix and investigated how the luminescence intensity of the excited donor is affected by the applied electric field [2,3]. In the calculation of the free energy change in the presence of an external electric field it is important to know that the electric field donors and acceptors dissolved in the polymer matrix actually feel is not equal to the applied electric field because of the dipoles induced on the polymer interface around donors and acceptors. We have developed a rigorous method of calculating the free energy change, which takes into account this effect [4]. Two cases were considered, one in which donors and acceptors are separately distributed randomly in the polymer matrix [2], and the other in which pairs of donor and acceptor linked through a spacer and separated by a fixed distance are dissolved in the polymer matrix [3]. We have shown that in both cases the variation in the steady-state luminescence intensity due to the applied electric field is proportional to the square of the field strength when the applied electric field is not so high (Fig. 1.1.1). In the case in which donors and acceptors are separately distributed randomly the applied electric field is shown to decrease the luminescence intensity (Fig.1.1.1). On the other hand, in the case in which a donor and an acceptor are linked through a spacer and separated by a fixed distance the luminescence intensity can be either increased or decreased by the applied electric field, depending on the values of the donor-acceptor distance and the free energy change. These results are in good agreement with recent experimental results. As mentioned above, in linked donor-acceptors systems the luminescence intensity of the excited donor can be either increased or decreased by the electric field, depending on the values of the donor-acceptor distance and the free energy change. We are considering the possibility of applying this phenomenon to develop organic electronic devices.

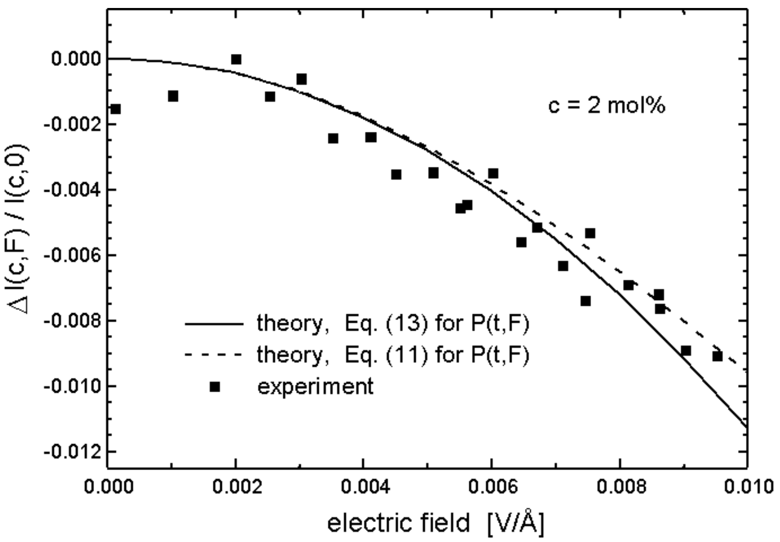


Fig. 1.1.1. Effect of an applied electric field on the luminescence intensity of the excited donor (ECZ) in the presence of acceptors (DMTP) with concentration of 2mol%. The symbols ■ show the experimental results, while the full and dashed lines show the theoretical curves.

Secondly we have presented a new theory for the charge mobility in doped polymers and succeeded in explaining the effects of the applied electric field and the temperature on the mobility almost quantitatively [5]. In these systems the charge mobility shows a strong electric field dependence. For many combinations of polymers and doped molecules the charge mobility is known to increase with increasing field strength and follow Gill's empirical rule over a wide range of field strengths (10^4 - 10^6 V/cm). It is established from the measurements on the temperature dependence of the mobility and so on that charge transport in these systems is due to hopping between doped molecules which play a role of hopping sites. According to recent experimental results, the charge mobility saturates and furthermore decreases with increasing field strength in the high electric field region. We have considered the following model to explain the above results. The dipoles of other doped molecules produce electrostatic potentials at hopping sites. The rate of charge transfer between hopping sites depends on the electrostatic potential difference between them. The Marcus equation is established for the rate of charge transfer in liquids. We have applied the Marcus equation for the rate of charge transfer between hopping sites in doped polymers and calculated the charge mobility. Our results explain Gill's empirical rule and the decrease of the mobility in the high electric field region in a unified manner (Fig. 1.1.2). The reason the mobility increases with increasing field strength as described by Gill’s empirical rule is attributed to the fact that the electrostatic potential fluctuates randomly from one hopping site to another. Furthermore the decrease of the mobility in the high electric field region can be explained on the basis of the Marcus inverted region. There is one problem in our theory. If the Marcus equation is used, the calculated charge mobility decreases too rapidly in the high electric field region, compared with the experimental results. In order to resolve this problem we are planning to calculate the charge mobility by using the equation in which the effect of intramolecular vibrations is incorporated into the Marcus equation. We are also planning to refine our theory further and establish a theory which is useful for designing the hole transport materials of electrophotography.

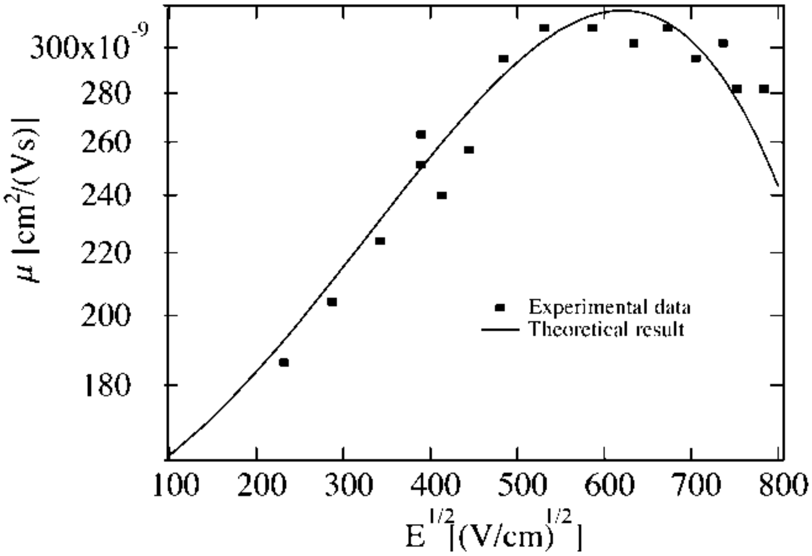


Fig. 1.1.2. Dependence of the charge mobility on the applied electric field. The symbols ■ show the experimental results by Hartensteinet al. on 17 wt% EFTP-doped polycarbonate and the full line shows the theoretical curve. The ordinate shows the mobility on a logarithmic scale, while the abscissa shows the square root of the electric field strength.

Thirdly we have proposed a new model for charge recombination process in dye-sensitized solar cells and succeeded basically in elucidating the influences of the excitation intensity, the applied electric field and the electrolyte on the charge recombination rate [6]. Electrons injected into nanocrystalline semiconductors from dye molecules by charge separation recombine with dye cations over a wide range of time scales from picoseconds to milliseconds and the kinetics of recombination is highly nonexponential. According to very recent experimental results including those of our institute, the rate of charge recombination depends on the excitation intensity. It also depends on the concentration of dark electrons that are injected into nanocrystalline semiconductors by applying an electric field or changing the composition of the electrolyte. The reason the charge recombination process occurs over a wide range of time scales is attributed to the fact that injected electrons are trapped by trap sites and that the trap depth has a distribution. We have considered the following model to explain the above results. Trap sites are distributed on the surface of semiconductor nanoparticles. The distribution of trap depths is assumed to be exponential. The rate constant for detrapping of electrons is given by an Arrhenius-type equation with the activation energy equal to the trap depth. Electrons diffuse within the conduction band of semiconductor nanoparticles after detrapping and are either trapped again by other trap sites or recombine with dye cations on the surface of the nanoparticles. As a result of these processes the distribution of electrons trapped by trap sites with different energies changes with time. We have calculated the energy distribution of trapped electrons as a function of time and on the basis of this distribution calculated the decay kinetics of dye cations due to recombination with electrons. If the concentration n_0 of electrons injected into nanocrystalline semiconductors from dye molecules by charge separation is much lower compared with the concentration N of trap sites, the survival probability $P(\tau)$ of dye cations at normalized time τ is approximately given by

$$P(\tau)^{-1} = 1 + (n_0/N)c\tau^{\alpha} \qquad (1)$$

where α is a parameter and c is given in terms of α. According to eq. (1), if one plots $P(\tau)^{-1}-1$ against τ on log-log scales, one should get a straight line. The theoretical results reproduce experimental data fairly well, as shown in Fig. 1.1.3. The influences of an applied electric field and the electrolyte on the rate of charge recombination can also be explained on the basis of the same model by taking into account the influence of dark electrons injected into nanocrystalline semiconductors by applying an electric field or changing the composition of the electrolyte. The distance an electron travels before it is trapped by the next trap depends on the ratio of the rate of trapping into trapped sites to the diffusion rate in the conduction band. In the very large limit of this ratio the electron is trapped by an immediate neighbor trap site. In this case the rate of charge recombination is controlled by the spatial diffusion of electrons. On the other hand, in the very small limit of this ratio the electron spreads over the whole space of the nanoparticle after it is detrapped. In this case the rate of charge recombination is controlled by the detrapping rate. According to our analysis, the rate of charge recombination is determined not by the spatial diffusion but by the detrapping rate of electrons. In conclusion we have elucidated the mechanism of charge recombination between dye cations and electrons injected into nanocrystalline semiconductors from dye molecules by charge separation. We are planning to refine our theory and establish a theory which is useful for designing efficient dye-sensitized solar cells.

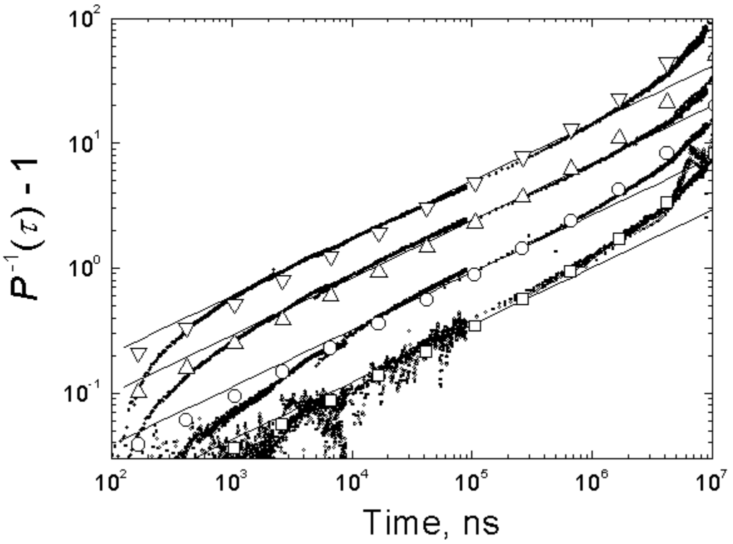


Fig. 1.1.3. Influence of the excitation intensity on the rate of charge recombination. $P(\tau)$ stands for the survival probability of $Ru(dcbpy)_2(NCS)_2$ cations adsorbed on TiO_2 nanoparticles. The points show the experimental results, while the symbols □, ○, △, ▽ show the corresponding theoretical results. The full line shows eq. (1) in the text. The excitation intensity is 0.12, 0.6, 3.5, 6 mJ/cm^2 from bottom.

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1-2. Experimental Study of Photoinduced Electron Transfer

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Purpose

Electron transfer plays important roles in photosynthetic and solar energy conversion systems. The purpose of this research is to elucidate experimentally the important physical factors that control electron transfer reactions. The basic properties of electron transfer reactions are investigated by time-resolved spectroscopy in the range from nanosecond to femtosecond. The topics studied include the distance dependence of electron transfer rate, ultrafast adiabatic electron transfer at short distances, determination of the magnitude of transfer integral of molecular complexes, control of electron transfer reactions by hydrogen bond formation, and efficiencies and dynamics of electron injection from excited dyes adsorbed on nanocrystalline semiconductor films.

Results and Future Problems

(1) Physical parameters of electron transfer

Fluorescence decay curves in the picosecond and femtosecond regions were measured by time-correlated single photon counting (TCSPC) and fluorescence upconversion, respectively. The TCSPC data were used to obtain the distance-dependence of the electron transfer rate $k(r)$ in solution. For this purpose, the transient effect in fluorescence quenching was measured and analyzed, and the parameters of electron transfer were determined. In polar solvents the slope of the $\log k(r)$ vs. r plot is almost unchanged for D-A pairs with different ΔG values, where ΔG is the free energy change of the reaction. In nonpolar solvents, the slope decreases with increasing $-\Delta G$. The strong dependence of the slope on ΔG in nonpolar solvent is attributed to the decrease in $-\Delta G$ with increasing r [1].

The upconversion measurement revealed that the decay of fluorescence of cyanoanthracenes quenched by aniline derivatives consists of two components [2]. The slower component is due to quenching by Marcus-type electron transfer. The faster component has a decay time of the order of a few hundred femtoseconds, and its intensity increases with the quencher concentration. In the case of 9-cyanoanthracene dissolved in *N,N*-dimethylaniline, the faster component decays with a decay time of ~ 210 fs which is very close to the rise-up time of the shorter wavelength region of the exciplex fluorescence found at longer wavelengths. The decay time of the faster component for several D-A pairs is roughly 200 fs, and is almost independent of ΔG of the electron transfer reaction. This shows that the quenching is due to a new type of electron transfer. This electron transfer is considered to be due to strong interaction between D and A* at short distances [3]. At such distances, the interaction between D and A* is strong enough to cause mixing of the DA* and D^+A^- states, resulting in potential curves shown by solid curves in Fig. 1.2.1. In this case, the electron transfer process $D + A^* \rightarrow D^+ + A^-$ is adiabatic in nature and becomes very fast. Since the product state is a mixture of DA* and D^+A^- states, it is the exciplex state. This type of electron transfer is considered to be important in many artificial systems, because in such systems D and A are placed close to each other to facilitate electron transfer.

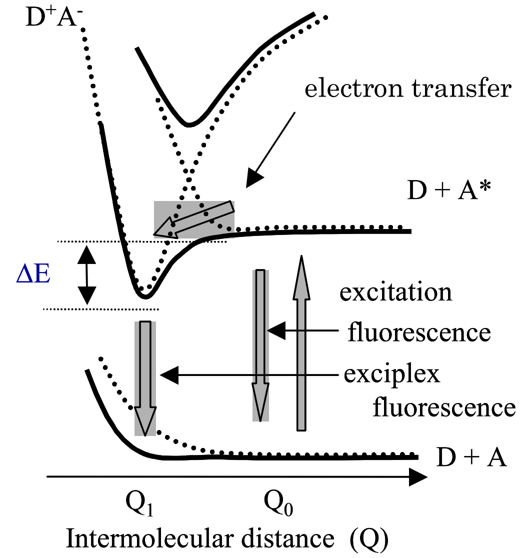
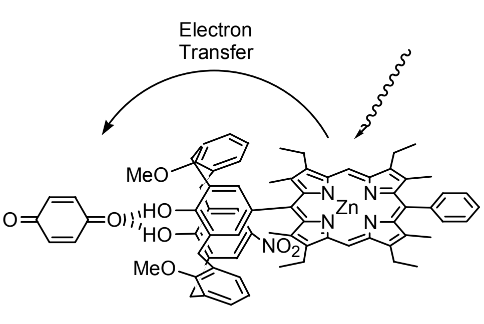


Fig. 1.2.1 Potential energy curves of a D-A pair as a function of distance

The charge transfer absorption bands of perylene excimer and triplet exciplexes between chloranil and alkylbenzenes were measured with a near infrared transient absorption spectrometer [4,5]. By analyzing the band shape of the charge transfer absorption, the transfer integral V between a neutral excited state and an ion-pair state can be estimated as 0.37 eV for perylene excimer. It is concluded from these results that the stabilization energy of perylene excimer consists of two components; exciton interaction (70 %) and charge transfer interaction (30 %). The origin of the stabilization energy of other aromatic excimers is also discussed. For triplet exciplexes between chloranil and alkylbenzenes, the charge transfer transition from a triplet excited state to an ion-pair state can be observed in the near-IR range (600-1600 nm). From the peak positions, the transfer integral V between the triplet excited state and the ion-pair state can be estimated as $V \approx 0.5$ eV.

The synthesis and characterization of a new supramolecular assembly **I**, wherein photo-induced electron transfer through non-covalent interactions may be probed, was carried out [6]. Ensemble **I** is based on supramolecular contacts between the phenolic hydroxyl groups of a calix[4]arene substituted Zn(II) metalloporphyrin donor and the carbonyl groups of a benzoquinone acceptor. Upon irradiation of the porphyrin subunit of **I** at 400 nm, intramolecular electron transfer from the Zn(II) metalloporphyrin to the benzoquinone occurs with a rate constant of $3.3 \times 10^{10}\ \mathrm{s}^{-1}$. Two phenolic hydroxyl groups of the calix[4]arene serve not only as tweezers to capture the benzoquinone by two-point hydrogen-bonding fixation, but also as useful building blocks in the construction of non-covalent electron transfer model systems. There are few systems capable of conformation-induced direct through-space electron transfer quenching. It is well known that the calix[4]arene moiety exists in four conformations in solution. These distinct conformers provide a unique opportunity for examining the two mechanisms of electron transfer, i.e. through-bond and through-space. We synthesized a new donor-acceptor system, *1,3*-alternate conformer **II**, in which calix[4] arene serves to juxtapose a pyromellitimide acceptor near the porphyrin photo-donor plane [7]. This provides a new supramolecular system in which through-space electron transfer is observed upon photoexcitation. By femtosecond time-resolved transient absorption studies, we determined the rate constants of electron transfer in **II** as: $k_{\mathrm{CS}} = 7.3 \times 10^{10}\ \mathrm{s}^{-1}$ and : $k_{\mathrm{CR}} = 3.1 \times 10^{9}\ \mathrm{s}^{-1}$. We conclude that a direct through-space (-solvent) process accounts for the observed electron transfer quenching process in this calixarene based model system for the first time. Although many obstacles still exist, these observations of controlling the pathways of electron transfer events might pave way towards the design of novel energy conversion systems suitable for optical molecular wire and storage functions at the single-molecule level.



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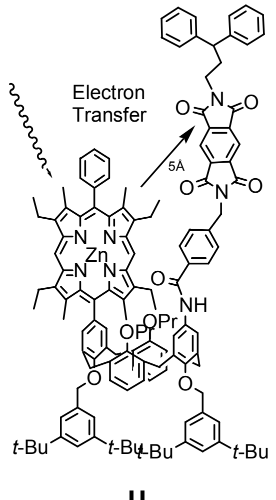

II

(2) Electron injection from dyes adsorbed on nanocrystalline semiconductors

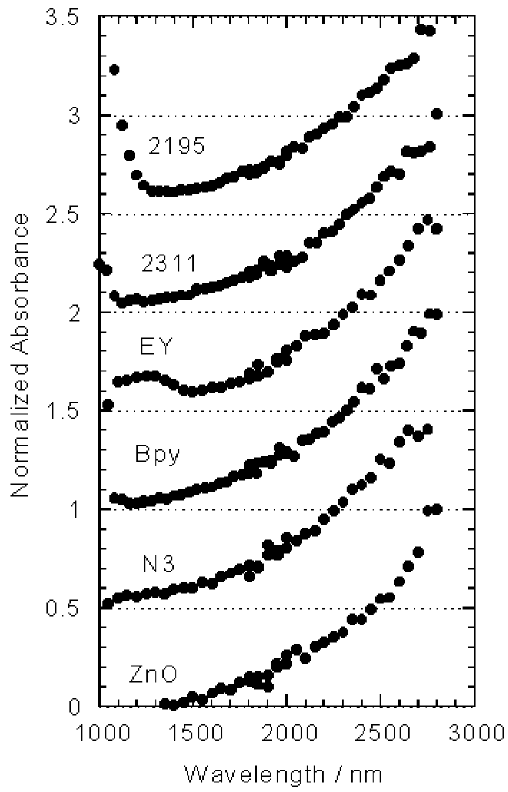

Fig. 1.2.2 Near IR absorption of injected electrons

By nanosecond transient absorption spectroscopy, the near-IR absorption spectrum of electrons injected from excited sensitizer dyes to nanocrystalline ZnO films was observed (Fig. 1.2.2). The absorbance due to injected electrons in ZnO films sharply increases with wavelength. This absorption can be assigned to the optical transition of conducting electrons to higher energy levels of the conduction band. From the measurements using various sensitizer dyes, the relative efficiencies of electron injection can be obtained as a function of free energy change ΔG of electron injection. The efficiency increases with increasing $-\Delta G$ below 0.2 eV, and above that, it seems to remain constant. This behavior can be explained by a model based on the non-adiabatic electron transfer theory including site heterogeneity.

In a shorter time range, the absorption by electrons is expected to show relaxation behaviors due to generation and trapping of electrons. Such behavior was observed for ZnO films in the picosecond range. After analyzing the result, we tentatively conclude that cation-electron pairs are produced initially, and then the electrons relax to conducting electrons. We are trying to measure the absorption band due to injected electrons in TiO_2, which is the most promising material for high performance solar cells.

Studies were also made on the effect of molecular structure of the sensitizer on the solar cell performance. It was reported that the number of anchoring carboxyl groups affects the solar cell performance of dye-sensitized nanocrystalline TiO_2 solar cells using Ru-phenanthroline complexes as sensitizing dyes. We found, using transient absorption spectroscopy, that the electron injection efficiency is strongly affected by the number of carboxyl groups in the sensitizer molecule [8]. From the analysis of the time profile of the transient absorption, we conclude that the low efficiency of Ru-phenanthroline complexes having one carboxyl group is due to the presence of inactive dye molecules on the TiO_2

surface, which are not effective for electron injection. It is also known that the cells using Ru dyes adsorbed on ZnO show lower performance compared to those with the same dyes on TiO_2. It was shown that the Ru dyes form aggregates with Zn ions and electron injection is hindered by the aggregates.

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1-3. Laser Control of Photochemical Reactions

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Purpose

The purpose of this group is to develop selective photochemistry using lasers, which will contribute to the development of a new chemical process that discharges the lowest amount of byproduct. There are certain merits of using photochemical process to realize selective chemistry. We can excite the targeted molecule to a special excited state with lasers. Thus we may also expect to break only the targeted bond by monochromatic lasers with the precision of a scalpel. However, despite the efforts of laser chemists since the discovery of the laser, there has been only one example of this kind of selective photochemistry concerning photodissociation of CH_2IBr by Butler et al. [1]. The weakest bond always dissociates in the photochemistry of other molecules due to fast energy relaxation or strong intra-molecular interactions in excited electronic states.

Many laser chemists have put a great deal of effort into overcoming these problems. In the COE project, we proposed two approaches of controlling photochemical process by lasers: 1) A coherent control of photo-dissociation of simple molecules in the frequency domain, and 2) Mode selective infrared predissociation of the hydrogen bonds.

Results and Future Problems

(1) Coherent control of a laser reaction

The coherent control of chemical reaction using nanosecond laser pulse has been intensely investigated theoretically and experimentally. The coherent control proposed by Shapiro and co-workers is based on the interference effect of simultaneous optical excitation between one photon transition and three-photon transition [2]. In general, the quantum interference between one-photon transition and three-photon transition has been used from the limitation of the selection rule in the optical transition. It has been proposed theoretically that the branching ratio in photodissociation of IBr ($IBr \rightarrow I+Br$ or $I+Br^*$) can be controlled by varying the relative phase between two simultaneously applied lasers of wavelengths of ~500nm and ~1500nm [3]. At first, we performed the experiment of ω-3ω coherent control of photodissociation of IBr to confirm the theoretical prediction [4]. We produced the 100 mJ, 10nsec pulse laser at 1590nm using the Nd:YAG laser and a Raman shifter. However, we could not observe the three-photon dissociation of IBr because the transition probability of three-photon absorption is much smaller (10^{-4}-10^{-6}) than that of one-photon transition. To overcome this problem, it is necessary to decrease the order of optical transition; that is, to use the excitation via the two-photon transition that has larger transition probability than that of three-photon transition. However, there is the problem regarding the selection rule of two-photon transition, as it is different from that of one-photon transition. Namely, we cannot excite the same state by one-photon excitation and two-photon excitation. Our strategies are as follows: (1) the ω-2ω coherent control using stark mixing induced by strong laser fields(2) the ω-2ω coherent control in the bigger molecular systems which have the symmetry breaking. We have performed the ω-2ω coherent control in the photodissociation of IBr and allyl iodide, and we have succeeded in observing the interference effect in the dissociated yield of excited iodine atoms. Fig. 1.3.1 shows the absorption spectrum and energy diagram of IBr. We used a 560nm laser and its SHG, and observed the quantum interference effect in the efficiency of the production of I atom (Fig. 1.3.2).

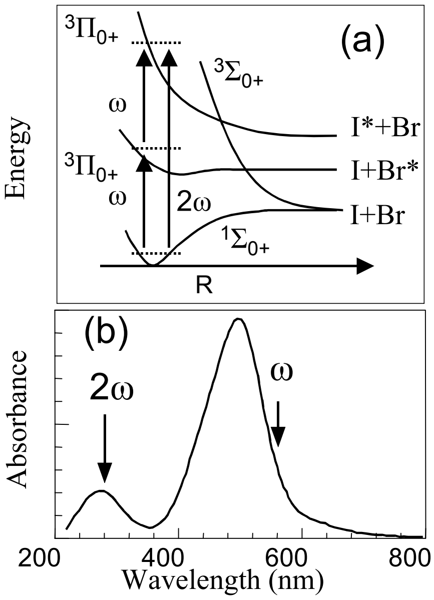

Fig. 1.3.1. (a) The energy diagram of IBr. (b) The absorption spectrum of IBr. Arrows show the wavelength of the lasers.

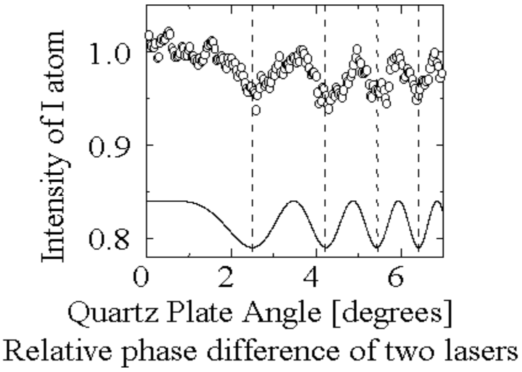

Fig. 1.3.2. The dissociation yield of spin-orbit excited iodine atoms as a function of quartz plate rotation angle θ. The quartz plate was rotated to vary the relative phase between fundamental and second-harmonic light. The solid curve shows the function of the form $A+B\cos(\Delta\Phi+\Phi_0)$ where A, B and Φ_0 are the adjustable parameters.

This method includes the possibility of more general coherent control: (1) High power lasers are not needed because the order of optical transition is reduced. (2) It is easier to produce the second harmonic light than third harmonic light.

(2) Bond selective photodissociation of hydrogen bonds

It is well known that the infrared band of X-H stretching mode shows a large red shift and band broadening when it makes a hydrogen bond with the lone pair or π-electron. This broadening seems to originate from the fast energy transfer from the X-H stretching vibration to the hydrogen bonds. If so, since the bandwidth of the infrared band ranges as large as 50-100cm^{-1}, the lifetime of the excited state should be less than 1ps if the interaction is strong. This relaxation lifetime is much faster than the usual IVR lifetime (~10ps), and as in Fig. 1.3.3 we can expect to control the fission of the hydrogen bond by exciting the XH stretching vibration adjacent to the hydrogen bond.

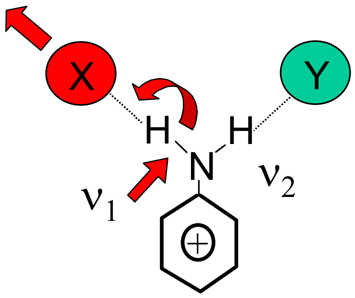

Fig.1.3.3 Mode selective reaction of aniline cluster.

We adopted the infrared predissociation reactions of the ternary aniline cluster cations, aniline-water-aromatic (benzene or pyrrole), as the model system [5,6]. This cluster dissociates and emits water or aromatic when it absorbs an infrared photon. The branching ratio R=(aniline-water$^+$/aniline-aromatic$^+$) has been determined using TOF mass spectroscopic technique. Fig. 1.3.4 shows the results for the aniline-water-pyrrole cluster cation. This cluster has four infrared bands at 3313, 3515, 3637 and 3721cm^{-1} in the region of 3800-3200cm^{-1}. They were assigned to the stretching vibration of NH of an aniline interacting with pyrrole, the NH stretching vibration of pyrrole, the symmetric and anti-symmetric stretching vibration of water whose oxygen is interacting with NH of aniline, respectively.

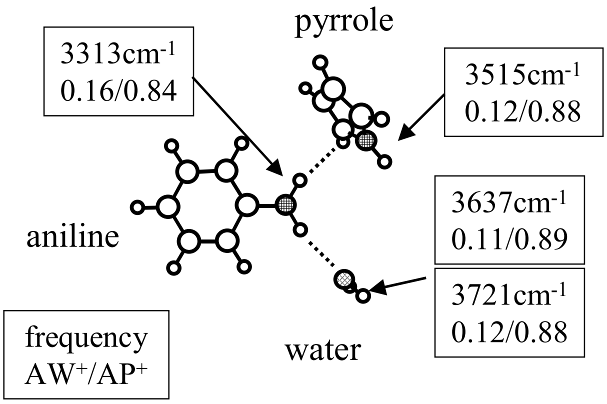

Fig. 1.3.4. The branching ratio of the infrared predissociation of the hydrogen bond of aniline-water-pyrrole cluster cation.

The branching ratio of the reaction observed for the NH stretching vibration interacting with pyrrole (0.16/0.84) is definitely larger than those observed for the OH stretching vibrations (0.11/0.89, 0.12/0.88) and NH stretching vibration of pyrrole (0.12/0.88). The differences between the first one and the others are larger than the experimental error (0.02). Similar result was also observed for the aniline-water-benzene cation. This result confirms the idea that the ultra fast energy transfer from the NH stretching vibration to the intermolecular vibration accelerates the infrared predissociation of the hydrogen bond.

We have shown that the infrared predissociation reaction of the hydrogen bond clusters depends on the vibrational mode. This is the second example of the mode selective photoreaction. The infrared predissociation of the hydrogen bond seems to be one general solution for the old dream of laser chemist, the mode selective laser chemistry, because the hydrogen bond is widely observed in various substances, and to control the hydrogen bond is to control the function of them.

(3) Infrared cavity ring-down spectroscopy

An infrared cavity ring-down spectrometer (IR-CRD) was developed as a sensitive and wide-band probe for photochemical reactions in gas phase. The bromine atom and iodine atom produced in the photolysis of IBr and CH_3I, respectively, show population inversion in their spin sub-states. In the IR-CRD experiments to probe these atoms, we have found that laser oscillation occurs in a ring-down cavity, and large enhancement in detection of these atoms has been attained (similar to intra-cavity laser spectroscopy). We can expect that this technique can be applied to photochemical systems which produce populationally inverted atoms and radicals. Fig. 1.3.5. shows the example of the observation of Br* by IR-CRD method.

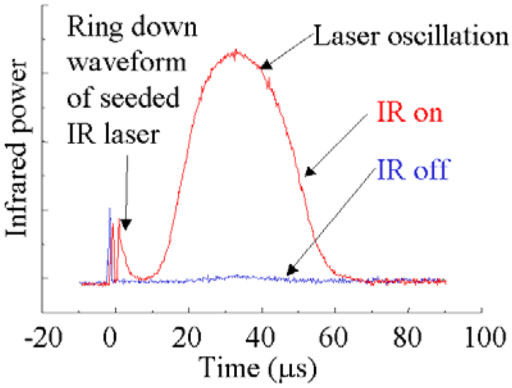

Fig. 1.3.5. Cavity ring down form of Br produced by the photodissociation of Ibr.

It is well-known that methyl iodide clusters produce the I_2 molecule in the UV photolysis, suggesting the proximity of iodine atoms in the clusters. Since there has been a lack of direct evidence of those structures, we have measured infrared spectra of methyl iodide clusters in the C-H stretching region. By combining the spectra with matrix IR spectra and quantum chemical calculations, we showed two stable isomers for methyl iodide dimer. The photodissociation of this cluster seems to be the next candidate that gives the mode selective photochemistry.

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2. Study of Light Energy Conversion

2-1. Research and Development of High Performance Dye-sensitized Solar Cells

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Purpose

The development of renewable energy sources is one of the most important issues in the world today. In order to establish the technology for effective solar energy utilization, we have been working to discover the unit process for photo-energy conversion and the development of molecular level devices, such as a novel type of solar cell that mimics a photosynthesis reaction center.

Results and Future Problems

(1) Development of dye-sensitized solar cells:

We focused our research on the development of new sensitizing dyes and the optimization of metal oxide semiconductor electrodes and electrolytes. Furthermore, we concentrate the molecular design of transition metal complexes so as to match their energy levels to those of other cell components. To improve the solar energy conversion efficiency, the tuning of energy levels of excited states and ground state is quite important. The metal-to-ligand charge transfer (MLCT) is the principal electron transition event for visible light energy utilization because of its intense absorption in visible range; it can be controlled by tuning the energy levels of the metal complexes.

(2) Development of metal complex sensitizers

a)Tuning of LUMO energy level: Quinoline based Ru(II) polypyridyl sensitizers

The π* energy levels of diimine ligands decreases in the order of 4,4’-dicarboxy-2,2’-bipyridine (dcbpy) > 4-carboxy-2-[2’-(4’-carboxypyridyl)]-quinoline (dcpq) > 4,4’-dicarboxy-2,2’-biquinoline (dcbiq), according to the degree of resonance (Fig.2.1.1). When we synthesized $RuL_2(NCS)_2$ type complexes, the absorption maximum by MLCT transition shifted from 532 nm to 627 nm. While maintaining ground state reduction potential nearly equal, the excited state oxidation potential estimated from the emission spectra was shifted more positively. Although $Ru(dcbiq)_2(NCS)_2$ shows an absorption edge at near IR region, little photoelectrochemical response was observed when anchored to TiO_2 nanocrystalline surface [1,2]. On the other hand, $Ru(dcbpy)_2(NCS)_2$ and $Ru(dcpq)_2(NCS)_2$

showed a large photoelectrochemical response toward visible light irradiation. Especially, $Ru(dcpq)_2(NCS)_2$ showed sensitization up to near IR region, which has rarely been achieved [3]. The oxidation potential of Ru(dcbiq)2(NCS)2 in excited state is less negative compared to the Fermi level of TiO_2, on the other hand, the oxidation potentials of both $Ru(dcpq)_2(NCS)_2$ and $Ru(dcbpy)_2(NCS)_2$ have enough driving force to inject an electron from the excited states to the conduction band of TiO_2.

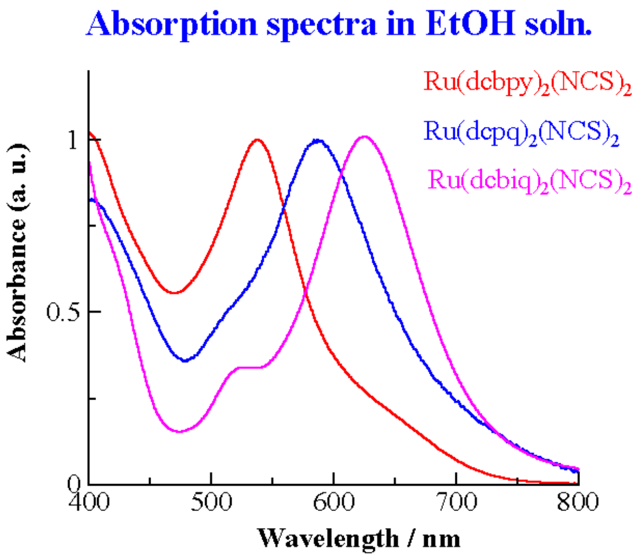
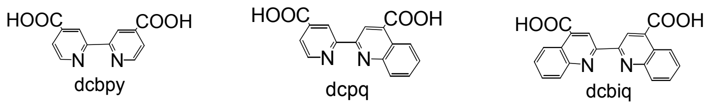


Fig. 2.1.1 Absorption spectra of $RuL_2(NCS)_2$ in EtOH.

b)Tuning of HOMO energy level: Dithiolate and diketonate-based Ru(II) polypyridyl sensitizers

Near IR response can be also be gained by upward shifting of the ruthenium t_{2g} (HOMO) energy levels. One of the most successful sensitizing complexes is $Ru(dcbpy)_2X_2$, where X = NCS. Ligand X can be replaced by another type of ligand with electron donating character. We have examined a series of Ru(dcbpy $)_2X_2$, where X_2 is one of the three dithiolates, quinoxaline dithiolate (qdt), ethyl-2-cyano-3,3-dimercaptoacrylate (ecda) and 3,4-toluene dithiolate (tdt). The absorption showed the peak maximum of MLCT from 670 nm to 517 nm in the order of $Ru(dcbpy)_2(tdt)$ > $Ru(dcbpy)_2(ecda)$ > $Ru(dcbpy)_2(qdt)$. The results reflect the electron donating character of dithiolates. The electrochemical responses of sensitized electrodes were in the order of $Ru(dcbpy)_2(qdt)$> $Ru(dcbpy)_2(ecda)$ > $Ru(dcbpy)_2(tdt)$. This result can be explained from the fact that the reduction potential of oxidized $Ru(dcbpy)_2(tdt)$ in the ground state is not positive enough to receive an electron from iodide [4] (Fig. 2.1.2).



dcbpy dcpq dcbiq

structures of diimine ligands

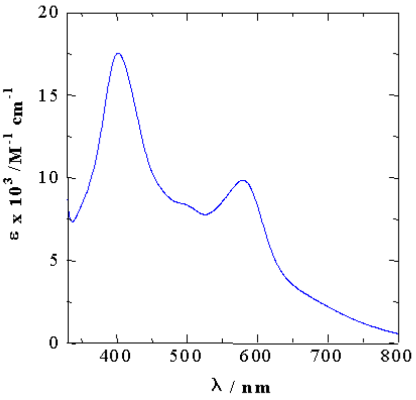
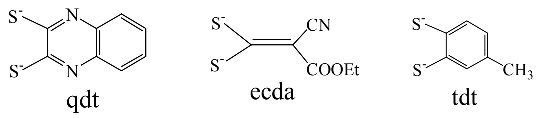


Fig. 2.1.2. Absorption spectra of Ru(dcbpy)$_2$(qdt) in EtOH/MeOH (4:1)

When dithiolate is replaced with β-diketonate, the complex showed superior absorption spectrum in red light region to that of thiocyanate complex [5]. Ru(tctpy)(NCS)$_3$, where tctpy is 4,4',4"-tricarboxy-2,2':6',2"-terpyridine, has been reported as a sensitizer for a longer wavelength region of light. We replaced two of the three NCS ligands with β-diketonate. Electron donating character can be tuned by changing the substitutents of diketones. So far, Ru(tctpy)(tfac)(NCS) showed the best performance, where tfac is 1,1,1-trifluoro-pentane-2,4-dionate, showing larger light absorption and photoelectrochemical response than corresponding thiocyanato complex between 720 nm and 900 nm [6] (Fig. 2.1.3).



qdt ecda tdt

structures of dithiolate ligands

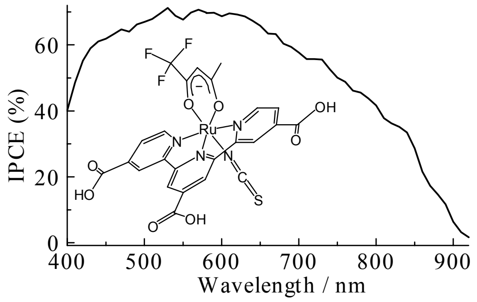


Fig. 2.1.3 Photoelectrochemical response of Ru(tctpy)(tfac)

(3) Development of organic dye sensitizers

Although the absorption area is narrower than metal complex dyes, organic dyes have advantages from an economical point of view. Various organic dyes such as merocyanin [7], coumarin [8], and styryl dyes were investigated. Ultimately, coumarin dyes were found to be very promising. Further molecular design for extension of absorption range, and conversion efficiency (up to 6% conversion efficiency was achieved), which is the highest conversion efficiency as an organic dye-sensitized TiO_2 solar cell reported thus far.

Systematic tuning of the LUMO and HOMO energy levels of ruthenium polypyridyl complexes achieved very efficient sensitization of nanocrystalline TiO_2 solar cells over the entire visible range, extending into the near IR region. To further improve the efficiency, dye adsorption condition onto semiconductor films and the combination of redox couple in the electrolytes should be optimized.

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2-2. Research and Development in Artificial Photosynthetic Technology

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Purpose

The problem of global warming has become a more severe issue than in the past; it is mainly caused by an excessive amount of CO_2 emission into the atmosphere. The development of clean energy is essential to establish the sustainable development of mankind in the 21st century. Solar light energy is absolutely clean and inexhaustible; however, we do not yet fully utilize this vast source of clean energy as one of our energy supply resources. Therefore, development of a new technology for effective utilization of solar light has become increasingly essential.

From the point of this view, we are conducting research and development in artificial photosynthetic technology, which follows the natural photosynthetic process in plants. Our first research objective is the development of efficient oxide semiconductor photocatalysts and a reaction process for stoichiometrical water splitting into H_2, a clean energy source that is carbon-free; and O_2 under visible light irradiation taking into consideration that approximately half of solar light is visible light energy. The second objective is the development of homogeneous catalysts, binuclear organometallic complexes which consist of both a light harvest center and a CO_2 reduction center for photo-reductive fixation of CO_2 under visible light irradiation. We are striving toward the development of practical photocatalytic reaction processes, based on the accumulated fruitful knowledge in optimization as well as clarification of light energy conversion steps in the overalll process.

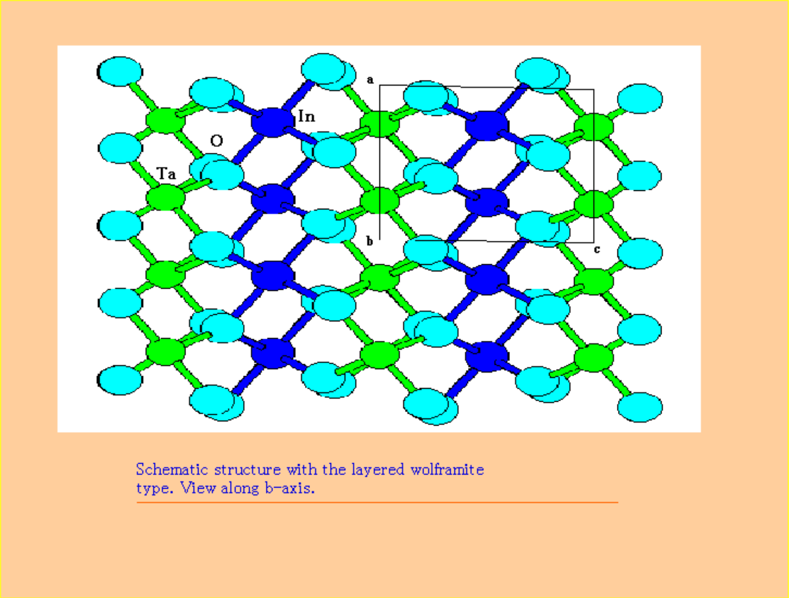
Results and Future Problems

(1) Development of one-step water splitting photocatalysts under visible light irradiation

Oxide semiconductor photocatalysts, which are enable to split water into H_2 and O_2 stoichiometrically under visible light irradiation, have not been developed so far, though various kinds of UV-light responding photocatalysts have been reported [1, 2]. We have focused on mixed oxide semiconductor materials, which have flexibility in changing band structures such as band gap energy, valence and conduction bands. As a result of wide screening of oxide semiconductor materials, mixed oxides having the ABO_4 structure, such as $BiNbO_4$ [7], $InNbO_4$ and $InTaO_4$ [8], as well as the $A_2B_2O_7$ structure, such as Bi_2InNbO_7 [3],

Bi_2AlNb_7 [4] and others [5,6], were found to have visible light responding properties. Particularly, an NiOx supported $InTaO_4$ photocatalyst was found to show water splitting ability into H_2 and O_2 stoichiometrically under visible light irradiation. This is the first example of a visible light water splitting photocatalyst in the world. Furthermore, the lattice substitution in part by other transition metals influenced the photocatalytic ability significantly [10]. For example, the photocatalytic activity of NiOx supported $In_{0.9}Ni_{0.1}TaO_4$ photocatalyst increased about 8 times compared with non-substituted $InTaO_4$ photocatalyst [9]. The improvement of photocatalytic activity was ascribed to both the band gap reduction and a better charge separation by small distortion of lattice structure by Ni substitution. Fig. 2.2.1 shows the crystal structure of $InTaO_4$. Fig. 2.2.2 shows visible light absorption properties of $InTaO_4$ and the $In_{0.9}Ni_{0.1}TaO_4$ photocatalyst. Fig. 2.2.3 shows the reaction time course of H_2 and O_2 evolution from pure liquid water by photocatalytic water splitting using NiOx supported $In_{0.9}Ni_{0.1}TaO_4$ photocatalyst under visible light irradiation.

The improvement of photocatalytic activity by an increase of catalyst surface area as well as a visible light absorption area up to 700 nm is required as the future subject for the practical application of this system.



Schematic structure with the layered wolframite type. View along b-axis.

Fig. 2.2.1. Crystal structure of $InTaO_4$

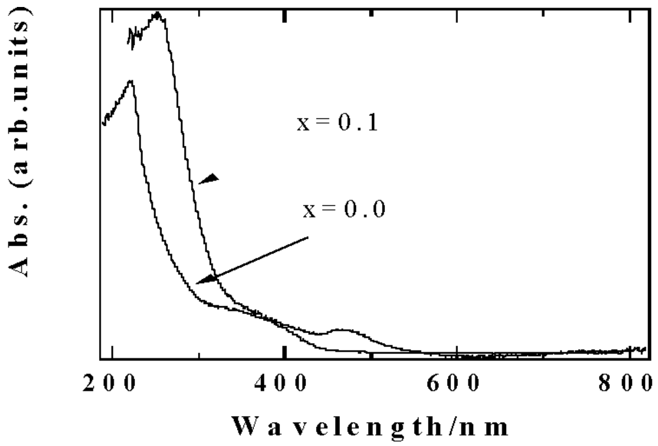


Fig. 2.2.2. UV-vis diffuse reflectance spectra of $In_{1-x}Ni_xTaO_4$ (X=0.0 and 0.1)

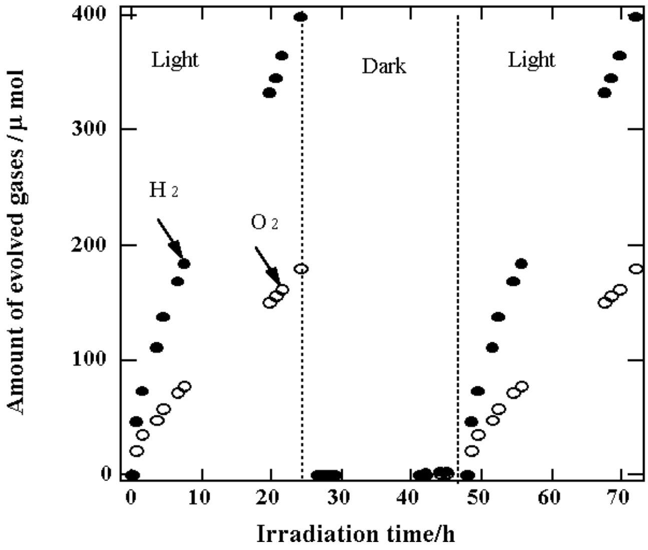


Fig. 2.2.3. Reaction time course of the photocatalytic splitting of water into H_2 and O_2 on $NiOx/In_{0.9}Ni_{0.1}TaO_4$ under visible light irradiation (サ >420nm).

(2) Development of two-step water splitting photocatalytic process

As an alternative water splitting system, we propose the combined system of water oxidation reaction and water reduction reaction connected by an electron transport mediator such as a redox reagent, which is a mimicking system of Z-scheme in natural photosynthesis. Fig. 2.2.4 shows the proposed reaction mechanism of this two-step water splitting process. The merit of this process is that the screening for water oxidation photocatalysts as well as water reduction photocatalysts is easier than that of the one-step photocatalyst process. However, it is not easy to ascertain the optimum redox mediator, which connects two reaction sites. As the result of a wide screening test, we found that Pt-WO_3 photocatalyst [11, 12, 13] and Cr and Ta co-doped $SrTiO_3$ photocatalyst [14] work well for water oxidation and reduction, respectively under visible light irradiation. Furthermore, it was proved that I^-/IO_3^- redox couple is useful as the electron mediator [15, 16]. By the combination of these two photocatalysts and I^-/IO_3^- redox couple, the two-step water splitting photocatalytic process under visible light irradiation was demonstrated for the first time in history. Fig. 2.2.5 shows the reaction time course of H_2 and O_2 evolution from pure liquid water by this two-step water splitting system. The photocatalytic activity of this process is still low at this time.. Therefore, the optimization of reaction conditions and the screening of new efficient photocatalytic materials will be future subjects of research.

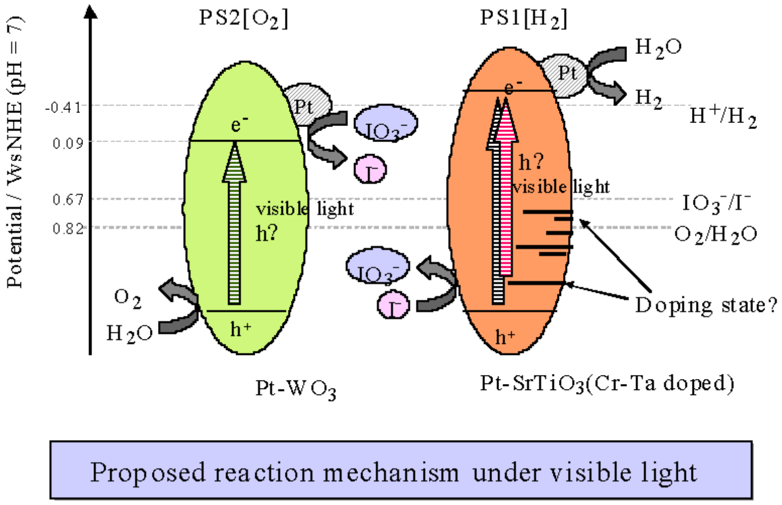


Fig. 2.2.4 Speculated reaction mechanism for water splitting using a mixture of Pt-SrtiO$_3$, Pt-WO$_3$ and NaI aqueous solution.

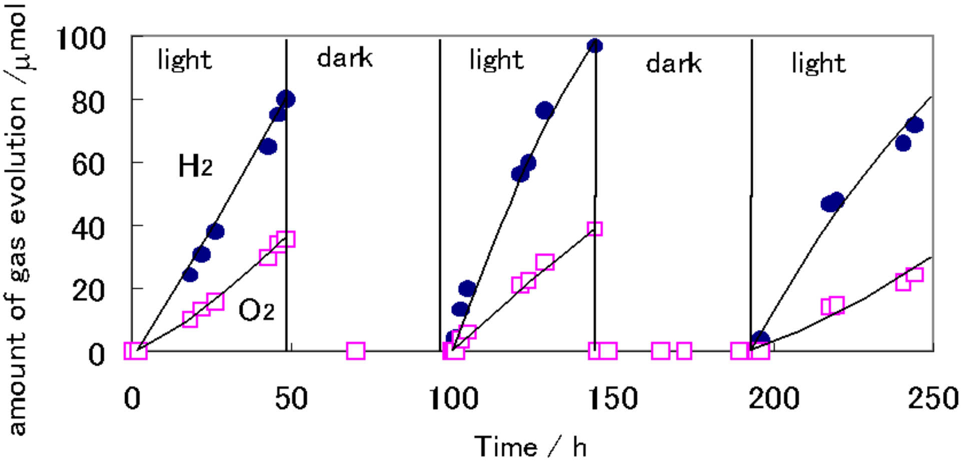


Fig. 2.2.5. Reaction time course of the photocatalytic splitting of water into H_2 and O_2 under visible light ($\lambda > 420$ nm). Pt-WO$_3$ and Pt-SrTiO$_3$(Cr and Ta doped) were suspended in the NaI aqueous solution.

(3) Study on photo-reductive fixation of CO_2

As the homogeneous photocatalyst for the photo-reductive fixation of CO_2, Ru-Co dinuclear complexes having bisphenanthrtoline bridging ligands were newly synthesized. It is estimated that the Ru center acts as a visible light harvesting site and the Co center acts as a CO_2 reduction site. Carbon dioxide was reduced to CO using this Ru-Co dinuclear complex photocatalyst and a sacrificial reagent under visible light irradiation. [17] The selectivity of produced CO to by-product H_2 was higher than the mixed catalyst system of Ru mononuclear complex and Co mononuclear complex. The catalytic activity of this system was still low; therefore, the improvement of catalytic activity is essential. Dye-sensitized solid catalysts might be one of efficient photocatalysts for photo-reductive fixation of CO_2.

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3. Study of Laser Induced Reaction

3-1. Research and Development of Laser-Induced Materials Processing

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Purpose

On the basis of photoreaction control techniques related to the interaction of photons and matter, we are developing new methods to synthesize valuable chemicals without unwanted byproducts, as well as new processes to prepare highly functional materials by highly selective photochemical reactions and photophysical changes. We study the chemical and physical changes that take place in various materials, such as organic and polymeric compounds, silica glasses, and inorganic compounds, under extreme and specific conditions by irradiation with ultraviolet and vacuum ultraviolet light. In particular, our objective is the development of new material processing to improve material performance, to add novel functions to materials, and to synthesize functional thin films that cannot be obtained by conventional methods [8,10-12].

Results and Future Problems

Reactive species are important chemical species that can be applied to various material processing. In our research on the preparation of reactive species by high-intensity pulsed UV laser irradiation of organic molecules in low-temperature matrixes, we have succeeded in the formation and direct spectroscopic observation of several unusual intermediates such as benzdiyne [1-3], naphthyne [4,5], enetetrayne [4,6], and nitrene [7-9] species, which are interesting reactive intermediates from the viewpoints of both fundamental and applied chemistry (Figs. 3.1.1 and 3.1.2). The generation of these species was attained by means of wavelength-selective irradiation using various lasers, and confirmed by good correspondence between experimental and theoretical results. Density functional theory (DFT) method was mainly used to obtain geometries of the intermediates and their optical spectra for vibrational and electronic transitions. We have also developed a new processing technique to apply reactive intermediates to the surface modification of polymer films using the reactivity of the intermediate with the polymer surface [8,10-12].

Taking advantage of an unexpected unique finding on a mechanistic study of polymer ablation, we have developed a novel one-step method to microfabricate a silica glass plate using laser-induced backside wet etching (LIBWE) upon irradiation with a ns-pulsed UV excimer laser (Figs. 3.1.3 and 3.1.4) [13-17]. Well-defined grating and grid micropatterns with 1-10 μm resolution were fabricated on a silica glass by laser-induced backside wet etching (LIBWE) with an aqueous dye solution (Figs. 3.1.5 and 3.1.6). This method originated from a fundamental photophysical process of organic molecules excited by laser irradiation (Fig. 3.1.4). The advantages of our LIBWE method are as follows: (i) micro-fabrication without debris and cracks formation at an atmospheric pressure (Figs. 3.1.5)(ii) simplification of pre-/post-treatment for a target substrate(iii) large area irradiation with a conventional ns-excimer laser through a mask projection (Fig. 3.1.6). In addition, we were able to select dye solution from various liquids of pyrene/acetone, pyrene/THF, toluene, and pyranine/water upon the irradiation with KrF and XeCl excimer lasers. To understand the etching mechanism, the formation and propagation of shockwave and bubble were monitored by time-resolved optical microscopy at the interface between the silica glass and the toluene solution during the etching process. Transient high-pressure as well as high-temperature generated by UV laser irradiation plays a key role in the etching process.

During the first five-year stage of the COE project, we have studied a variety of laser-induced reactions for many types of materials under various conditions in order to develop new laser-induced methods for synthesis and material processing. Our laser material processing techniques are based on photochemical control under specified reaction conditions corresponding to the properties of the materials. We are aiming to create material processes derived from new phenomena that have been reported in fundamental studies on the interactions between photons and matter. We will continue systematic studies to the goal of embodying the results obtained into further sophisticated material processing techniques.

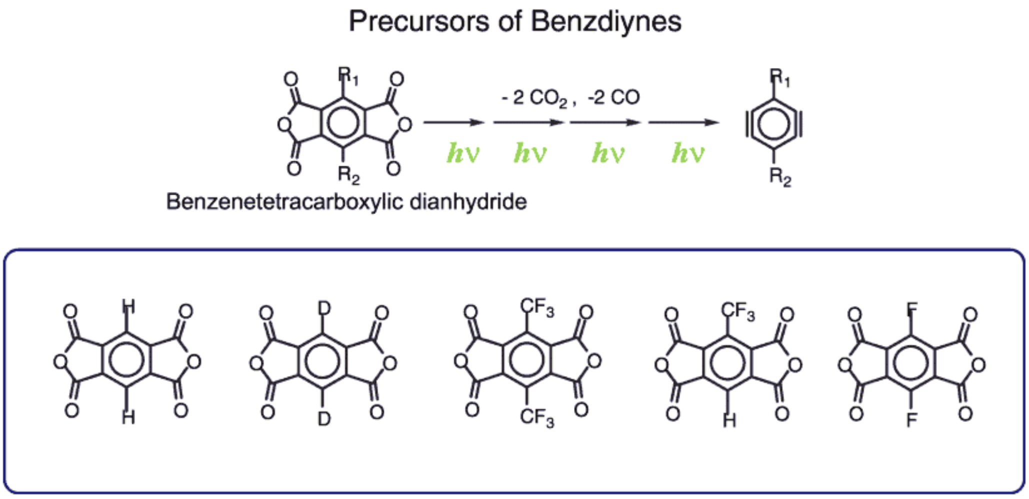

Fig.3.1.1 Reaction scheme for the generation of benzdiynes and chemical structures of five precursors.

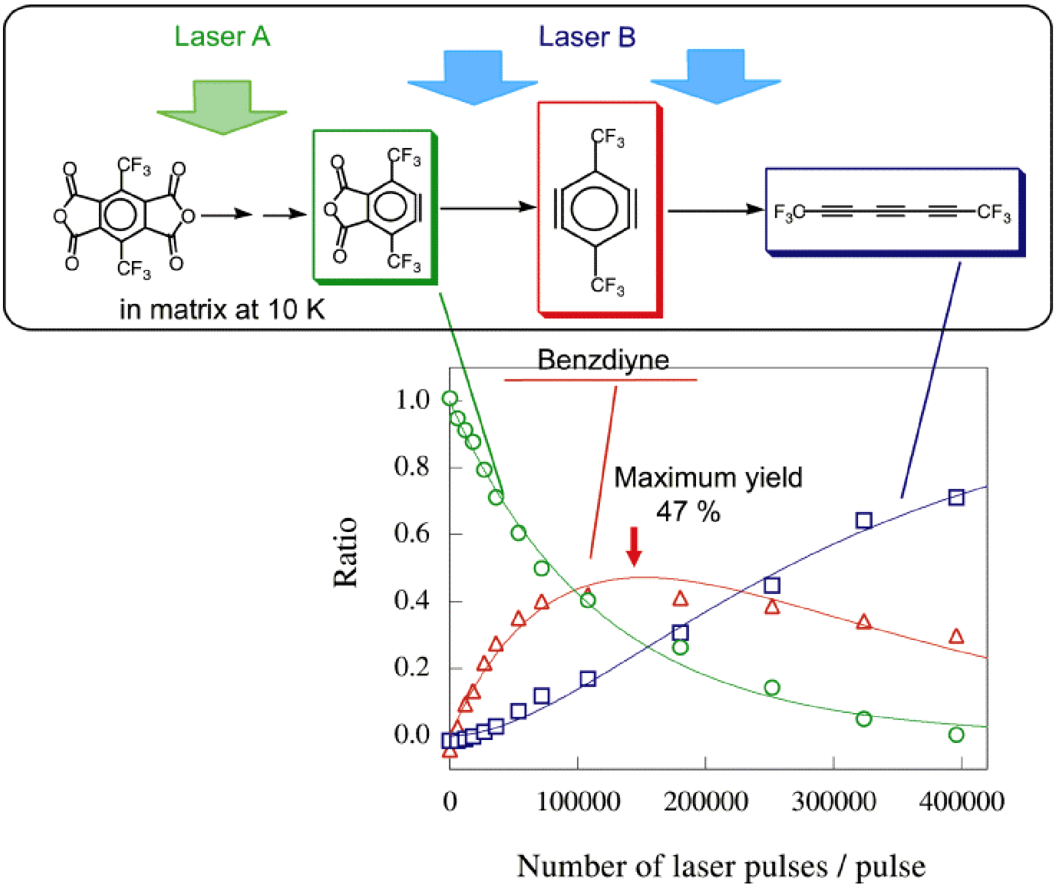

Fig.3.1.2. Benzdiyne formation with a high yield upon wavelength-selective laser irradiation.

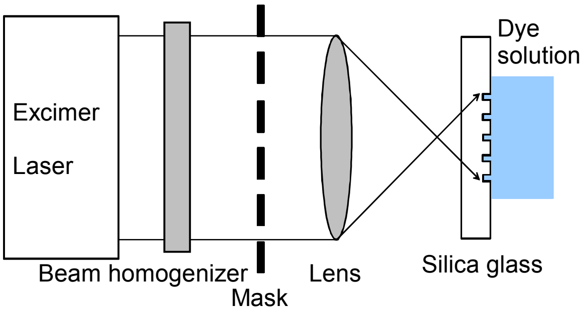

Fig.3.1.3 Experimental setup for LIBWE method.

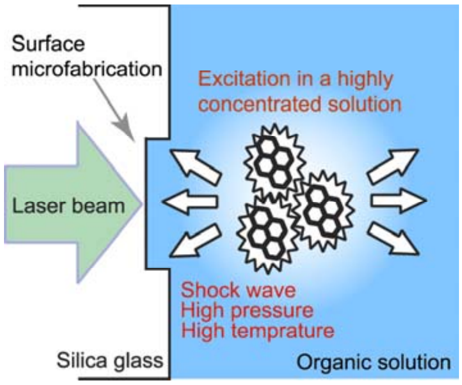

Fig.3.1.4 Laser ablation of a highly concentrated dye solution.

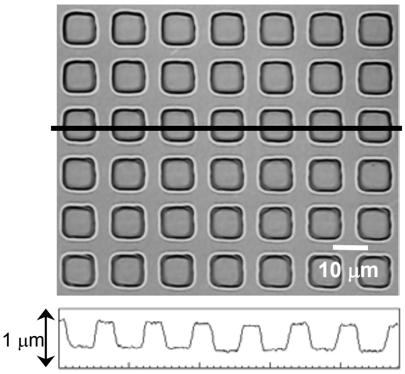

Fig. 3.1.5 Confocal scanning laser micrographs of silica glass; top-view (upper) and cross-section (lower).

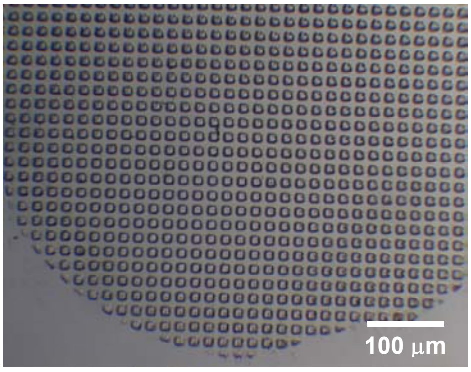

Fig.3.1.6 Optical micrograph of silica glass.

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3-2. Laser Processing for Nanomaterial Preparation

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T. Tsuchiya (Institute for Materials & Chemical Process)

Purpose

Crystal structure control and low temperature processing are important issues in material preparation aiming for functional applications, especially with oxide-based materials. For instance, the formation of perovskite complex oxide phase generally requires temperatures of approximately over 500 °C, and thus it is considered difficult to integrate the films with silicon monolithic circuits. Meanwhile nanocomposite materials often require independent crystal structure control of either matrix or dispersing elements to obtain better performance of properties.

Laser processing can be considered as a powerful tool for the crystal structure control of intricate materials like complex oxides and nanocomposite materials, because lasers, the ultraviolet pulse laser in particular, have advantages of fewer thermal effects and high energetic state confined in a small space for a brief amount of time. Here we focus on the perovskite complex oxide preparation from two types of approaches using lasers: the coating photolysis process and pulsed laser deposition (PLD) under off-axis configuration, both of which have been developed through the COE project. The former process is a film preparation technique done by the decomposition of metallorganic coating using a laser. This process evidently maintains the atomic ratios of the metallic components in the perovskite structure during processing. On the other hand, the latter process will also maintain the target composition because of the well-known compositional transcription of the target to the films, according to the study on the preparation of oxide superconductors using the laser ablation technique. Furthermore, the deposition of crystallized nanoparticles grown in the plume confined by the high pressure ambient greatly lower the processing temperature. Here we report our attempts to form crystalline thin films of complex oxides using both techniques without extra heating of the substrate.

Results and Future Problems

(1) Coating Photolysis Process for Epitaxial Perovskite Thin Film Preparation

We developed a new method, which we refer to as the "coating photolysis process" [1-3] for the preparation of metal oxide films. By extending this technique, we succeeded in the preparation of epitaxial PZT ($PbZr_{0.5}Ti_{0.5}O_3$) films using excimer laser irradiation without heat-treatment.

Homogeneous starting solution was prepared by mixing commercially available zirconium 2-ethylhexanoate oxide mineral spirits solution, lead 2-ethylhexanoate and titanium 2-ethyl-1-hexanolate in toluene with the molar ratio of Pb:Zr:Ti = 2:1:1. This solution was spin-coated onto single-crystal $SrTiO_3$ (001) substrate at 4000 rpm for 24 s. The coated films were dried at 150 °C for 20 min in atmosphere to eliminate the solvent, and directly irradiated by an unfocused ArF excimer laser (Lambda Physik, Compex 110) at repetition rates of 10 Hz for 1 min. The laser fluence on the films was changed from 40 to 80 mJ/cm^2. Crystallinity and epitaxy of the films were examined by X-ray diffraction (XRD; MAC Science, MXP^{3A}) θ-2θ scanning, rocking curve analysis and pole-figure analysis.

Fig. 3.2.1 (a)-(e) shows XRD θ-2θ scans for obtained films on STO (100) prepared by the coating photolysis process. When the spin-coated film was irradiated at a fluence of 40 mJ/cm^2, no peaks were observed except for those from STO substrate. On the other hand, when it was irradiated at fluence above 50 mJ/cm^2, peaks of PZT (001) and (002) appeared on the shoulders of the strong STO peaks, though weak PZT (110) peak was also observed for all specimens (b)-(e). Based on these results, the specimens (b)-(e) are considered to be highly oriented despite slight amounts of polycrystalline phase. The θ-2θ scan of the specimen (d) showed the highest intensity and narrowest FWHM of rocking curve for the (001) peak among all the specimens. XRD pole-figure analysis for the in-plane alignment investigation showed that the films were found highly aligned in-plane because of four distinct spots due to (110) reflection. Thus, the epitaxial PZT films were successfully obtained with the coating photolysis process without heat treatment.

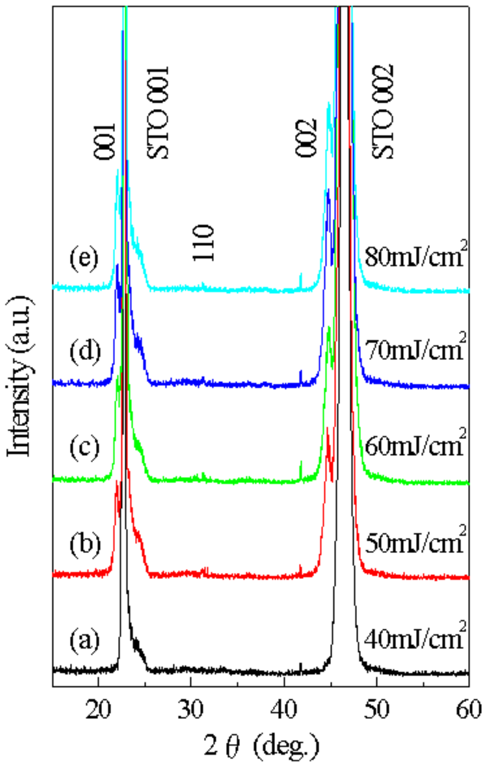

Fig.3.2.1 XRD θ-2θ scans of the obtained films on STO (100) substrate, by coating photolysis process irradiated with ArF laser under different fluence with a repetition rate of 10Hz for 1min.

(2) Nanoparticle-Aggregated Films of Perovskite by Laser Ablation Process

We also developed a technique to prepare crystallized simple oxide nanoparticles with narrow size distribution by adopting off-axis configuration between target and substrate under inert gas condensation scheme [4,5]. We applied this technique for the preparation of complex oxide nanoparticle aggregated films.

A Lambda Physik ArF excimer laser (wavelength: 193 nm, LPX110i) was used for the ablation. The laser repetition rate and pulse width were 10 Hz and 17 ns, respectively. The pulse energy was 200 mJ/pulse. The laser was focused to a spot sized 1 x 4 mm^2 on a target pellet rotating at 40 rpm. The target material of $BaTiO_3$ was commercially available ceramic pellet (Furuuchi Chemical Co.) with 99.9% purity, 18 mm in diameter x 5 mm thick. The quartz glass substrates were placed near the target at an off-axial position with respect to the targets. The targets were irradiated with 20,000 pulses. The pressure of background Ar gas was varied from 1 to 200 Pa. Phase identification was conducted with an X-ray diffractometer (Rigaku, RAD-C) and crystallite size was calculated with Scherrer's formula. Surface morphology was observed using a field-emission scanning electron microscope (FE-SEM: Hitachi, S-800). The composition of thin films was investigated by X-ray photoelectron spectroscopy (XPS: PHI 5600ci).

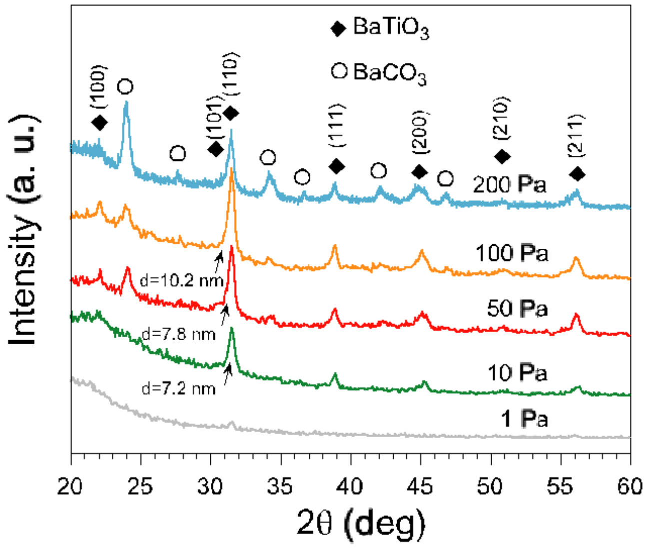

Fig. 3.2.2 XRD diffraction patterns of $BaTiO_3$ thin films deposited at 200 mJ/pulse under various Ar pressure by laser ablation under off-axis configuration.

Fig. 3.2.2 shows XRD diffraction patterns of $BaTiO_3$ thin films deposited at 200 mJ/pulse under various Ar pressure by laser ablation under off-axis configuration. An amorphous phase was observed on the film deposited at 1 Pa. The film deposited at 10 Pa was single phase of $BaTiO_3$ with a crystallite size of around 7.2 nm. With increasing Ar pressure to 200 Pa, XRD peaks of $BaTiO_3$ as well as $BaCO_3$ were observed. This by-product could be due to reaction with air after removing the sample from the chamber. From the observation of surface morphology by FE-SEM, the homogeneous films without droplets were observed below 100 Pa. On the other hand, the aggregates of nanoparticles with porous structure were observed at 200 Pa. From XPS analysis for film composition, oxygen deficiency was observed at low-pressure range below 10 Pa. Over 50 Pa, atomic concentration of all constituent elements was almost constant, especially with the [Ba]/[Ti] ratios to be nearly unity. From these results, we have succeeded in thin film preparation of nanocrystalline complex oxides at room temperature in a certain pressure range. This pressure-controlled PLD process is suggested to be effective in the preparation of nanocrystalline oxides thin film at room temperature for application to oxide electronic devices.

We have therefore succeeded in the preparation of crystallized complex oxide films without heat-treatment by laser processing. For the practical application of such functional oxides, oxygen stoichiometry plays a crucial role in properties. Thus, control of oxygen stoichiometry by the laser processing parameters still remains a future problem.

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4. Study of Photonic Materials

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Purpose

It has been aimed to control the light wave by light in the special group of optoelectronic materials. To achieve the purpose, we have devoted ourselves especially into the molecular design and synthesis of high performance organic nonlinear optical compounds, and the molecular orientation control of σ or π conjugated substances such as permethyloligosilanes (CH_3-$[Si(CH_3)_2]_n$-CH_3 (n=10, 12)) (oligosilane) and sexiphenylene (C_6H_5-$(C_6H_4)_4$-C_6H_5) (oligophenylene) by using capillary filling method and friction transfer method, respectively, both of which were originally developed in our Institute. And azo-benzene polymer with high glass transition temperature was newly synthesized to examine the photo-induced surface relief grating (PSR). The final targets are the deeper understanding for the optoelectrical features in well-defined molecular structures and taylor-made molecular orientations in thin solid films, and the device applications to the rewritable holographic memories by PSR and the photo-responsive organic electroluminescent (EL) device combined with the photoconducting and EL dyes.

Results and Future Problems

(1) The silicon-based polymer; polysilane, is one of the candidates for heat-resisting and excellent hole transporting abilities in the σ conjugated system. Poly-(dimethyl-silane) (PDMS), poly-(diethyl-silane) (PDES) and poly-(diphenyl-silane) (PDPS) having short side chains are very rigid without any glass transition temperature and insoluble in any solvents to be difficult to make thin films. While, these oligosilane with the limited number of silicon backbone is liquid crystalline and easy to form well-oriented film, as shown in Fig. 4.1.1. We have developed the capillary filling method, in which a pair of flat glass plates with a gap of 10 μm was filled by the oligosilane in the space. Due to the self-organization mechanism, molecules tended to align normal to the glass surface. According to the time-of-flight (TOF) measurement, as shown in Fig.4.1.2, it was confirmed that the oriented film represented high hole mobility of 10^{-3} cm^2/Vs, about an order of magnitude improvement over polysilanes, which is the highest value in the world [1].

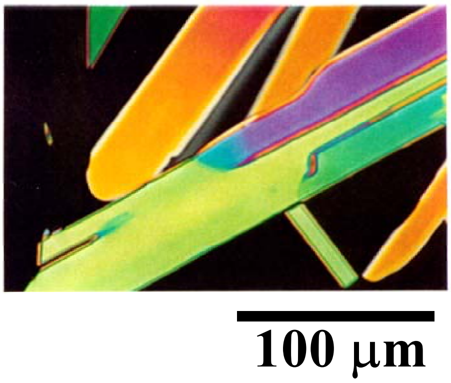


Fig.4.1.1 Polarized optical micrograph of oligosilane (n=12) in smectic B phase.

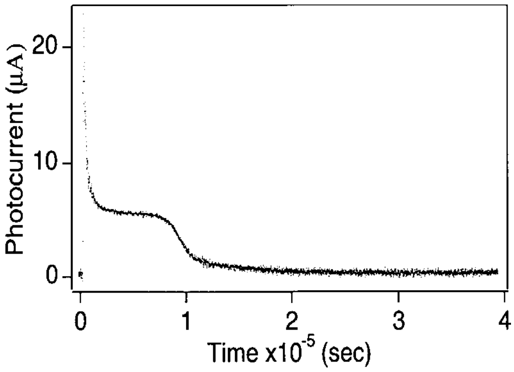


Fig.4.1.2 A typical TOF photocurrent transient for C-phase oligosilane (n=10) at 293 K under an electric field of 1 x 10^5 V/cm.

(2) We have also developed the fabrication technique of one-dimensionally oriented polymer film, friction transfer method [2]. It is a simple technique to slide a polymer pellet onto the solid surface such as glass or quartz plates and a transparent conductive layer, indium-doped tin oxide (ITO), on glass, as shown in Fig. 4.1.3. The transferred polymer films were characterized by TEM, AFM, glazing-incidence X-ray diffractometery, polarized UV, Vis and PL spectroscopy to be confirmed that the polymer main chains oriented along the sliding direction. In addition, when the low molecular weight compounds were deposited in vacuum and soluble substances were casted, spin-coated or polymerized on the one-dimensionally oriented polymer film, they tended to align the same direction.

Figs. 4.1.4 and 4.1.5 is a typical demonstration of the oriented oligomer thin films on friction transferred polymer film [3]. The upper and lower rows of each photograph in Fig. 4.1.5 are pertinent to thin films deposited onto friction transferred PPP and quartz substrates, respectively. In the former case, the polarization direction (shown with a white arrow) is either a) parallel or b) perpendicular to the friction direction of PPP. The films showed isotropic emission on the quartz substrate. One can see the strong photoluminescence from the thin films with parallel orientations.

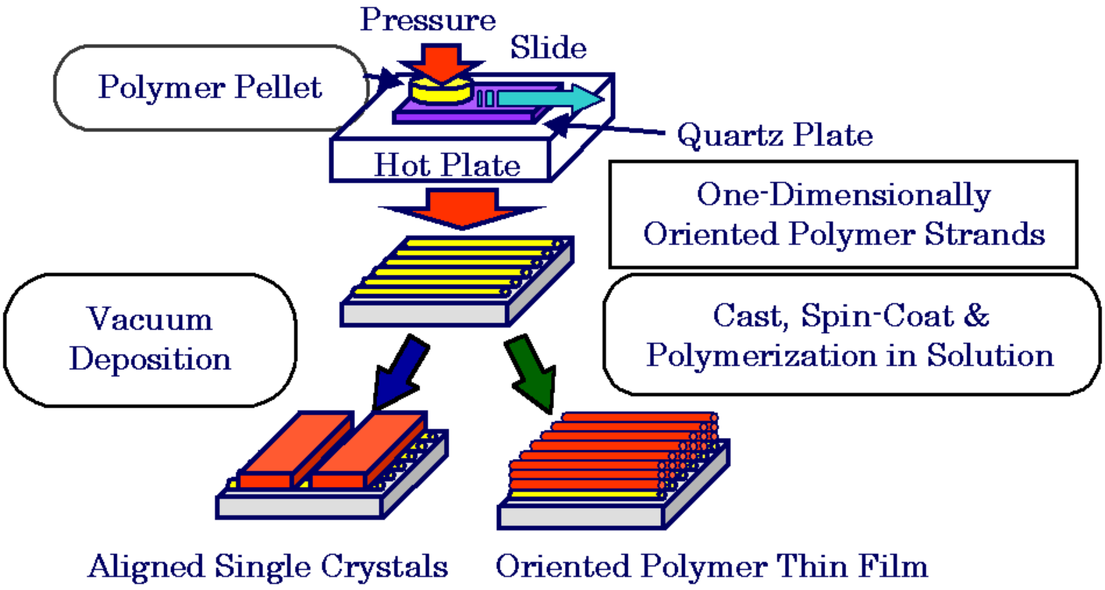
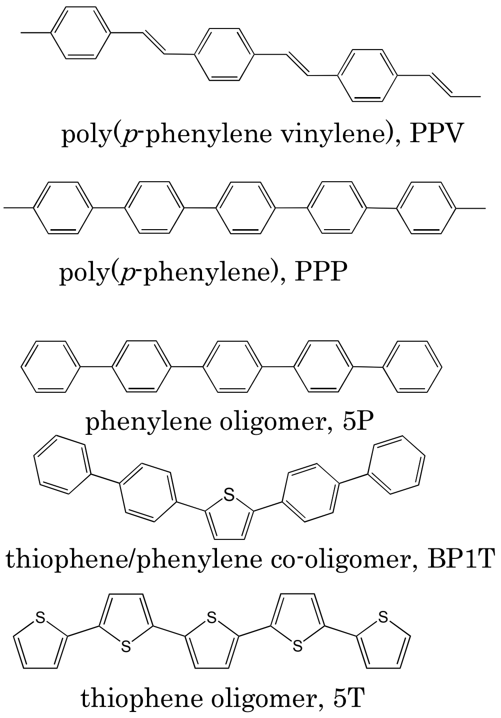


Fig. 4.1.3 Scheme of friction transfer method.



poly(*p*-phenylene vinylene), PPV

poly(*p*-phenylene), PPP

phenylene oligomer, 5P

thiophene/phenylene co-oligomer, BP1T

thiophene oligomer, 5T

Fig. 4.1.4 Molecular structures of p-conjugated polymers and oligopmers

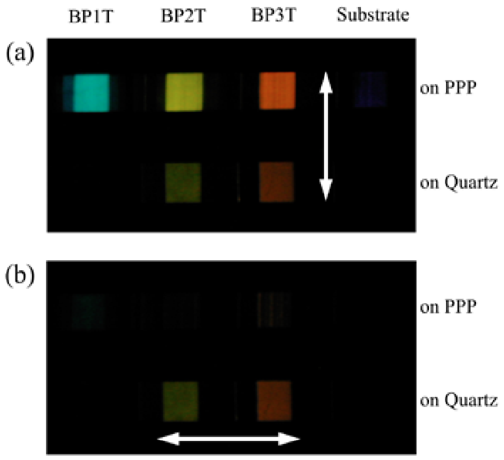


Fig. 4.1.5 Photoluminescence of oriented and non-oriented thin films of oligomers through a polarized filter.

(3) A new type of organic electroluminescent (EL) devices, photoresponsive organic EL devices, have been successfully fabricated, as shown in Fig. 4.1.6 [4]. The green- and blue-emitting EL devices consist of titanyl phthalocyanine (TiOPc) as photoconductive layer in addition with tris-(8-hydroxy quinoline) aluminum (Alq_3) and N,N'-diphenyl-N,N'-di(m-tolyl) benzidine (TPD), and *N,N'*-Diphenyl-*N,N'*-bis(1-naphthyl)-1-1'biphenyl-4,4'-diamine (α-NPD) and 2,9-Dimetyl-4,7- diphenyl-1,10-phenan-throline (BCP), respectively. Each layer of a thickness of 60 nm has been vacuum-deposited on transparent indium-tin-oxide film for photoconductive layer. When the device is illuminated by red or near infra- red laser with a wavelength of 650 nm or 780 nm below the turn-on voltage, the green (550 nm) or blue (450 nm) emission was switched on ,as shown in Fig. 4.1.7. And above the turn-on voltage, the emission is effectively enhanced by laser irradiation. It is confirmed that the photoresponsive EL device can be used as a very fast optical switch (100 μsec order) and a light amplifier (800 – 1000 times) in addition to a red-to-green or red-to-blue light converter.

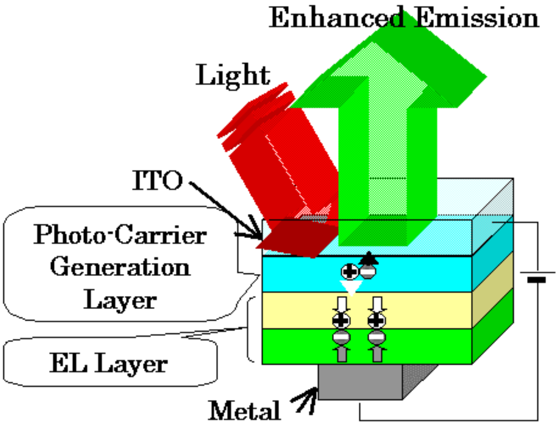


Fig. 4.1.6 Photoresponsive electroluminescent device.

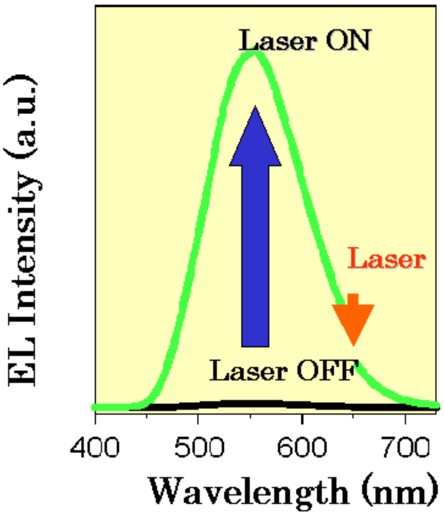


Fig. 4.1.7 EL enhancement by laser irradiation.

(4) It is well-known that the azo-benzene moieties change from trans-conformation to cis in being irradiated by UV to green light, and reversibly transform from cis to trans by IR light or heating. We have developed the photoisomerization of azo polymer (Fig. 4.1.8), which was newly synthesized with high glass transition temperature up to 250°C, to the rewritable holographic memory. When the spin-coated film were illuminated by the linear and circular

polarized light in the near-field condition, it was found that the surface of azo polymer changed to a pair of arcs and a ring shape, respectively, as shown in Fig. 4.1.9[5]. The surface relief can be erased by elevating the temperature or irradiated by IR light. And it also enables to characterize the polarization and the probe tip in the optical fiber.

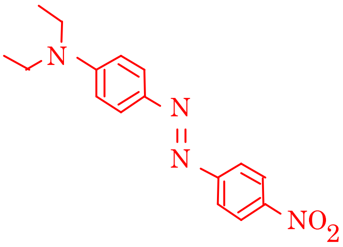


Fig. 4.1.8 Molecular structure of azobenzene polymer

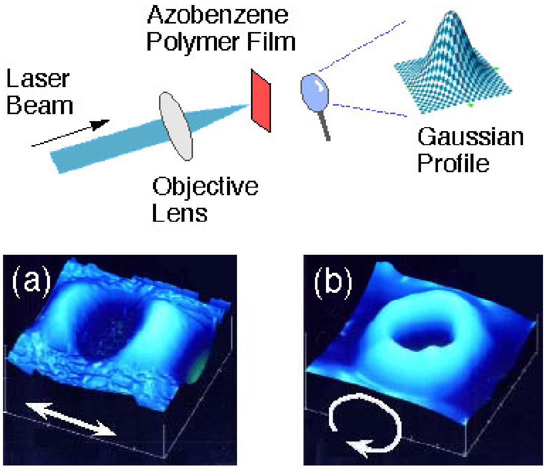


Fig. 4.1.9 Formation of surface relief by using laser beam with linear (a) and (b) circular polarized light.

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5. Study of Photoreactive Materials

M. Matsumoto (Nanotechnology Research Institute)

Purpose

Light-induced structural changes of proteins are key processes of signal transduction and amplification in the vision systems of living creatures. Using such vision systems as one of the models, the goal of this research is to contribute to the development of photoresponsive organic materials and devices having the ability of signal transduction, signal amplification, permeability control, separation and sensing.

Results and Future Problems

Light-triggered J-aggregation of dyes in Langmuir-Blodgett (LB) films was investigated. Mixed LB films composed of an amphiphilic azobenzene and cyanine were fabricated. J-aggregation of the cyanine dye was induced by the photoisomerization of azobenzene, which was accompanied by the development of three-dimensional cone-shaped structures from the film surface. These three-dimensional structures should consist of J-aggregates of the cyanine dye. Similar results were obtained for mixed LB films of azobenzene with another cyanine and merocyanine. These results provide guiding principles for the construction of prototype vision systems composed of molecular materials.

Structural change of the LB film caused by the photoisomerization of azobenzene was studied using AFM. The monolayer of an azobenzene derivative complexed with polycations was transferred onto solid substrates as LB films. Three-dimensional cone-shaped structures appeared and disappeared reversibly from the LB film surface with alternate irradiation by UV and visible light. These results indicate that the two-dimensional LB film structure undergoes significant modification by photoisomerization. This finding is in contrast with the widely accepted concept of "free volume," which does not consider the possibility that LB film structures may change into a three-dimensional structure.

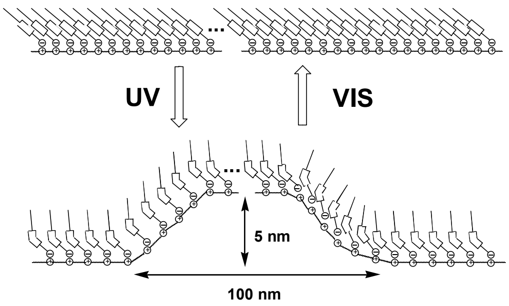

Fig. 5.1.1. Light-induced structural change of an azobenzene/polycation LB film.

Photopolymerization of amphiphilic diacetylenes was examined in the LB films. Transparent LB films of diacetylene became blue and then red with photopolymerization. Morphological change was observed during the polymerization reactions though the polymerization of diacetylenes was assumed as a topochemical reaction. Structures and polymerization properties of the diacetylene LB films were modified by changing the position of the butadiyne moiety and by complexing with polycations.

A photoresponsive amphiphilic polypeptide was prepared by joining two amphiphilic α-helical copolypeptides, poly[(γ-methyl L-glutamate)-co-(L-glutamic acid)], with an azobenzene moiety. The polypeptides were incorporated into a lipid bilayer membrane, forming a transmembrane bundle that acted as an ion-permeable path in the dark. UV light irradiation induced bending of the polypeptide by trans-to-cis photoisomerization of the azobenzene moiety. The photoinduced structural changes of the polypeptide resulted in a destabilization of the transmembrane bundles due to variations in the amphiphilic character of the polypeptide. When stored in the dark, the polypeptides in the membrane returned to the original transmembrane structure. These results indicate that photoinduced reversible structural changes of polypeptides in a bilayer membrane can regulate transmembrane ion transport, and that these molecular systems can work as photoresponsive ion channels.

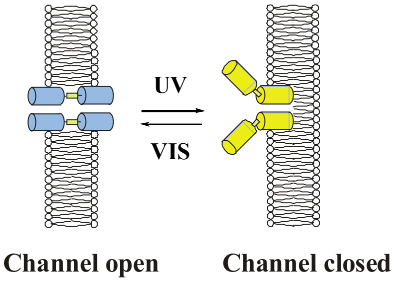

Fig. 5.1.2. Permeability control through membranes.

Photoresponsive amphiphilic polypeptides with an azobenzene moiety in the main chain associated with each other and formed micelles in an aqueous solution in the dark. Hydrophobic compounds were incorporated in the interior of the micelles composed of polypeptides. Photoisomerization of the azobenzene moiety of the polypeptides induced de-aggregation of the micelles, via a bending structure formation of the polypeptide at the azobenzene moiety. These photoinduced structural changes of the micelles resulted in the release of hydrophobic compounds from the interior to the aqueous phase. These systems will be useful for separation and delivery systems.

A newly synthesized vinyl monomer of azobenzene, phenylazoacrylanilide (PhaAAn) formed mechanically stable, quite flexible membranes using mixtures of ethylene glycol dimethacrylate and tetraethylene glycol diacrylate as cross-linking agents. The membranes were synthesized by the thermally induced free radical polymerization between two glass plates. The completely reversible and reproducible process of the photochemical interconversion between *trans* and *cis* isomers of PhaAAn was shown for the monomer and for the PhaAAn-containing membranes as well. Photoisomerization kinetics of PhaAAn-containing membranes was investigated by varying the concentration of the monomer and the ratio of cross-linking agents to solvent. In both cases dependence of the *trans*-*cis* photoisomerization was much more pronounced than *cis*-*trans* photoisomerization. These membranes will be photo-switchable intelligent materials with the ability of separation and sensing.

Direct irradiation of *trans,trans,trans*-1,6-bis(p-formylphenyl)-1,3,5- hexatriene in chloroform under an argon atmosphere led to a fast equilibrium between *trans,trans,trans* (62%) and *cis,trans,trans* (38%) isomers. Other isomers or photoproducts were not detected by HPLC in the photostationary mixture. This is completely different from the reaction of *trans,trans,trans*-1,6-diphenyl-1,3,5- hexatriene on irradiation under similar conditions, in which dimers and solvent adducts were formed via various types of *cis* isomers.

Trans,trans,trans-1,6-Bis(p-formylphenyl)-1,3,5-hexatriene underwent stereoselective photoisomerization to give its *cis,trans,trans* isomer regardless of the solvent polarity. Quantum yields of *trans, trans, trans-to-cis*, *trans,trans* isomerization decreased dramatically with decreasing solvent polarity, whereas the quantum yields of the reverse reaction gradually increased. This led to a *cis, trans, trans-to-trans, trans,trans* "one-way" isomerization in nonpolar solvents. These results present an example of stereoselective photoreaction in structurally nonrestricted systems.

Ten kinds of *trans,trans,trans*-1,6-diphenyl-1,3,5-hexatrienes were prepared by Wittig reactions. Among them only the formyl and cyano derivatives were photoreactive. On irradiation of crystalline powder of the formyl derivative with $\lambda > 370$ nm light at room temperature in air, a mirror symmetric dimer was obtained as a main product. The dimer was identified from MS, ^{1}H NMR, IR and UV-vis spectra. The cyano derivative underwent similar intermolecular [2+2] cycloaddition to give oligomers in the solid state. These results show that photoreactions depend strongly on the substituents and the environment of the molecules.

6. Study of Photon-Beam Analysis

I. Kojima (Metrology Institute of Japan)

Purpose

Our research is based on the advanced photon-beam technology, and aims to develop new measurement techniques for materials characterization, as well as, to accelerate the development of the new photo-functional materials. Following three researches were undertaken. (1) Slab optical waveguide spectroscopy(2) Non destructive surface analysis(3) Spectroscopy under high pressure

Results and Discussion

(1) Slab optical waveguide spectroscopy

The adsorption behavior of cytochrome *c* was investigated using slab optical waveguide (SOWG) absorption spectroscopy at the near ultraviolet region utilizing thin quartz plates as a SOWG. SOWG absorption spectra of cytochrome *c* measured at constant time intervals showed significant influence of surface hydrophilicity and solution properties on the adsorption of this important heme protein in quartz surface. Being polar and typically amphoteric, cytochrome *c* preferred adsorption on hydrophilic surface than on hydrophobic surface as implied by the lower absorbance data obtained in the latter than in the former. At lower ionic strength and in the absence of buffer, cytochrome *c* tends to adsorb on the quartz surface. Plots of near steady-state absorbance versus protein concentration follow hyperbolic pattern in the absence of buffer or at low ionic strength in the sample solution and become more linear as the buffer concentration is increased. The results presented here are explained in terms of the general qualitative understanding of protein adsorption on solid/aqueous interfaces and gives us further aids in elucidating the properties of protein monolayers and films.

A novel refractometer has been developed based on changes in the effective refractive index (RI) of the highest-order mode in a prism-coupled multimode planar waveguide induced by interaction between an evanescent field and a liquid sample (Fig. 6.1.1). The waveguide was composed of a 100-μm-thick quartz plate fixed on a poly(methylmethacrylate) support containing a flow cell. A pair of prism couplers contacts the quartz plate in the flow-cell region. Such an optical sensor can detect the RI of liquid in a wide range by monitoring the resonant angle of the highest-order mode that changes with changes in the sample's RI. When a highest-order mode corresponding to a given RI range is used as the sensor probe, a slight RI change in this range can be detected by measurement of the output light intensity change (Fig.6.1.2). With this method it is demonstrated to have a resolution of 3×10^{-5} for the RI of an aqueous solution. With combining this result with theoretical calculation indicates that the sensor can detect a 0.5-nm-thick monolayer adsorbed on solid/liquid interfaces. Therefore, the sensor is suitable for in situ detection of biomolecular interactions.

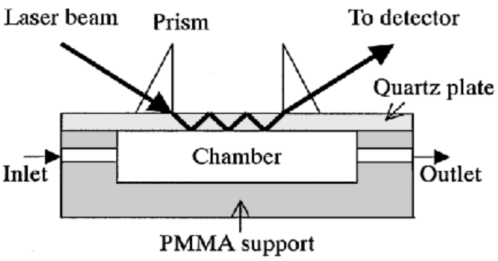

Fig. 6.1.1 Configuration of the sensor

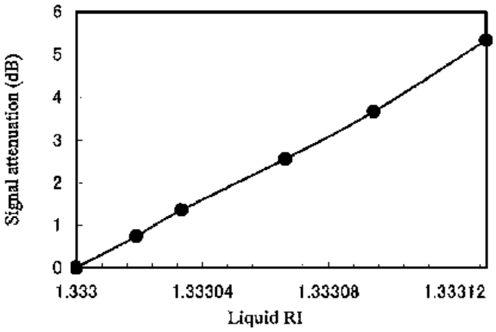

Fig. 6.1.2 Signal attenuation vs. RI

(2) Non destructive surface analysis

There is a strong demand to measure the electronic properties of very thin layers of the nm scale by using X-ray photoelectron spectroscopy (XPS). To obtain the information from several monolayers, it is effective to generate and detect low energy photoelectrons. The escape depth of electrons with a kinetic energy between 15 and 200 eV is less than three monolayers. In a photoelectron peak of such a low kinetic energy region, the intensity ratio of surface atoms to bulk ones becomes relatively high. So surface sensitive measurements need X-rays with longer wavelengths than conventional XPS, which uses Al Kα and Mg Kα X-rays.

Synchrotron radiation emitted x-rays is one of the excellent excitation sources to achieve very sensitive surface analysis, however, it has a disadvantage to require a fairly large facility. We have constructed a laboratory-scale XPS system with a soft X-ray source (Fig.6.1.3). It consists of a carbon X-ray source, a toroidal mirror with a Cr/Sc multiplayer and a hemispherical energy analyzer. The multilayer was fabricated by using the r.f. magnetron sputtering deposition system in our laboratory. Its curvatures are 400 mm and 300 mm on the meridional and saggital plane, respectively. The distance from both the source and the sample was equal to 346 mm and the incident angle used was 30 º(Fig.6.1.4). The multilayer was designed for a periodic thickness of about 2.6 nm, thickness ratio of 0.414 and 120 layer pairs. The photoelectron spectra were recorded for surfaces such as gold, silicon and molybdenum sulfide. The line width of C Kα X-rays irradiated surface was estimated to be about 2.5 eV. The difference in surface sensitivity between C Kα and Al Kα X-ray was observed clearly from the detection of photoelectrons from molybdenum sulfide and silicon.

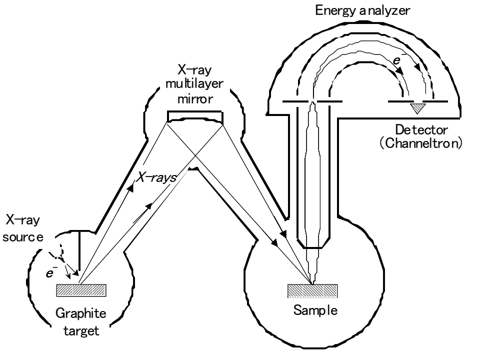

Fig. 6.1.3 Schematic layout of XPS system.

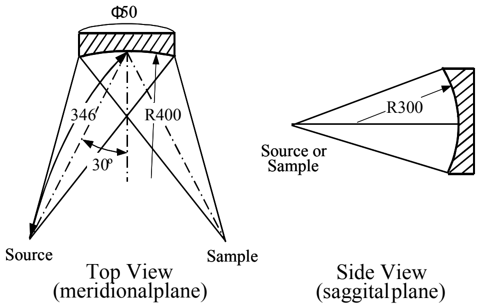

Fig. 6.1.4. Optical parameters of the toroidal mirror. The unit is mm.

(3)Spectroscopy under high pressure

Behavior of materials under high pressure has been studied by using spectroscopic techniques such as infrared absorption and Raman scattering measurements.

Ice, a prototype of hydrogen bonded solids, is a long-standing substance attracting scientists in various research fields. The structural, bonding, electrical properties etc. have intensively been investigated over a wide temperature and pressure region. However protonic diffusion, which is one characteristic of ice, remains still unknown.

In ice, water molecules are connected by hydrogen bonding to form a three-dimensional network. Proton may move in the network by transfer along a hydrogen bond and jump to another one by molecular rotation. The protonic diffusion specific to ice has not been investigated experimentally, since the diffusion rate is small too much compared with the molecular diffusion rate. The diffusional motion is hidden behind the molecular diffusion in usual ice at ambient pressure and low temperature.

We have developed a spectroscopic technique for measuring the protonic diffusion in ice at high temperature and pressure. The mutual diffusion process of proton and deuteron is investigated by collecting infrared reflection spectra time dependently with an H_2O/D_2O ice bilayer. Fig.6.1.5 shows the variation of reflection spectra with time measured for the back surface of a D_2O ice layer. The OH and OD stretch peaks show increase and decrease in intensity with time, respectively, as a result of the H/D mutual diffusion through the ice bilayer specimen.

The diffusion coefficient was determined by a conventional analysis based on Fick's equation of diffusion to be 6.2×10^{-16} m^2/s at 400 K and 10.2 GPa. This value is larger by a factor of 10^4 than that estimated for ambient-pressure ice at 258 K. Further diffusion measurements at higher pressures at a fixed temperature of 400 K revealed that the protonic diffusion coefficient decreased monotonically with pressure up to 62 GPa. Even at 62 GPa, where the energy barrier at hydrogen bond center smears out and hence the proton can transfer freely between the neighboring water molecules, a tendency of increase in diffusion rate was not observed. Proton transfer associated with molecular rotations likely plays a dominant role in controlling the proton diffusion process in dense ice.

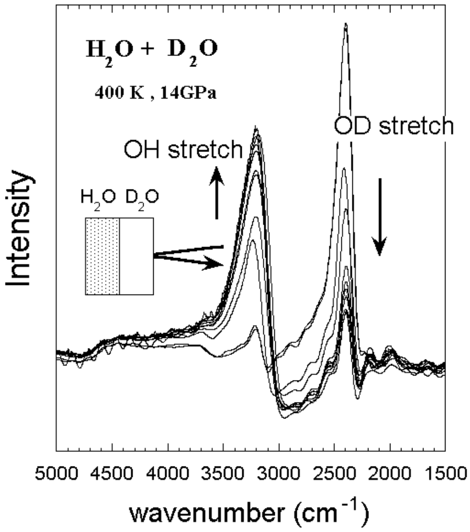

Fig. 6.1.5 Variation of reflection spectra with time.

Conclusion

In the research for advanced materials characterization, it is intensely requested that a method will be developed that can detect extremely small amounts with high sensitivity and can probe phenomena occurring on the atomic or molecular scale. The slab optical waveguide spectroscopy is a fairly sensitive analytical method, and is expected to play an important role to reveal the mechanisms of dynamic reactions occurring at solid/liquid interfaces. Laboratory-scale XPS equipped with soft X-ray will be very useful for in-situ and highly sensitive surface analysis. The spectroscopic characterization of materials under an ultimate condition will shed light on the future development of photo-functional materials.

IV. Research Activities in the COE Development Area

1. Summary of Research Activities in Fiscal Years 2000 and 2001

(1) Publications and presentations*

	Original papers	Review articles	oral presentations	Total
Domestic	41 (25)	35 (18)	348 (212)	424 (255)
International	207 (120)	8 (4)	272 (171)	487 (295)
Total	248 (145)	43 (22)	620 (383)	911 (550)

* Figures in the parentheses: Special Coordination Fund supported area

(2)Patents applied for*

Total	Domestic	Overseas
67 (46)	42 (22)	25 (24)

* Figures in the parentheses: Special Coordination Fund supported area

(3) Awards*

Total	Domestic	Overseas
3 (3)	2 (2)	1 (1)

* Figures in the parentheses: Special Coordination Fund supported area.

(4)Numbers of papers published in journals with high impact factors (Only papers from Special Coordination Fund supported area are included)

Journal	I. F.*	Photo-reaction mechanism	Light energy conversion	Laser-induced reaction	Total
Nature	25.8		1		1
Chem. Rev.	20.0		1		1
J. Am. Chem. Soc.	6.0	1			1
Biophys. J.	4.5	2			2
Chem. Commun.	3.7		3	2	5
Chem. Mater.	3.6		1		1
J. Phys. Chem. B	3.4	3	2	1	6
J. Chem. Phys.	3.3	10			10
Phys. Rev. B.	3.1	1			1
Langmuir	3.0	1	1		2
New J. Chem.	3.0		2		2
J. Comput. Chem.	2.9			1	1
J. Phys. Chem., A	2.8	2		1	3
Inorg. Chem.	2.7		1		1
Tetrahedron Lett.	2.6		1		1
J. Chem. Soc., Dalton Trans.	2.5		2		2

Adv. Colloid. Interface Sci.	2.5	1			1
Chem. Phys. Lett.	2.4	5	4		9
J. Appl. Phys.	2.2			2	2
J. Opt. Soc. Am., B	1.9			1	1
Chem. Phys.	1.9	1			1
Bull. Chem. Soc. Jpn.	1.8	1	1		2
Catal. Lett.	1.8		2		2
J. Polym. Sci. A: Polym. Chem.	1.7			1	1
J. Mol. Catal. A, Chem.	1.7		1		1
Chem. Lett.	1.6		3		3
Appl. Phys., A: Mater.	1.6			2	2
Appl. Catal., A	1.6		2		2
Appl. Organometal Chem.	1.6		1		1
J. Mater. Res.	1.3		1	1	2
Solid. State. Comm.	1.3		2		2
Appl. Surf. Sci.	1.2			1	1
Inorg. Chim. Acta	1.2		3		3
J. Sol-gel.. Sci. Tech.	1.0			1	1

* I.F: Impact factor.

(5) Organizing symposia and other meetings

FY	Title	Outline	Duration (days)	Participants	
				Total	From Overseas
2000	The 4th NIMC International Symposium on Photoreaction Control and Photofunctional Materials (PCPM2001)	Symposium on photoreaction control and photofunctional materials. 14 invited lectures, and 10 oral and 97 poster presentations.	3	276	46
2001	The 1st PCRC Symposium - Photoreaction Control Technology for the 21st Century-	Symposium to introduce the purpose of and the research at PCRC. 2 invited lectures and 2 oral presentations.	1	124	4
2001	The 5th AIST International Symposium on Photoreaction Control and Photofunctional Materials (PCPM2002)	Symposium on photoreaction control and photofunctional materials. 16 invited lectures, and 7 oral and 105 poster presentations.	3	252	36

2. Publications in Fiscal Years 1997-2001

2-1. FY1997

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