

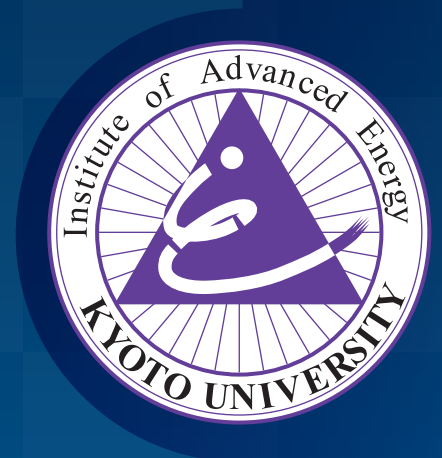
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Institute of Advanced Energy  
Kyoto University

# ANNUAL REPORT

## 2024



京都大学エネルギー理工学研究所

Institute of Advanced Energy, Kyoto University



# **ANNUAL REPORT**

**2024**

**Institute of Advanced Energy  
Kyoto University**

Gokasho, Uji, Kyoto 611-0011  
Japan



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## FOREWORD



"Finally, the times have caught up with us." Knowing that this sounds arrogant, we the Institute of Advance Energy think this way. Fourteen years ago, in 2011, our Institute started a project recognized by the Minister of Education, Culture, Sports, Science and Technology for joint use and collaborative research. The project is named "Zero Emission Energy Research", and its mission is to research energy that minimizes emissions of harmful substances such as carbon dioxide. It is hard to imagine now, but at the time we started this project, there were quite a few skeptical opinions regarding the significance and importance of this mission. This project received high evaluations at the end of both the first phase from 2011 to 2015 and the second phase from 2016 to 2021. We believe this reflects the broad recognition of the significance and importance of our mission in society and the appreciation of the contributions our Institute has made toward achieving this mission. Following this, we are currently executing the third phase of the project from 2022 to 2027. While the times have caught up to us, to stay ahead and not just keep pace

with the times, we established the "Integrated Research Center for Carbon Negative Science (ICaNS)" in 2022 to research "Carbon Negative Energy," which further advances "Zero Emission Energy," and we continue and progress research that stays ahead of the times.

The Institute of Advance Energy was established in 1996 with the aim of exploring the nature of energy by returning to natural laws and principles and creating new energy theories to be carried on by the next generation, as well as developing cutting-edge technologies to lead and realize them. The Institute consists of three departments, each named after energy generation, transformation, and utilization, encompassing 14 research fields. Additionally, it includes the "Laboratory for Complex Energy Processes" that provides shared equipment for the aforementioned joint use and collaborative research project, and the previously mentioned ICaNS. The Institute has established two key interdisciplinary research areas as its cornerstone: one aimed at realizing nuclear fusion, called "Plasma and Quantum Energy," and the other aiming for efficient energy utilization and transformation based on biological energy utilization principles and material science, called "Soft Energy."

Each research field of this Institute serves as a cooperative course of the Graduate School of Energy Science, Kyoto University, with a considerable number of master's and doctoral students from that Graduate School assigned, doing education in a cutting-edge research environment. Additionally, we contribute to undergraduate education through the provision of university-wide common subjects at Kyoto University.

"Rather than asking what the Institute can do for you, I would like you to consider what you can do for the Institute." When I became the director last April, I made this greeting at a meeting of all faculty and staff of the Institute. This carries my sentiments based on the famous speech of the late President Kennedy. When I first heard his speech, I thought, "What a terrible demand to say, 'The country does nothing for you, but I want you to serve the country.'" However, as I encountered this phrase a few more times, I began to feel it was quite a good word. I came to understand that the country is not something to rely on and depend upon but is to be developed through the strength of each individual. Furthermore, this phrase seems to envision a country where each person can shine. An Institute where each member can shine is certainly a wonderful thing, isn't it? With this thought, I made the aforementioned greeting. Currently, Kyoto University is united in aiming to be selected as a University for International Research Excellence (Kokusai Takuetsu Kenkyu Daigaku). Shining members are expected to play important roles as significant pieces in that University.

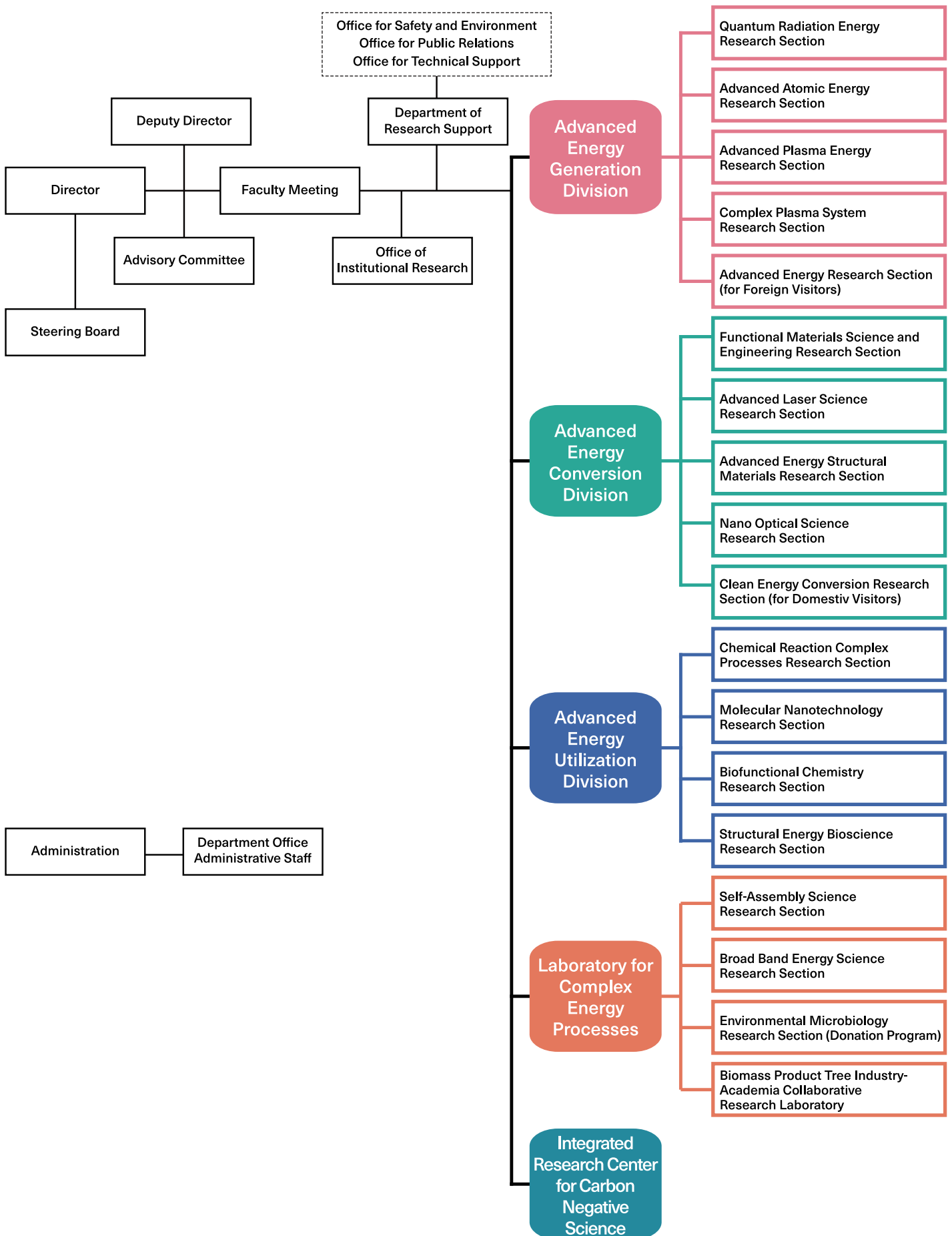
This annual report summarizes the major research achievements of each of IAE's research divisions for FY2024 (April 2024-March 2025) to represent IAE's research activities. I hope you will enjoy our Institute's ongoing research in a wide range of scientific disciplines, which will surely provide innovative solutions to meet the demands of achieving carbon neutrality.

March 2025

A handwritten signature in dark ink that reads "M. Katahira". The signature is fluid and cursive.

Masato KATAHIRA  
Director  
Institute of Advanced Energy  
Kyoto University

## 2. ORGANIZATION CHART





### **3. RESEARCH ACTIVITIES**



## **3-1. RESEARCH ACTIVITIES IN 2024**



## Quantum Radiation Energy Research Section

H. Ohgaki, Professor

H. Zen, Associate Professor

Ju Yoon Hnin Bo, Assistant Professor

Jordi Cravioto Caballero, Program-Specific Assistant Professor

## 1. Introduction

Coherent-radiation energy with a wide wavelength tunability and high power is an indispensable tool for exploiting cutting-edge energy science. The research in this section aims to generate and apply new quantum-radiation energy. Free-electron laser (FEL) is one of such radiation. We have been developing a mid-infrared FEL, KU-FEL. To extend study field wider wavelength region, a coherent A compact THz source, and Laser Compton Gamma-ray (LCS) for isotope imaging have been carried out. Transdisciplinary research on renewable energy has also been promoted through international collaborations.

## 2. Free-electron Laser

FEL is a next generation light source because of its wide wavelength tunability where the conventional lasers cannot reach, potential high efficiency, and high peak power. We have been developing several new approaches to improve our FEL system and application research by using a collaboration research program.

## 2.1 KU-FEL

The target wavelength of KU-FEL is MIR (Mid infra-red) regime, from 5 to 20  $\mu\text{m}$ , with high-power and tunability for basic research on energy materials. Figure 1 shows a schematic drawing of the KU-FEL system. The KU-FEL consists of a 4.5-cell thermionic RF gun, a 3-m travelling wave accelerator tube, a beam transport system, and a 1.8-m undulator and a 5-m optical resonator. The FEL device now can cover the wavelength range from 3.4 to 28  $\mu\text{m}$ . The maximum macro-pulse energy which can provide is around 80 mJ in a 2- $\mu\text{s}$  macro-pulse at the wavelength of 8.5  $\mu\text{m}$ . The FEL is routinely operated and opened for internal and external users.

For increasing the peak power of the KU-FEL, the photocathode operation of the 4.5-cell thermionic RF gun has been established. Under the photocathode operation, the micro-pulse energy of 100  $\mu\text{J}$  and the world's highest extraction efficiency (9.4%) of the oscillator-type FEL has been achieved. Then, the micro-pulse duration was shortened down to 150 fs ( $\sim 4.2$  cycles at 11  $\mu\text{m}$ ). In addition, nonlinear compression of 8.6- $\mu\text{m}$  FEL pulse was achieved, and the pulse duration was compressed from 146 to 106 fs (from 5.1 to

3.7 cycles) by passing through a 30-mm thick Ge plate.

For further increase of the peak power of KU-FEL, newly fabricated 1.6-cell RF gun has been installed at the upstream side of the accelerator tube. The commissioning of the new RF gun was successfully finished and FEL lasing with the electron beam generated from the gun with higher bunch charge than the 4.5-cell RF gun has been achieved.

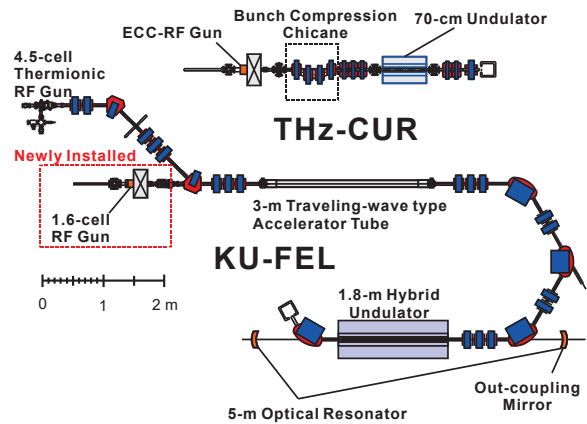


Fig. 1 Schematic drawing of the KU-FEL and THz-CUR

## 2.2 THz Coherent Undulator Radiation Source

A compact terahertz coherent undulator radiation source (THz-CUR in Fig. 1) has been constructed. It consists of an energy-chirping-cell-attached RF-gun, a solenoid magnet, a magnetic chicane bunch compressor, a triplet quadrupole magnet, a planar undulator, and a laser system for photocathode. In this device, short electron bunches are generated by the photocathode RF gun and the bunch compressor. The electron bunches are injected to the undulator and intense coherent undulator radiation can be generated.

The polarization control method of the THz-CUR has been developed under collaboration with Dr. Kashiwagi, Tohoku University. The polarization state of the THz-CUR can be easily controlled from linear to left-handed circular and right-handed circular without significant power loss.

## 2.3 Application of MIR-FEL and THz-CUR

Many application researches of MIR-FEL and THz-CUR have been performed under the Joint Usage/Research Center for Zero Emission Energy

Research of our Institute. In JFY2024, 15 external user groups used KU-FEL.

One of the many applications achieved using MIR-FEL is mode-selective phonon excitation. In strongly correlated systems, it is beneficial to understand the role of each phonon mode's impact on the material properties. Due to the widely tunable wavelength range of MIR-FEL, many phonon modes in the mid-infrared region are accessible. Selective excitations of Raman active phonon modes (LO mode of 6H-SiC,  $A_1$  (LO) mode of GaN), Raman inactive mode (LO3 mode of  $\text{SrTiO}_3$ ), and even, infrared inactive mode ( $T_{2g}$  of single crystal diamond) have been successfully observed through pump-probe experiments. Most recently, the antiferromagnetic domain pattern change has been observed when 2TO phonon mode of nickel oxide is excited. The domain patterns were visualized via magnetic linear birefringence using ns-Nd: YAG laser. Temporal evolution of the phonon induced pattern changes has been recorded by controlling the ns laser via electronic delay generator.

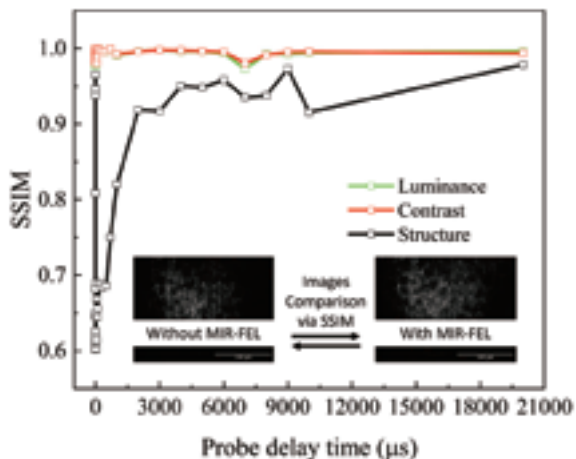


Fig. 2 Measurement results of structural similarity index measure (SSIM) of antiferromagnetic domain pattern changes without and with MIR-FEL irradiation

### 3. Isotope Imaging for Nuclear Safety and Security

Multi-isotope imaging method has been developed at BL1U beamline in UVSOR, Institute of Molecular Science. However, a quantitative evaluation has not been achieved yet because of a high spatial dependence of the Laser Compton Gamma-ray beam. We proposed a new method to generate a flat spatial distribution with a beam size of a few mm in diameter, called a flat-LCS gamma-ray beam. The Proof-of-Principle experiment has been performed, and we are trying to measure the abundance of natural lead target by using Flat-LCS gamma-ray beam in UVSOR. Enriched  $^{206}$ ,  $^{207}$ ,  $^{208}\text{Pb}$  rods of 6 mm $\phi$  were used for witness targets and natural lead absorber target was irradiated by normal LCS and Flat-LCS gamma-ray beams. The NRF gamma rays emitted from the witness were measured

with two Ge detectors. The evaluation of the abundance in a natural lead absorber is undergoing.

### 4. Social aspects of energy use

In Southeast Asia, significant progress has been made toward universal electrification. Yet, today, around 35 million people remain without access. Most research on electrification challenges has focused on regions like Africa and South Asia, primarily examining economic, technological, and institutional factors while neglecting social dimensions. In response, our group has explored the social impacts of various electrification projects from a quality-of-life perspective since 2016. We have assessed changes in living conditions and social disparities across different contexts in Southeast Asia (Fig. 3). Utilising mixed methods from the social sciences, our findings indicate that renewable electrification offers several benefits. These include reduced reliance on expensive kerosene lamps and car batteries, improved educational opportunities for students, and enhanced social interactions. However, a limited number of households have utilized these systems to increase their economic activities. Additionally, the transition to electrification may unintentionally reinforce existing inequalities, favouring those with higher socio-economic status before the change. Other critical factors identified in the electrification process include changing lifestyles and limited system capacity. Furthermore, we are actively engaged in additional research projects that focus on energy justice and the clean and sustainable energy transition.



Fig. 3 Rural electrification fieldwork locations 2016-2025

### Acknowledgment

All our research works have been supported by the KAKENHI, Q-LEAP(MEXT), JASTIP(JST), UVSOR Collaboration Research, CSEAS DASU (Kyoto University), and the Laboratory for Complex Energy Processes Collaboration Research (IAE).

## Collaboration Works

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## Advanced Atomic Energy Research Section

K. Nagasaki, Professor (concurrent)

J. Yagi, Associate Professor

## 1. Introduction

Main objective of our research division is to realize advanced energy systems for the sustainable development under global environmental constraints. We have shown a Zero-emission energy scenario based on fusion energy with biomass-based recycling system where biomass waste is converted into liquid fuel or hydrogen. And further we now propose an innovative Negative emission scenario to isolate CO<sub>2</sub> in the atmosphere by a carbonization process. Our research section focuses on development of hydrogen isotopes fuel circulation system, breeding blankets, fusion material R&D, feasibility study for fusion-biomass hybrid power system, conversion of biomass waste, and fusion neutron generation/measurement. The following are the main research achievements in the fiscal year of 2024.

- Accomplishment of the highest efficiency for fusion fuel extraction from lead lithium eutectic alloy using the droplet system in the fusion study field.
- Hydrogen solubility investigation for new liquid breeder Li-Pb-Na.
- Corrosion study of fusion reactor materials in supercritical carbon dioxide
- Syngas production from biomass using microwave reactor.

## 2. Fusion fuel (hydrogen isotope) recovery system development from liquid lead lithium droplets

Lead lithium eutectic alloy (Pb-17at%Li, Pb-Li) is a promising liquid material for the fuel (hydrogen isotope) breeding on a fusion reactor. Efficient recovery of the bred fuel is a key viable issue of liquid blanket.

We are studying the recovery method from liquid Pb-Li droplets in a vacuum, named as vacuum sieve tray (VST). This fiscal year, on a collaboration work with National Institute for Fusion Science (NIFS), we challenged a further efficiency enhancement by the tandem extraction method. As shown in Fig. 1, we obtained the efficiencies of between 0.8 and 0.9 [1]. The results exceeded the pre-estimated values of between 0.6 and 0.8. The turbulent flow at the nozzle is considered a probable contributory factor. Previously, under turbulent region of the nozzle jet, it is anticipated to disperse and droplets formation is unattainable. These results are expected to open the new design window for the liquid blanket system. We plan to proceed further detail study under turbulent region.

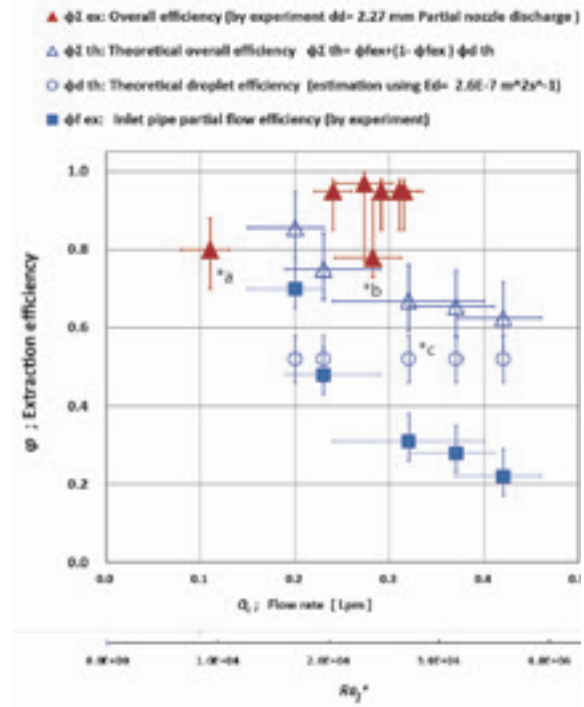


Fig.1 The obtained extraction efficiencies, marked as  $\blacktriangle$ , as a function of the liquid flow rate [1]. The obtained overall efficiencies exceeded the pre-estimated value marked as  $\triangle$ . The turbulent flow at the nozzle jet is considered the prob-

## 3. Hydrogen solubility and tritium breeding ratio of advanced liquid breeder Li-Pb-Na

Lead lithium eutectic alloy (Pb-17at%Li, Pb-Li) has a low hydrogen solubility, which poses a problem for tritium leakage.

We are focusing on Li-Pb-Na (Pb-19at%Li - 5at%Na, Li<sub>19</sub>Na<sub>5</sub>Pb<sub>76</sub> and Pb-15at%Li-35at%Na) liquid alloy to reduce the mass density and increase hydrogen solubility relative to Pb-Li. Fig. 2 shows the temperature dependence on the hydrogen solubility of the liquid metal materials. The hydrogen solubilities of Li<sub>19</sub>Na<sub>5</sub>Pb<sub>76</sub> and Li<sub>15</sub>Na<sub>35</sub>Pb<sub>50</sub> were similar, and higher than that of Pb-Li. Furthermore, the mass density measured by Archimedes' method was reduced by 30% when 35at% Na was used. On the other hand, the tritium breeding ratio (TBR) was calculated and the TBR was lower than 1.0 when Li<sub>15</sub>Na<sub>35</sub>Pb<sub>50</sub> was used. The liquid blanket should have a TBR more than 1.0, and adding too much Na was found to be inappropriate. The TBR of Li<sub>19</sub>Na<sub>5</sub>Pb<sub>76</sub> and Pb-Li was similar, indicating that Li<sub>19</sub>Na<sub>5</sub>Pb<sub>76</sub> has a higher hydrogen

solubility than Pb-Li and reduces the pump load. We continue to develop liquid metal materials with high hydrogen solubility, low mass density, and high TBR.

#### 4. Corrosion of Fusion Reactor Structural Materials by Supercritical Carbon Dioxide

Recently, supercritical carbon dioxide has attracted attention as a breeder material that can be easily separated from tritium, does not cause MHD pressure drop, and can be used for driving turbine directly. On the other hand, the corrosion behavior of supercritical carbon dioxide on F82H has not been researched sufficiently.

We are researching corrosion behavior of structural materials in response to supercritical carbon dioxide and coatings on structural materials for corrosion resistance, as collaboration research with the Chemical Reaction Complex Processes Research Section in IAE (Prof. Nohira lab.) and NIFS. Corrosion of F82H in a supercritical CO<sub>2</sub> 25 MPa, 550 °C atmosphere for 500 hours resulted in the formation of magnetite and iron-chromium oxide layers on the F82H surface. Ni was coated by electrodepositing on F82H (F82H+Ni) to prevent these oxidations. F82H+Ni cor-

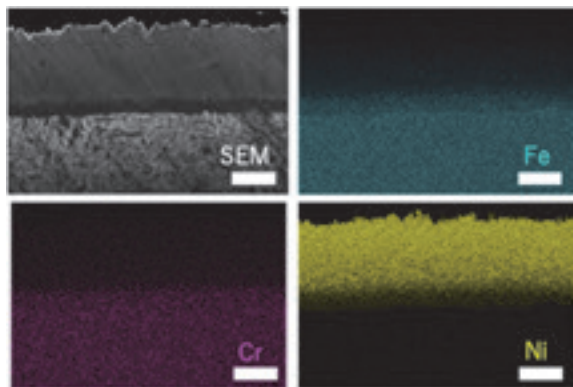


Fig.3 Cross-sectional SE image and elemental mapping of F82H+Ni after 500h test (scale bar = 30µm)

roded under the same conditions showed that oxidation was prevented, and that the Ni coating imparts corrosion resistance (as shown in Fig. 3). We continue to develop advanced Ni and W coated structural materials for F82H and other structural materials such as V.

#### 4. Fusion-biomass hybrid power system

We propose to apply the enormous energy from fusion power generation to the conversion of biomass resources. Fusion power generation produces enormous amounts of electrical and heat energy from condensers and other equipment. We are studying microwave heating of biomass in a steam atmosphere, using electrical energy for microwave oscillation and heat energy for steam generation. While this method is expected to use both electrical and heat energy from fusion effectively, the effects of steam addition and

microwave heating have not been sufficiently investigated. To investigate the effect of steam addition, biomass mixed with a susceptor was heated by microwave at 800°C with and without steam.

The results showed that steam addition induces a water gas shift reaction, leading to hydrogen-rich products (Fig. 4) [2]. It was also suggested that the combination of microwave heating and steam reduces the formation of secondary char that can cause pipe blockage

(Fig.5). In addition, a techno-economic assessment of the possible application of fusion systems to biomass conversion is conducted to examine the feasibility of a biomass-fusion hybrid system.

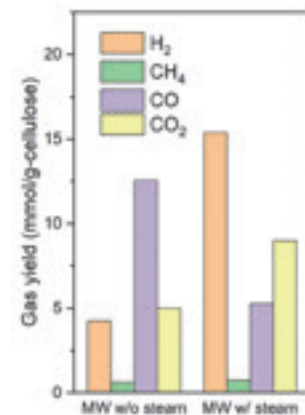


Fig.4 Microwave gas generation at 800 °C with and without steam

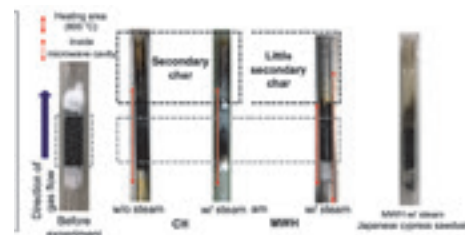


Fig.5 The appearance of the reaction vessels before and after experiments at 800°C.

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## Advanced Plasma Energy Research Section

K. Nagasaki, Professor  
 S. Kobayashi, Associate Professor  
 S. Inagaki, Assistant Professor

### 1. Introduction

The current subjects of this research section are to study the properties of high-temperature plasmas in order to control and improve the plasma energy confinement from the physical viewpoint of nuclear fusion research. The experimental and theoretical investigations for optimizing the helical-axis heliotron configuration are in progress under collaboration with other groups of international/national institutes and groups of other universities under the auspices of the Collaboration Program of the Lab. Complex Energy Processes, IAE, and the Collaborative Research Program of NIFS (National Institute for Fusion Science).

In this report, a remarkable result obtained in the Heliotron J experimental study in FY2024 is reported focusing on (1) plasma dynamics of edge/scrape-off-layer (SOL) and (2) stochastic acceleration study for relativistic electron formation. Since Heliotron J has low magnetic shear, the role of plasma current is important to control the edge/SOL plasmas. Recently, a unique characteristic of the SOL plasma dynamics was observed by controlling electron cyclotron current drive, which indicates the oscillating phase transition between divertor and limiter configuration. The relativistic electrons exceeding 2MeV have been observed when a non-resonant microwave was launched in Heliotron J. To reveal the acceleration mechanism, we conducted a synthetic diagnostic to understand the relativistic electron energy distribution using combination of X-ray scintillator measurement and radiation transport simulation.

### 2. Transition from divertor to limiter configuration and associated transient plasma oscillation

A transition from a divertor configuration to a limiter configuration and associated transient plasma oscillation were observed in configurations whose confinement region expanded with the codirected plasma current in the Heliotron J device [1]. Figures 1(a)-(e) show the discharge waveforms for different ECH injection angles ( $N_{\parallel} = 0.0$  and  $0.38$ ) with the same magnetic configurations. Although the line-averaged electron density  $n_e$  and stored energy  $W_p$  were practically the same, the plasma current  $I_p$  was approximately 0.8 kA for  $N_{\parallel} = 0.0$  and approximately 2 kA for  $N_{\parallel} = 0.38$ , flowing in a direction that increased the rotational

transform. Figure 1(d) and (e) show color maps of the time evolution of the ion saturation current  $I_{is}$  measured using the divertor probe during low and high  $I_p$  discharges, respectively. The peak position of the  $I_{is}$  shifted from  $R = 1.47$  m to approximately  $R = 1.40$  m at a high  $I_p$  discharge. Such changes in the edge magnetic structure were qualitatively consistent with calculations performed using a field line tracing code based on the assumption where the plasma current flows on the magnetic axis. This supports the idea that the plasma current modifies the edge magnetic structure.

At the time of the magnetic structure transition, oscillations with a toroidal mode number  $n = 1$  were observed at approximately 2 kHz. The transition to the limiter configuration altered the transport in the edge region, causing the edge plasma to oscillate as a transient response.

In the future, we will compare measurements from several upgraded multiple diagnostic instruments to extensively clarify the spatial structure of the oscillations, including the core region, and investigate the underlying physical mechanisms.

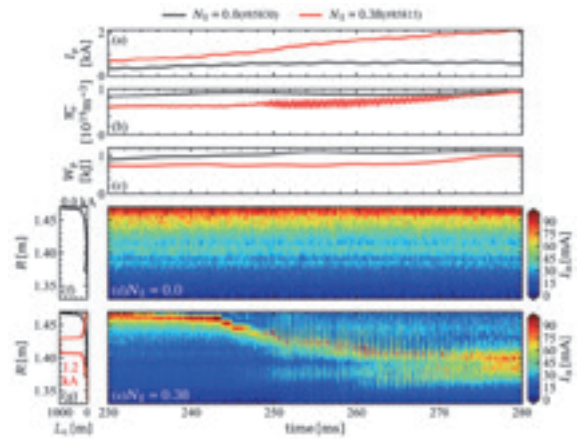


Figure 1 (a) Waveform of plasma current  $I_p$ , (b) line-averaged electron density  $n_e$  and (c) stored energy  $W_p$ . The black and red lines indicate the discharge waveforms of  $N_{\parallel} = 0.38$  and  $0.0$ , respectively. (d) Connection length  $L_c$  at  $0.0$  kA (black) and  $1.2$  kA (red). (e) Time evolution of the profile of the ion saturation current  $I_{is}$  on the divertor probe.

### 3. Measurement of X-ray bremsstrahlung radiation from high energy electrons by stochastic acceleration phenomena

A synthetic diagnostic of X-ray bremsstrahlung radiation and a Monte-Carlo radiation transport simulation were carried out to obtain the electron energy distribution in stochastic acceleration experiments in Heliotron J. Three sets of LaBr<sub>3</sub>(Ce) scintillator and photomultiplier tubes were installed in Heliotron J in three directions relative to the magnetic field line, co, counter, and perpendicular, to determine the velocity distribution of the high-energy electrons. These systems are positioned about 5 m away from the vacuum chamber and shielded by lead blocks and magnetic shields to reduce the influence of stray radiation and magnetic fields. The vacuum chamber of Heliotron J is made of stainless steel with a 3-D helical shape. Since its X-ray shielding effect is not negligible when obtaining the X-ray energy distribution in the vacuum chamber, the Monte Carlo radiation transport code PHITS (Particle and Heavy Ion Transport code System) was applied to Heliotron J to clarify the shielding effect.

Figure 2 shows the CAD model for Heliotron J used in the transport simulation. Given the vacuum chamber and coils shape information and X-ray energy distribution expected in the vacuum chamber, this

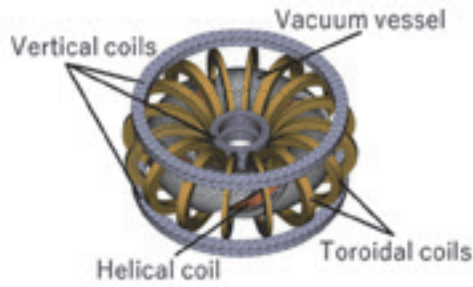


Fig. 2. CAD model for Heliotron J vacuum vessel and magnetic coils. The main vertical coils are not illustrated in the figure because the radius (4 m) is more than twice the major radius (1.2 m) of the machine.

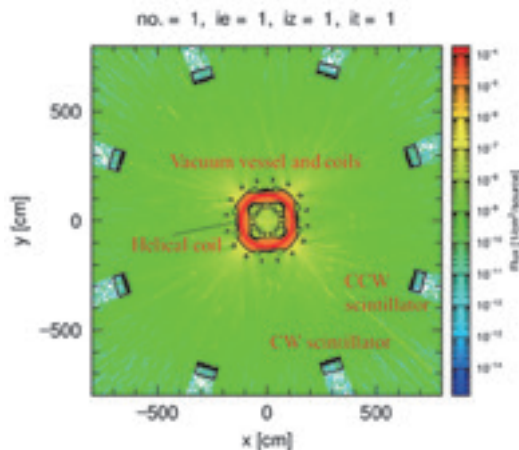


Fig. 3. Contour map of X-ray flux around Heliotron J calculated by radiation transport simulation.

code can calculate X-ray energy distribution considering the shielding effect of Heliotron J. This simulation model was based on CAD (Computer Aided Design) data of Heliotron J devices. The X-ray energy distribution in the vacuum vessel was adapted until the simulated and measured X-ray spectrum outside the vacuum vessel match with each other. Figure 3 shows one example of the X-ray trajectory calculated by radiation transport simulation. The emitted X-ray flux is relatively high in the region where the thickness of the vacuum vessel is thin.

By comparing the simulated and measured X-ray spectrum, we obtained the X-ray expected in the vacuum vessel by the iterative calculation. Figure 4(a) shows the shapes of resultant result distribution have two types of shape: power-law distribution at more than 450 keV and Maxwell distribution at less than 450 keV. Except for the low energy range less than 200keV, whose statistical error is large, X-ray bremsstrahlung energy distribution is consistent with the model analysis having power law energy distribution as shown in Fig. 4(b), indicating the high-energy electrons produced by the non-resonant microwave heating in Heliotron J has power law electron energy distribution. The characteristic indicates the acceleration mechanism is similar to stochastic acceleration commonly observed in space, magnetosphere and high-intense laser experiments.

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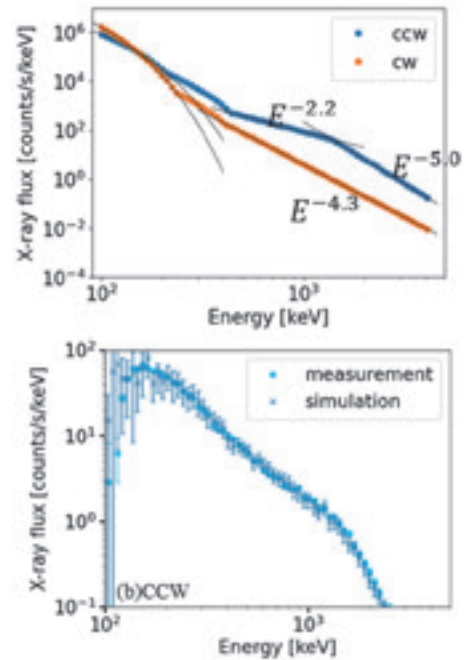


Fig. 4. (a) Source X-ray energy distribution obtained by the iterative retrieval method. (b) Comparison of measured distribution and simulated distribution for scintillator of the CCW sightline.

## Collaboration Works

長崎百伸, 小林進二, 稲垣滋, 門信一郎, 金史良, Univ. Wisconsin (アメリカ), Oak Ridge National Laboratory (アメリカ), Max Plank Institute (ドイツ), Stuttgart Univ (ドイツ), CIEMAT (スペイン), Australian National Univ., (オーストラリア), Kharkov Institute (ウクライナ), Southwest Institute of Physics (中華人民共和国), 先進ヘリカルシステムにおける周辺プラズマ・ダイバータ研究

長崎百伸, 西南物理研究所 (中華人民共和国), IPP, Greifswald (ドイツ), University of Wisconsin (アメリカ), 先進ヘリカルシステムにおける反射計を用いた電子密度・揺動解析

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長崎百伸, 小林進二, 稲垣滋, 門信一郎, 金史良, Wisconsin University (米国), CIEMAT (スペイン), Max-Planck Institute (ドイツ), 先進ヘリカルシステムにおける周辺揺動解析

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小林進二, 長崎百伸, 稲垣滋, 門信一郎, 金史良, CIEMAT (スペイン), Kurchatov Institute (ロシア), ORNL (アメリカ), 先進ヘリカル磁場配位の最適化に向けたネットワーク拠点形成

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長崎百伸, 核融合科学研究所・双方向型共同研究, 波動加熱による高速電子生成時の非熱的放射機構の研究

長崎百伸, 核融合科学研究所・双方向型共同研究, Ka バンドマルチチャンネル周波数コム型マイクロ波ドップラー反射計の開発

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小林進二, 核融合科学研究所・一般共同研究, ヘリオトロン型磁場配位を利用した統計加速の実験室シミュレーションの検討

## Financial Support

### 1. Grant-in-Aid for Scientific Research

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## 2. Others

長崎百伸, 三菱電機 (株), 非開示

長崎百伸, 自然科学研究機構, 2024 年度双方向型共同研究

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## Complex Plasma Systems Research Section

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## 1. Introduction

Nuclear fusion energy has some attractive features as a future option for the base-load electrical power source and thus magnetic field confinement fusion research is thus being conducted all over the world. To realize fusion reactor, there are however several urgent issues that need to be resolved. We are thus performing the Heliotron J experiment to solve these issues: for example, (1) improvement of plasma energy confinement, (2) enhancement of fueling ability. To solve these problems through experiments, excellent diagnostic tools are needed.

Results in FY2024 featured in this report are about the development of advanced diagnostics and discovery of improved confinement state.

## 2. Development frequency comb reflectometry

Two types of frequency comb reflectometer have been developed to observe particle avalanche, turbulence spreading, and staircase formation at the periphery of Heliotron J. One is for frequency comb Doppler backscattering measurements. One is for frequency comb Doppler backscatter measurements with a comb frequency bandwidth of 12-26 GHz and a comb spacing of 0.5 GHz. The corresponding O-mode cutoff density is  $0.2\text{--}0.9 \times 10^{19} \text{ m}^{-3}$ , and 24 spatial points in the peripheral region of a typical discharge with average density  $> 0.5 \times 10^{19} \text{ m}^{-3}$  are measured simultaneously. From these measurements, we aim to identify turbulence spreading by observing the spatio-temporal structure of the plasma flow velocity in the poloidal direction and micro density fluctuations. The other is for FM frequency comb reflectometer, in which the base frequency of the comb is swept at 0.5 GHz for 0.5 ms while maintaining the comb spacing. This allows us to observe the time-varying structure of the staircase by surveying a wide plasma region in a short period of time. In addition, by modulating the frequency in a triangular wave shape, the Doppler shift in the radial direction can be obtained from the difference in the beat frequencies of the incident and reflected waves in the phases where the frequency of the incident microwave is increasing (up) and decreasing (down). This Doppler shift can be used to identify convective ballistic propagation of density perturbations such as particle avalanche. Both systems use an ultra-fast digital storage oscilloscope to directly digitize the incident and reflected waves, and digital signal processing instead of conventional analog mixers and

fixed filters to enable very flexible and fast detection. Both systems have completed bench tests.

The backscattering system was applied to the Heliotron J experiment to observe the flow velocity, which can be estimated from Doppler frequency. Figure 1 shows the time evolution of the flow velocity. While the plasma is in steady state, the flow velocity has large fluctuations, but the average value is negative. It is necessary to confirm whether these large fluctuations are due to plasma or noise. When obtaining the Doppler frequency using the center-of-gravity method, there is a trade-off between frequency resolution and time resolution. If the time resolution is increased, the frequency resolution decreases, and the influence of noise increases in the region where the Doppler shift is small. Therefore, it is necessary to set the optimal resolution for both.

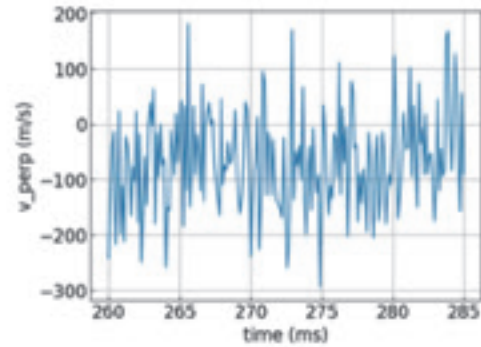


Fig. 1 Typical temporal evolution of flow velocity. Comb frequency component of 20 GHz was used.

The spatial structure of the time-averaged velocities is shown in Figure 2. Error bars indicate the standard deviation of the time variation. The horizontal axis is the injected Comb frequency, but the corresponding cutoff density is greater at higher frequencies, so the cutoff layer is located more in the center of the plasma. At lower frequencies, on the other hand, the cutoff layer is located at the periphery of the plasma, thus the velocity profile with respect to the injection frequency corresponds to the radial profile of the flow velocity. Figure 2 shows that the flow velocity, which is almost zero at the center, increases negatively toward the periphery of the plasma. If a flow is formed in the periphery, it is expected to suppress the edge turbulence.

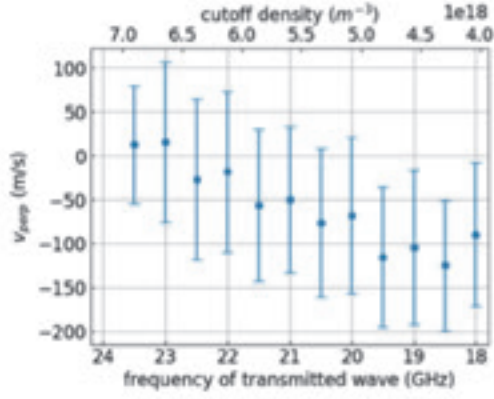


Fig. 2 Radial profile of time-averaged flow velocity.

### 3. Noble Island Identification Method

The stochastic structure in the peripheral region is a common feature in helical fusion devices due to the intrinsic resonant perturbation even in the vacuum magnetic field. A region with stochastic field lines and remanent islands is formed outside the last closed flux surface (LCFS). A numerical method to distinguish the closed magnetic surface from stochastic field lines is regarded as a useful tool to characterize helical confinement.

In this study, we implemented an algorithm that extracts a field line's dominant resonant poloidal/toroidal mode number  $m/n$  from the rotational transform. The magnetic islands or closed flux surfaces enclosed by the field line can be identified based on the shape of the  $m/n$  resonant periodic structure of the field line trajectory in the Poincaré plot. The method was applied to the vacuum field of a Heliotron J device as an example, as shown in Fig. 3.

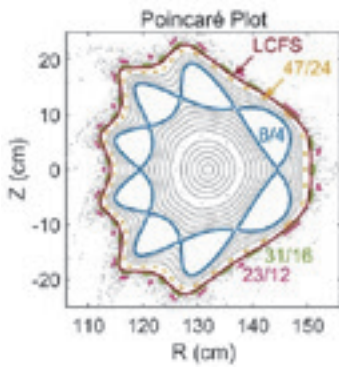


Fig. 3 Several identified magnetic islands with their mode number in Heliotron J.

As increasing the current in the inner vertical coils, large islands with a mode number of  $8/4$  emerged outside the LCFS and then were enveloped by the LCFS. We observed an increase in the volume of the confined region corresponding to the expansion of LCFS and its enveloping of external magnetic islands,

which is a preferable feature for better confinement of a helical fusion device. In principle, the volume of each island and stochastic region can be assessed quantitatively for various configurations using this method.

### 4. Fluctuation and turbulence study in High-confinement mode (H-mode)

The high-confinement mode (H-mode) is essential for improving the energy confinement time and is crucial for achieving fusion energy. However, compared to tokamaks, the achievable energy confinement improvement in stellarators/heliotrons is limited ( $\sim 30\%$ ). Therefore, we investigate fluctuations and edge cross-field transport during the H-mode transition in Heliotron J. The experiment is conducted in the so-called  $n/m = 4/9$  configuration, which is a favorable magnetic configuration for the H-mode transition. During the H-mode transition, we observe a characteristic low-frequency (LF) fluctuation with a frequency of approximately 14 kHz in several fluctuation diagnostics. As shown in Fig. 4, the LF fluctuation extends over a long radial range and correlates with the edge cross-field transport. The LF fluctuation also modulates the micro-scale density turbulence in the edge region, which is measured by Doppler reflectometry. At the H-mode transition, the edge density turbulence is reduced in association with the enhancement of the radial electric field. Additionally, the modulation of the turbulence amplitude by the LF fluctuation is suppressed, and the edge cross-field transport induced by the LF fluctuation is reduced, which is confirmed by the  $\text{Da}$  emission and divertor probes. On the other hand, the LF fluctuation itself persists after the H-mode transition, as detected by the electron cyclotron emissions, magnetic probes, and edge poloidal rotations. This indicates that the coupling between the LF fluctuation and turbulence plays a significant role in the edge transport driven by the LF fluctuation. Although the precise identification of the LF fluctuation remains unresolved and is left for future work, one possible candidate is pressure-driven MHD instability.

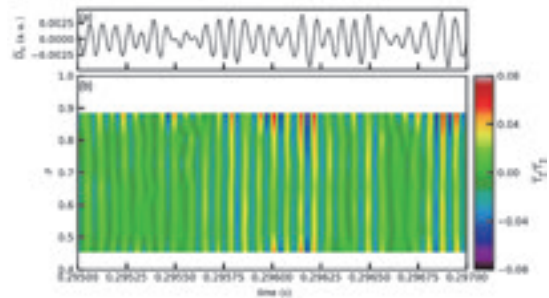


Fig. 4 Spatio-temporal evolution of low-frequency fluctuation. (a)  $\text{Da}$  emission indicating the edge transport, and (b) radially elongated temperature fluctuation.

## Collaboration Works

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門信一郎, 京都大学 × 堀場製作所包括連携協定「HONMAMON (ほんまもん) 共創研究」, フュージョンエネルギー確立を目指したプラズマ発光の可視化に関する研究

金史良, 核融合科学研究所・双方向型共同研究, 電子内部輸送障壁に対する磁場の三次元効果の解明

金史良, 核融合科学研究所・双方向型共同研究, 高エネルギー粒子駆動モードと背景乱流の相互作用

金史良, 核融合科学研究所・一般共同研究, 核燃焼プラズマに向けたトーラスプラズマの閉じ込め・輸送に関する研究

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### 1. Grant-in-Aid for Scientific Research

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### 2. Others

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## Advanced Energy Research Section

Daniel Alberto Scherson, Visiting Professor  
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### 1. Summary

The author spent one month at Kyoto University, Uji Campus in Prof. Nohira's group. Here is a summary of the study on the microparticle electrodes and single particle microbatteries using electrochemical and in-situ micro-Raman spectroscopic studies [1].

### 2. Introduction

The author discuss the study of the electrochemical, structural, and electronic properties of microparticles of energy storage materials for battery applications. This report summarizes efforts toward the development and implementation of methods for the in situ electrical, optical, and spectroscopic characterization of microparticles of materials including nickel hydroxide, zinc, carbon, and lithiated manganese and cobalt oxides. In the case of nickel hydroxide, the darker appearance of NiOOH compared to the translucent character of Ni(OH)<sub>2</sub> allowed for the spatial and temporal evolution of charge flow within spherical microparticles of Ni(OH)<sub>2</sub> to be monitored in real-time. In situ Raman scattering measurements involving single microparticles of zinc revealed that passive films formed in strongly alkaline solutions displayed a largely enhanced feature at approximately 565 cm<sup>-1</sup> ascribed to a longitudinal optical phonon mode of ZnO. This effect is associated with the presence of interstitial zinc and oxygen deficiencies in the lattice. Significant amounts of crystalline ZnO could only be detected for passive films formed at the same two potentials after the electrodes had been roughened by a single passivation-reduction step. Quantitative correlations were found in the case of LiMn<sub>2</sub>O<sub>4</sub> and KS-44 graphite between the Raman spectral properties and the state of charge. In the case of KS-44, a chemometrics analysis of the spectroscopic data in the potential region in which the transition between dilute phase 1 and phase 4 of lithiated graphite occurs made it possible to determine independently the fraction of each of the two phases present as a function of potential, without relying on the coulometric information. The author also developed methods for the assembly and electrochemical characterization of Zn|MnO<sub>2</sub> and nickel metal-hydride (NiMH) alkaline batteries incorporating single microparticles of the active materials.

Voltage-time profiles for constant current operation for both types of devices were similar to those of commercially available batteries involving the same chemistries. The ability to monitor the state of charge of individual particles based strictly on spectroscopic data is expected to open new prospects for visualizing the flow of charge within electrodes in Li-ion batteries.

### 3. Experimental Considerations

The author also discusses some experimental considerations for studying single-particle electrodes and assembling single-particle batteries. Manipulation of the microparticles is critical, requiring the placement of  $\mu$ -particles in contact with an electronic conducting support. Several strategies have been developed to manipulate  $\mu$ -particles, including: A glass capillary attached to a syringe, which allows fragile particles to be captured by suction and then delivered to the substrate surface. A sharp spear of hard metal that can be inserted into a softer malleable material. A  $\mu$ -disk electrode made out of a soft metal, into which hard particles can be embedded by applying pressure. The substrate should also exhibit a low and featureless capacitance within the potential range in which the charge storage material undergoes changes in redox state. Because of the small size of the materials involved, all manipulation and assembly operations must be performed under a microscope or telescope. Raman spectra were collected with a Raman 2000 system using a Verdi laser (532 nm) focused through an Olympus microscope as the excitation source. An all-glass, airtight, spectro-electrochemical cell was constructed for studies in nonaqueous electrolytes.

### 4. Single Microparticle Electrode Studies

The author also performed single microparticle electrode studies using both aqueous and non-aqueous systems.

#### 4-1. Aqueous Systems

Nickel hydroxide is a charge storage cathode material for rechargeable batteries that power many hybrid vehicles. In its pristine form, this material consists of transparent spheres with diameters in the micrometer range. In situ measurements of the resistance of a single microparticle of spherical-Ni(OH)<sub>2</sub> were performed as a function of the state of

charge. Metallic zinc electrodes in Zn|MnO<sub>2</sub> batteries consist of particles tens of micrometers in characteristic dimensions dispersed in a concentrated aqueous KOH solution mixed with a polymer. In situ Raman spectra were recorded by inserting an electrochemically sharpened tungsten tip coated with a thin polymer layer into a single zinc particle harvested from a Zn/MnO<sub>2</sub> battery.

#### 4-2. Nonaqueous Systems

The author's initial studies in nonaqueous electrolytes focused on the development of techniques for the electrochemical and Raman characterization of microparticles of lithiated transition metals oxides and carbonaceous materials, used as cathodes and anodes in commercial Li-ion batteries, in the absence of binders or conductivity enhancers. The strategy employed involved embedding a small number of  $\mu$ -particles into gold for the cathodes and thermally softened nickel for the anodes, using high pressure. Nickel is suited for the study of anodes as it does not form alloys with lithium at room temperature.

#### 5. Single Particle MicroRaman Spectro-electrochemistry

The author implemented methods to attach a single microparticle to the surface of a microelectrode, allowing for correlations between the current and the spectral features associated with that single microparticle. The results obtained for a single graphite microflake and for particles of LiMn<sub>2</sub>O<sub>4</sub>, both in polycrystalline and single crystal forms, are summarized in the article. Raman and electrochemical data for a single particle of KS-44, a commercially available graphitic powder, were in agreement with results reported by other groups using more conventional electrodes.

#### 6. Single Particle Electrode Microbatteries

The same strategies developed for the manipulation of charge storage  $\mu$ -particles were used to assemble single particle  $\mu$ -batteries. Electrochemical tests were carried out by immersing the entire assembly

into a 9 M KOH solution. A performance assessment of commercial batteries usually involves applying a constant current to the battery while monitoring the potential across it. The rates of discharge are given in terms of C units, where 1C corresponds to a full discharge in one hour. Voltage versus time discharge curves were recorded for Zn|MnO<sub>2</sub>  $\mu$ -batteries incorporating single particles of Zn and MnO<sub>2</sub>. Charge-discharge curves for Ni|MH microbatteries were recorded at three different C rates. The data collected from these single particle  $\mu$ -batteries closely mimic those found for technical devices. Single  $\mu$ -particle LiMn<sub>2</sub>O<sub>4</sub>/carbon batteries were assembled by placing individual cathode and anode particles onto two isolated microelectrodes encased in a single glass rod, which was inserted into the cell filled with electrolytes. The self-discharge of the battery was monitored by measuring its potential as a function of time after the two microparticles in the battery had been fully charged independently. The potential was very stable for about 10 hours.

#### 6. Concluding Remarks

These works aimed to develop methods of correlating the state of charge of battery microparticles with their spectroscopic characteristics, as a step toward monitoring the flow of charge within electrodes in actual operating batteries in real-time. The approach involves scanning the edge surface of an operating battery during charging and discharging using a Raman microscope. Further refinements will be required before the results of such experiments can be compared with theoretical models proposed in the literature to simulate the macroscopic behavior of actual devices.

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## Advanced Energy Research Section

Sadat Mohamed Rezk Khattab, Visiting Associate Professor,  
(Associate Professor of Microbial Biotechnology, Faculty of  
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### 1. Summary

The author spent the 2024 academic year (April 1, 2024 – March 31, 2025) as a Visiting Associate Professor, hosted by the Institute of Advanced Energy (IAE) within the Research Unit for the Realization of a Sustainable Society (RURSS), in collaboration with Professor Masato Katahira at his laboratory on the Uji Campus of Kyoto University.

In his report, the author presents recent developments in the efficient utilization of woody biomass.

### 2. Introduction

In pursuit of a sustainable future, we developed a biorefinery concept emphasizing environmental compatibility and process efficiency. In this approach, glycerol was employed as a green solvent to aid lignin decomposition during the initial biomass processing phase. Specifically, acid-catalyzed glycerolysis combined with microwave assistance was used to depolymerize lignin from sugarcane trash into antiviral compounds. These compounds significantly inhibited encephalomyocarditis virus (EMCV) replication in L929 cells without cytotoxic effects (1), as illustrated in Fig. 1.

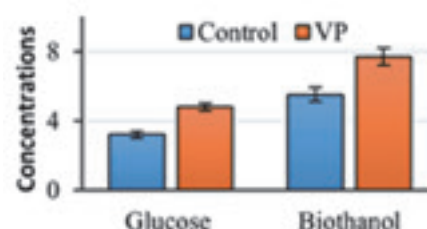


**Fig. 1.** Integrated biorefinery of sugarcane trash producing antiviral lignin and bioethanol via acid-catalyzed glycerolysis, saccharification, and co-fermentation by two mixed engineered *S. cerevisiae* strains: glycerol- and xylose-fermenting yeasts.

To streamline processing and reduce associated costs, the resulting pulp was enzymatically hydrolyzed and subsequently fermented using a mixture of two metabolically engineered *Saccharomyces cerevisiae* strains: one capable of co-

fermenting glycerol and glucose (2), and the other capable of fermenting xylose (3). Together, these strains efficiently converted the released glucose, xylose, and glycerol into bioethanol, achieving a high titer of approximately 10%, as depicted in Fig. 1.

In collaboration with Professor Katahira's lab, a versatile peroxidase (VP) enzyme was introduced as an eco-friendly biocatalyst for lignin degradation. Its application significantly enhanced enzymatic hydrolysis, fermentation rates, and overall bioethanol yield (4), as shown in Fig. 2.



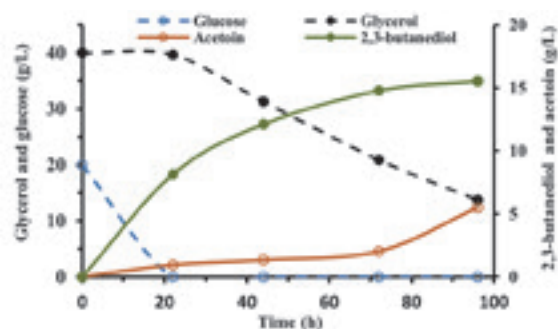
**Fig. 2.** Effect of versatile peroxidase (VP) treatment on glucose release during enzymatic hydrolysis and bioethanol production during fermentation, relative to control.

### 3. Results and achievements

#### (1). 2,3-butanediol production from glycerol

2,3-Butanediol (2,3-BDO) has attracted our attention due to its versatile applications as a bulk chemical. It serves as a precursor in the production of pharmaceuticals, cosmetics, synthetic rubber, inks, resins, perfumes, food additives, fuel additives, and even aviation fuel. Beyond industrial uses, 2,3-BDO also functions as an antifreeze agent, a plant growth stimulator, a drought resistance enhancer, and a biological control agent against plant pests and viruses.

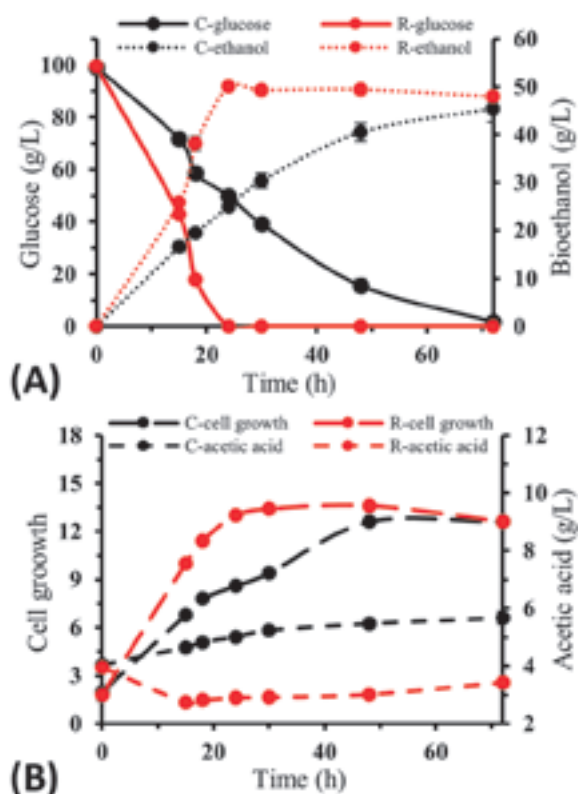
We successfully redirected the native fermentation end product of baker's yeast from ethanol to 2,3-butanediol through metabolic engineering of the fermentation pathway, using glycerol as the main substrate, as depicted in Fig. 3. This represents the first study to demonstrate the conversion of glycerol to 2,3-butanediol by baker's yeast (5).



**Fig. 3.** Time-course profiles of glycerol and glucose consumption and production of 2,3-butanediol and acetoin during fermentation by engineered *S. cerevisiae*.

## (2). A novel acetic acid co-utilization strategy during fermentation of glucose

Acetic acid, a byproduct of glucose fermentation, represents over 2% of the glucose consumed by our baker's yeast strain (**Fig. 4**). In second-generation bioethanol production from lignocellulosic biomass, additional acetic acid released during biomass decomposition may further hinder fermentation, as also seen in **Fig. 4**. To address this, we engineered a yeast strain that increases ethanol production rate by over 3.4-fold and enables acetic acid co-utilization. The strain performs well under low-oxygen conditions and shows ethanol-induced flocculation, aiding downstream recovery (6). The enhanced performance is demonstrated in **Fig. 4**.



**Fig. 4.** Glucose fermentation and acetic acid co-utilization by control (C) and recombinant (R) *S. cerevisiae* strains. (A) Glucose consumption and ethanol production. (B) Cell growth and acetic acid utilization. The recombinant strain shows faster glucose depletion, higher ethanol yield, improved growth, and active acetic acid consumption.

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## Advanced Energy Research Section

Geir Martin Haarberg, Visiting Professor  
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### 1. Summary

The author spent one and half months at Kyoto University, Uji Campus in Prof. Nohira group. A review of the most promising technologies for electrochemical reduction of CO<sub>2</sub> for carbon negative technologies is presented. Recent efforts to convert CO<sub>2</sub> into valuable carbon materials using molten salt electrolysis are summarized and discussed here.

### 2. Introduction

In order to achieve carbon neutrality by 2050, it is necessary to not only capture and store carbon dioxide (CO<sub>2</sub>) emissions (Carbon dioxide Capture and Storage; CCS), but also to capture and effectively utilize CO<sub>2</sub> (Carbon dioxide Capture and Utilization; CCU). Electrochemical conversion of CO<sub>2</sub> to carbon using molten salts is considered to be one of the most promising CCU technologies, and is attracting attention [1]. Several research groups have reported the electrodeposition of various carbon allotropes, such as graphite [2] and carbon nanotubes [3]. Prof. Nohira's group has reported that they obtained diamond and amorphous carbon [4]. This study summarizes the use of molten salt electrolysis for the synthesis of high-value-added carbon materials such as graphite from carbon dioxide.

### 3. Electrochemical approaches and their challenges

Methods for breaking down CO<sub>2</sub> into other carbon-containing substances include the reduction of CO<sub>2</sub> by electrolysis or photoelectrolysis in aqueous solutions [5-7]. One of the advantages of this process is that it can produce a variety of products, including alcohols, aldehydes, carboxylic acids, CO, and even hydrocarbons. These substances are important raw materials in the chemical industry, and it is beneficial to be able to synthesize such chemicals from carbon dioxide. Furthermore, this process can also be seen as an energy storage process that converts electrical and light energy into chemical energy. The fact that it can produce many different types of products can also be a disadvantage. The fact that it can produce many different types of products can also be a disadvantage. It is difficult to improve the selectivity of the desired products and maximize

the yield. Since most of the products remain in aqueous solution, it is always difficult to separate them efficiently and economically. The low solubility of CO<sub>2</sub> gas in water is also a problem. Furthermore, the greatest challenge is the presence of water as a solvent. The decomposition reaction of water is thermodynamically and kinetically more favorable than the CO<sub>2</sub> reduction reaction, so it leads to the generation of hydrogen rather than the formation of the desired substance.

### 4. Molten salts as promising electrolytes

Inorganic molten salts are promising for CO<sub>2</sub> electrolysis due to high CO<sub>2</sub> solubility, stability, and wide potential windows, with high temperatures aiding kinetically challenging C=O bond breaking. This can enable cost-effective electrode materials and the direct splitting of CO<sub>2</sub> into valuable carbon and oxygen. Despite its potential for carbon material production and environmental benefits, this method has been overlooked, partly due to existing carbon sources and process understanding. However, the growing demand for sustainable carbon material synthesis has renewed interest in molten salt CO<sub>2</sub> electrolysis as a key technology. Research is focusing on various molten salt systems like Li<sub>2</sub>CO<sub>3</sub> and CaCl<sub>2</sub>-based electrolytes to control the resulting carbon materials, with CaCl<sub>2</sub>-based salts offering advantages in stability, oxide solubility, and cost-effectiveness, despite requiring further investigation compared to other systems. LiCl-KCl, a long-established representative alkali metal chloride molten salt, is also promising.

### 5. Various carbon materials obtained by molten salt electrolysis

Early research in the 2000s explored carbon film deposition from molten carbonates, yielding mostly amorphous films controlled by carbonate ion diffusion. Hsu et al. previously reported CNT formation in molten LiCl via lithium intercalation of a graphite cathode, not CO<sub>2</sub> electrolysis [8]. Electrolytic CO production was also investigated using Ti cathodes in Li<sub>2</sub>CO<sub>3</sub> and stainless steel in CaCl<sub>2</sub>-CaO, with high CO<sub>2</sub> favoring CO evolution. Ren et al. later achieved controlled CNF production directly from CO<sub>2</sub> in molten carbonates with metal additives acting as nucleation sites [9]. Metal encapsulation in carbon nanostructures, like Ge in

CNTs, was also demonstrated using molten salt electrolysis. Furthermore, graphene sheets were produced from CO<sub>2</sub> in CaCl<sub>2</sub>-based melts, attributed to catalytic effects and CO microexplosions. Nanodiamonds were synthesized in LiCl with moisture and CO<sub>2</sub> injection, involving graphite exfoliation and a nanoreactor mechanism. Carbon nanospheres with unique morphologies were obtained from molten LiCl-KCl-CCO<sub>3</sub>, likely influenced by CO bubbles. Other carbon nanomaterials like nano-onions and 3D graphene scaffolds have also been synthesized via CO<sub>2</sub> electro-splitting in molten salts.

## 6. Reaction mechanism

While the synthesis of diverse carbon materials via CO<sub>2</sub> electro-splitting in molten salts under varying conditions has been demonstrated, research focusing on the underlying reaction mechanisms and detailed cathodic behavior remains scarce. Ito et al. explored the carbonate reduction and re-oxidation processes in LiCl-KCl melt [10], proposing a two-step electrochemical reduction mechanism. Using chronopotentiometry, they estimated the diffusion coefficient of carbonate ions at 450°C, and the activation energy was determined from the Arrhenius plot. Ge et al. investigated the carbon nucleation process in LiCl-Li<sub>2</sub>CO<sub>3</sub> molten salt, primarily using SEM analysis of electrolysis products [11]. Through short-term electrolysis under different conditions, they revealed that the morphology of the resulting carbon materials is influenced by both the substrate and the applied cell voltage.

## 7. Inert anodes

Inert anodes for CO<sub>2</sub> electrolysis in molten salts, categorized as metals, alloys, and oxides, each present distinct advantages and disadvantages. Metals and alloys generally offer superior physicochemical properties like electrical conductivity and mechanical strength but often suffer from poor stability, especially in halide molten salts, and some, like Ir or Pt, are cost-prohibitive compared to oxides. Given the significance of halides as electrolytes, developing stable oxide inert anodes is crucial. SnO<sub>2</sub> has shown relative stability in corrosive molten salt environments; however, its inherent physical limitations necessitate the use of additives like CuO and Sb<sub>2</sub>O<sub>3</sub> for electrode applications. SnO<sub>2</sub>-based electrodes have demonstrated reasonable inertness in various molten carbonate and chloride systems. Despite their accepted stability, their conductivity and mechanical strength remain inferior to metals. Furthermore, the formation of a poorly conductive

CaSnO<sub>3</sub> layer on SnO<sub>2</sub> electrodes in CaO-containing melts and its potential reduction by alkali/alkaline earth metals leading to electrode consumption pose additional challenges. To overcome these limitations, recent efforts have focused on developing composite oxide materials such as CaRuO<sub>3</sub>, CaTi<sub>1-x</sub>Ru<sub>x</sub>O<sub>3</sub>, and TiO<sub>2</sub>·RuO<sub>2</sub> inert anodes. While the high cost of Ru compared to SnO<sub>2</sub> is a significant drawback, these composite oxides have exhibited improved conductivity and commendable stability in both molten chloride and carbonate salts.

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## Functional Materials Science and Engineering Research Section

Y. Miyauchi, Professor

T. Nishihara, Junior Associate Professor

**1. Introduction**

Our research section focuses on the physical properties, functions, and energy applications of quantum materials that exhibit significant quantum mechanical effects, such as carbon nanotubes, two-dimensional semiconductors, and recently discovered topological materials. The aim is to create new technologies for highly efficient use of solar light/thermal energy that will contribute to the realization of a sustainable energy society. To understand the unique physical properties of these materials from the fundamental principles and extract superior functions that exceed the limits of conventional materials, we are promoting interdisciplinary research that covers basic sciences, including materials synthesis and condensed matter physics, as well as thermal, mechanical, electronic, and optical engineering along with the fabrication of integrated nanomaterials. Followings are main research achievements in the year of 2024.

**2. Coalescence of carbon nanotubes while preserving the chiral angles**

One of the most difficult challenges in the science of nanocarbons, such as carbon nanotubes and fullerenes, is the solid-state chemical reaction of sequentially cleaving and recombining numerous carbon-carbon bonds into new bonds with atomic-level precision. In particular, coalescing large nanocarbon molecules such as fullerenes and carbon nanotubes and converting them into even larger nanocarbon molecules (Figure 1) is known to be a difficult problem. For fullerenes, which are made up of 60 carbon atoms, it is known that two fullerenes can be coalesced together using a solid-state reaction (Figure 1(a)). Moreover, many fullerenes can be coalesced together and converted into nanotubes by confining them in a carbon nanotube and then heat-treating them at high temperatures. However, synthesizing many new nanotubes by coalescing carbon nanotubes together (Figure 1(b)), which are made up of several thousand to tens of thousands of carbon atoms, has been considered to be extremely difficult due to the large number of carbon-carbon bonds that have to be rearranged.

The electronic, optical, chemical, thermal, and mechanical properties of carbon nanotubes are strongly dependent on their geometric structure, which is defined by the diameter and chiral angle of the nanotube (Figure 1(a)). Therefore, to date, extensive research

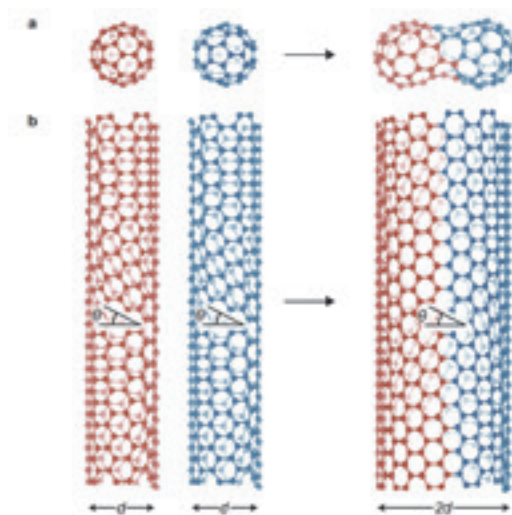


Fig. 1 Coalescence of fullerenes (a) and carbon nanotubes (b) into large coalesced molecules.  $\theta$  and  $d$  in (b) are the chiral angle and diameter, respectively.

has been conducted to achieve complete structural control synthesis and structural separation of nanotubes. However, the methods known to date have only enabled structure-selective synthesis and separation on a macroscale for thin nanotubes with diameters of around or less than 1 nm. This is because, for relatively thick nanotubes with diameters of around 1.3 nm or more, there are a very large number of geometrically feasible nanotube structures with similar diameters and properties, making it difficult to create or separate specific structures. The precise atomic-level carbon nanotube coalescence achieved in this study could be one of the most promising approaches to solving this problem. Since it is possible to produce high-purity samples of thin nanotubes, if it is possible to cause efficient coalescence reactions in them, it will be possible to achieve structure-selective synthesis of thick nanotubes using thin nanotubes as precursors. Furthermore, by using this reaction, it will also be possible to cause partial coalescence reactions by performing post-processing on nanotube aggregates, and to form  $sp^2$  covalent bonds between nanotubes. The technology of dramatically changing the macroscopic properties of nanotube assemblies through post-processing is expected to be very useful for various applications of nanotube assemblies on a macroscale.

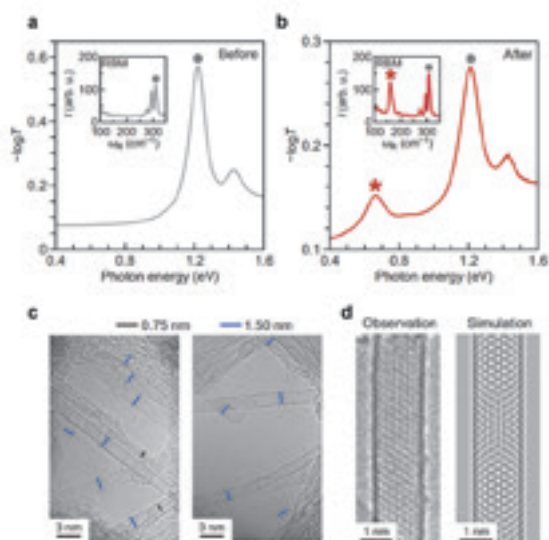


Fig. 2 Optical absorption ( $-\log T$ , where  $T$  is transmittance) spectra of (6,5) nanotube membranes before (a) and after (b) the vacuum heat treatment. The insets show the radial breathing mode (RBM) features of the Raman spectra.  $I$  and  $\omega_R$  are intensity and Raman shift. The gray filled circles indicate the peaks corresponding to the (6,5) nanotubes with  $d \approx 0.75$  nm and the chiral angle of  $\approx 27^\circ$ . (c) Transmission electron microscope (TEM) images obtained for the samples after vacuum heat treatment at  $900^\circ\text{C}$ . Bars with different colors indicates lengths corresponding to diameters of typical nanotubes found in the sample. (d) Comparison of the observed TEM image of the newly generated nanotube with  $d \approx 1.5$  nm, and simulated one for a (12,10) nanotube with  $d \approx 1.5$  nm and the chiral angle of  $\approx 27^\circ$ .

In this study, we discovered that thin carbon nanotubes can be efficiently coalesced into thicker nanotubes with twice the diameter using a simple heat treatment. This process involves heating structurally separated carbon nanotube aggregates at  $900\text{--}1000^\circ\text{C}$  under a reduced pressure of approximately  $5 \times 10^{-4}$  Pa (Figure 2). Furthermore, we found that in the presence of a trace amount of oxygen (around 10 Pa), oxygen acts as a catalyst and the temperature required for the coalescence reaction can be reduced to  $600^\circ\text{C}$ , which is more than  $1000^\circ\text{C}$  lower than the required heat treatment temperature reported in previous studies. The obtained thick nanotubes were confirmed by Raman scattering spectroscopy (Inset of Figure 2(a,b)) and aberration-corrected transmission electron microscopy (TEM, Figure 2(c,d)) to have doubled in diameter while maintaining the chiral angle of the precursor thin nanotubes. In addition, the measurement of the light absorption spectra confirmed that the nanotube coalescence reaction occurred efficiently throughout the entire nanotube film (Figure 2(a,b)). This study presents the first clear observation of exciton resonance peaks, demonstrating that the nanotubes synthesized through coalescence exhibit the characteristic

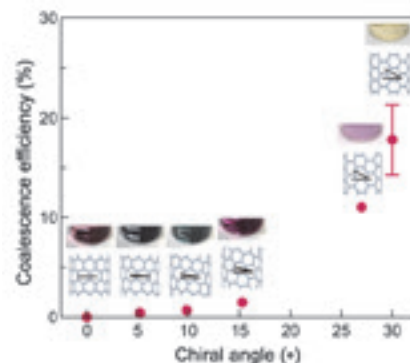


Fig. 3 Efficiency of the coalescence reaction as a function of chiral angle. The inset diagrams show photos (above) and partial structures (below) of the dispersion solution of thin carbon nanotubes (precursors). The angle between the black lines indicates the chiral angles of each nanotube structure.

optical properties of a specific carbon nanotube structure. This also clearly shows that the coalesced nanotubes exist in large numbers in the macroscopic sample, and that they actually have the electronic structure unique to a specific type of nanotube. It is noteworthy that only “armchair”-type and “near-armchair”-type nanotubes with a chiral angle of exactly or near  $30^\circ$  efficiently coalesce, and there is almost no coalescence of nanotubes with a small chiral angle (Figure 3). This result strongly indicates the structure dependence of the energy cost of coalescence reactions due to geometric factors. In this study, we also proposed a rational mechanism to understand the structure dependence of the observed nanotube coalescence efficiency.

This research has provided useful findings for various future applications of carbon nanotubes, such as the structure-selective synthesis of relatively thick carbon nanotubes, which has been difficult to achieve until now, and the modification of the physical properties of carbon nanotube aggregates through post-processing. Furthermore, from a broader perspective, this work presents a novel concept in nanomaterials science: macroscale materials with new physical properties can be created using precisely structure-controlled nanomaterials enabled by recent advances in nanotechnology.

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## Collaboration Works

宮内雄平, 浙江大学 (中華人民共和国), 高純度半導体型カーボンナノチューブ試料の作製とヘテロ構造化

## Financial Support

### 1. Grant-in-Aid for Scientific Research

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宮内雄平, 挑戦的研究 (開拓), 量子非平衡吸収体を用いた太陽光熱利用の原理的革新

西原大志, 基盤研究(B), 熱励起子ポラリトン状態の実現と熱放射制御原理の開拓

西原大志, 基盤研究(S), エネルギー科学展開に向けた量子熱光物性の基盤構築 (分担金)

Wu Hengkai, 特別研究員奨励費, カーボンナノチューブの量子熱光物性に基づく超高効率太陽光選択吸収体の実現

川上未央子, 特別研究員奨励費, 精密熱ふく射スペクトル制御に向けた異種ナノ物質薄膜の多重積層技術の開拓

### 2. Others

宮内雄平, (公財) 井上科学振興財団, 第 15 回エネルギー理工学研究所国際シンポジウム

西原大志, 科学技術振興機構 創発的研究支援, ナノシステム制御による太陽光利用の技術革新

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## Advanced Laser Science Research Section

T. Nakajima, Associate Professor

## 1. Introduction

We use our knowledge and skills on laser science to fabricate the functional materials and probe the associated dynamics without perturbing the various processes. This year we have developed two different techniques: The first one is to fabricate Ni electrodes with enhanced performance for hydrogen evolution reaction (HER) and the second one is to fabricate high quality holes/lines for various purposes including the micro/nano-structuring of electrodes.

## 2. Efficient hydrogen evolution reaction with Ni electrodes textured by nanosecond laser pulses

Efficient production and use of hydrogen gas is one of the important means for renewable energy production. Water electrolysis using renewable energy is a practical and cost-effective way to produce hydrogen gas. In particular, alkaline water electrolysis (AWE) has a certain advantage that non-noble metals such as Ni, Fe, or their alloys can be used for electrodes and catalysts. Among others Ni has a very nice resistance against corrosion for AWE.

To attain the high efficiency of hydrogen evolution reaction (HER) through AWE texturing the surface of the electrode or the catalyst layer is one way, where the purpose of texturing is to enlarge the electrochemical surface area (ECSA) without/with the additional functionalization of the surface. Several recent works report the texturing of electrode surface by femtosecond laser with improved electrochemical performance. The main drawback of the use of femtosecond laser texturing is its cost and long processing time. A nanosecond laser system is much lower than that of a femtosecond laser system, and if nanosecond laser texturing of electrodes works well, it can be a practical alternative to fabricate the efficient electrode. To our knowledge there are no studies on nanosecond laser texturing of electrodes for efficient HER.

The purpose of this work is to perform nanosecond laser texturing of Ni electrodes under the different conditions, and study their electrochemical performance.

We employ a nanosecond MOPA fiber laser at the wavelength of 1064 nm and a nanosecond DPSS laser at the wavelength of 355 nm to texture the Ni substrates. Hereafter we call them near-infrared

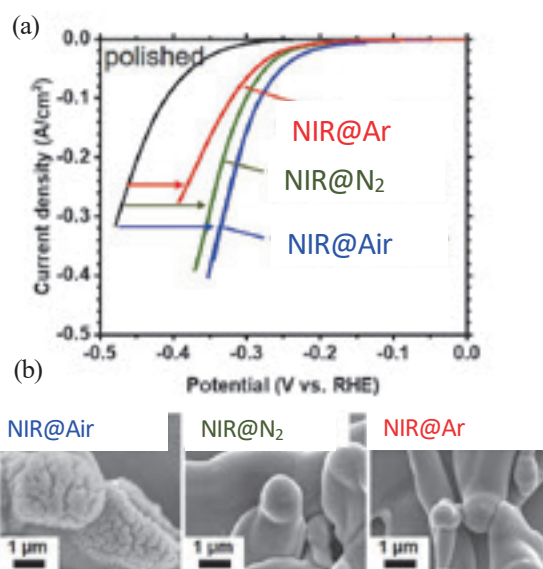


Fig. 1 (a) Polarization curves for HER by Ni electrodes textured with NIR (1064 nm) laser in air, N<sub>2</sub>, and Ar gas. (b) SEM images of the NIR laser textured Ni electrodes.

(NIR) and ultraviolet (UV) lasers, respectively. We texture the Ni substrates by nanosecond NIR and UV lasers in three different ambient gases, air, N<sub>2</sub>, and Ar at 2 atm, and study their electrochemical performance for HER by taking the polarization curves and compare them with that of polished Ni electrode (roughness R<sub>a</sub> ~ 5 nm).

Figure 1(a) shows the IR-corrected polarization curves for the NIR laser-textured electrodes in different ambient gases. We notice that the NIR@Air electrode shows the smallest overpotential. The main reason for this can be understood from the corresponding SEM images shown in Fig. 1(b), where the NIR@Air exhibit the most complicated cauliflower-like micro/nano-structures arising from the oxidation and hence volume expansion of the micro structures to result in cracks. Figure 2(a) shows the similar results for the Ni electrodes textured by UV laser. Different from the results for NIR laser texturing, UV@N<sub>2</sub> and UV@Ar show the smallest overpotential. Again, the main reason for this can be understood by the inspection of the corresponding SEM images (Fig. 2(b)): The UV@N<sub>2</sub> and UV@Ar electrodes exhibit very fine micro/nano-structures which enhances the ECSA by more than several tens of

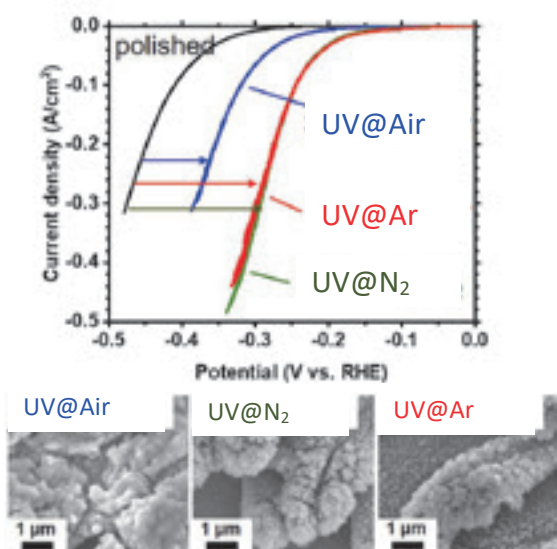


Fig. 2 (a) Polarization curves for HER by Ni electrodes textured with UV (355 nm) laser in air, N<sub>2</sub>, and Ar gas. (b) SEM images of the NIR laser textured Ni electrodes.

times. The SEM image of the UV@Air electrode shows less micro/nano structures because the volume expansion associated with oxidation collapses the very fine structures to result in the reduced ECSA compared with those of the UV@N<sub>2</sub> and UV@Ar electrodes. We have also tested the durability of the fabricated Ni electrodes to find that the electrode textured by UV laser is most durable (Fig. 3).

### 3. Fabrication of depth-controlled high quality holes and lines on a metal substrate by picosecond laser pulses

To add some functions through materials processing is one of the important technologies in materials science. In particular, laser processing is a simple technique that is applicable in both dry and wet environments. The underlying mechanism of laser processing is of course laser ablation. As well known, the use of ultrafast lasers has a clear advantage to reduce the thermal effect. However, this holds only for the single shot process, and if the hole/line is fabri-

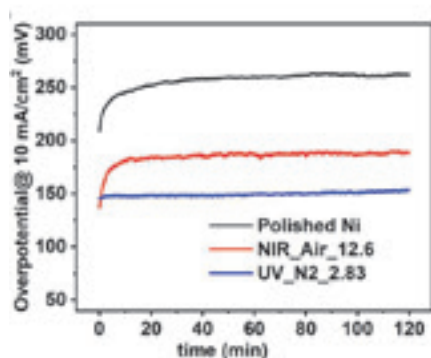


Fig. 3 Chronopotentiometric curves for the HER at the current density of -10 mA/cm<sup>2</sup>.

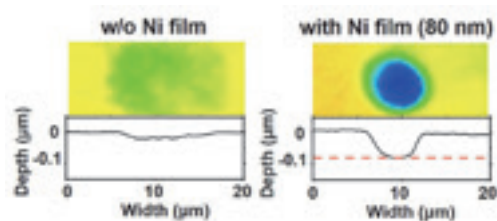


Fig. 4 False-colored morphologies and corresponding cross-sectional view of the fabricated holes on the Ni substrates (a) without and (b) with Ni film (thickness 80 nm)

cated on a metal substrate by multiple ultrafast laser pulses, the formation of ablation rims is inevitable even with ultrafast laser pulses.

Based on the above thoughts we propose a simple idea to use a metal substrate with a thin metal film rather than a bare metal substrate to fabricate high quality holes/lines. Because the interface between the metal film and metal substrate is not metallurgically bonded, the metal film may be selectively blown out by the single laser pulse at a much lower laser fluence than that in the case of a bare metal substrate. As a result the metal substrate remains intact to show a flat metal surface with a nearly vertical sidewall.

We use mechanically polished Ni substrates with and without 80 nm Ni films as targets to fabricate holes and lines by picosecond laser pulses at 532 nm. Figure 4 shows that we can fabricate a high quality hole on the Ni substrate with a Ni film. Fabrication of high quality lines is much more challenging, since it requires multiple irradiation of laser pulses to form a line, and every single laser pulse can produce a very small thermal effect to deteriorate the quality of lines. Nevertheless, we are able to fabricate high quality lines only if we choose the appropriate laser fluence to minimize the thermal effects, as shown in Fig. 5. It is very interesting to point out that the slope of the sidewall of the line fabricated on a Ni substrate with a Ni film is nearly vertical with an extremely flat bottom, as shown in the right panel of Fig. 5.

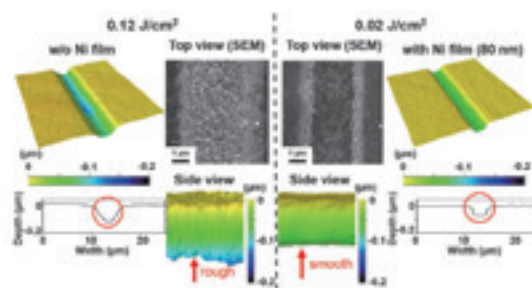


Fig. 5 False-colored morphologies and corresponding cross-sectional view of the fabricated lines on the Ni substrates (a) without and (b) with Ni film (thickness 80 nm)

## Financial Support

### 1. Grant-in-Aid for Scientific Research

中嶋隆，基盤研究(B)，電極表面のガス種依存濡れ性制御による電解効率の飛躍的向上

### 2. Others

中嶋隆，（公財）市村清新技术財団，水素社会の実現に向けた高性能光学式水素センサーの開発

中嶋隆，新エネルギー・産業技術総合開発機構，燃料電池等利用の飛躍的拡大に向けた共通課題解決型産学官連携研究開発事業／水素利用等高度化先端技術開発／常温水電解の実用化基盤研究プラットフォームの構築

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## Advanced Energy Structural Materials Research Section

K. Morishita, Associate Professor

K. Yabuuchi, Assistant Professor

Y. Chen, Researcher

**1. Introduction**

The development of robust materials and the implementation of reliable system management are crucial for ensuring the safe and efficient operation of advanced nuclear energy systems. This section explores the mission of establishing maintenance management methodologies and advancing materials research and development for cutting-edge nuclear energy systems, including fusion and fission reactors. Our research interests include:

**(1) Theory, modeling, numerical simulation, and data-driven science & technology:** Radiation damage in materials during irradiation occurs across various time and length scales. To gain a comprehensive understanding of the processes, a multiscale perspective and statistical approaches are essential. This section focuses on modeling material behavior under irradiation through a combination of computational techniques, including molecular dynamics (MD), ab-initio calculations, kinetic Monte-Carlo, rate-equation analysis, finite element method (FEM), and computational fluid dynamics (CFD). Recently, additional efforts have also been dedicated to integrating machine learning and data-driven methodologies to enhance this research.

**(2) Plant integrity analysis:** Structural integrity of a reactor pressure vessel (RPV) during pressurized thermal shock (PTS) events is crucial for the quantitative assessment of reactor safety. To evaluate this, we employ 3D computational fluid dynamics (CFD) and finite element method (FEM) simulations. The risk of RPV function loss is then quantified for optimizing maintenance strategies.

**(3) Effects of irradiation on microstructure and mechanical property changes:** High-energy particle irradiation generates oversaturated interstitials and vacancies, whose behavior drives microstructural evolution and may lead to the degradation of material's mechanical properties. Understanding the behavior of these point defects is essential for elucidating the mechanisms behind such property changes. In our research, we investigated microstructure evolution under irradiation through both experiments and computational modeling.

**2. Evaluation of cluster formation energy**

The safety of reactor pressure vessel (RPV) steel is a critical issue in the field of nuclear energy. In particular, atomic clusters in alloys influence the crystalline

properties of materials and accelerate the embrittlement of RPV steel. The growth rate of clusters depends on their energy, with size and structure being key factors. Therefore, to evaluate material embrittlement, it is essential to evaluate the cluster energy with different sizes and structures. However, the model of cluster structure has not yet been established. In this study, simulations were conducted to analyze clusters structural characteristics and formation energy.

Fig. 1 illustrates the stable structure of clusters with a size of 5, based on the minimum cluster formation energy. The analysis revealed that in Cu clusters, the lowest-energy state corresponds to atoms occupying first-nearest neighbor positions, gradually evolving into an octahedral structure. In contrast, Mn clusters exhibit a lowest-energy structure dominated by third-nearest neighbor atoms, showing a tendency to transform into a tetrahedral structure. Meanwhile, for Ni clusters, the lowest-energy state corresponds to atoms distributed at second-nearest neighbor positions, forming an expanded planar structure.

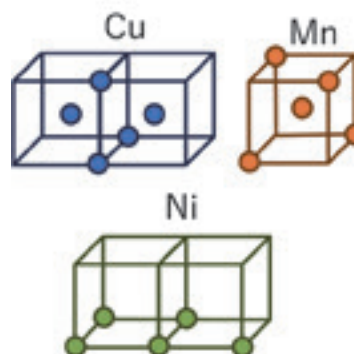


Fig. 1 Stable structure of defect clusters (Size 5)

**3. Effect of irradiation environments on microstructural evolution in Fe during irradiation**

Many studies have been done so far using numerical simulations to develop tools to predict material degradation under irradiation. However, many conventional predictions mainly evaluate the “average” behavior of the changes due to irradiation without considering the effects of fluctuations. The fracture behavior of a material does not depend on the average behavior of the material but rather is dominated by the material's property at its weakest point. Therefore, it is important to focus on fluctuations in defect microstructural evolutions.

In this study, the fluctuations in defect generation

and associated microstructural changes caused by irradiation are focused. The probability distribution function (PDF) of the number of point defects and that of the number of cascade clusters are obtained as a function of recoil energy using our MD simulations. The recoil energy spectrum for a specific neutron irradiation facility, e.g., high flux isotope reactor (HFIR) in ORNL, is then obtained using the nuclear library JENDL constructed by JAEA. Using these data evaluated here, the average and fluctuations of point defect generation rate and those of cascade cluster generation rate are evaluated individually for the HFIR. These rates are further employed as a source term in the rate theory equations to evaluate the microstructural changes of materials during irradiation. Our results show that in the early stage of irradiation, most of the defect clusters are generated by cascade clusters. As the irradiation progresses, the fluctuations flow into the larger-sized clusters, and the fluctuations in the larger-sized clusters increase. The swelling fluctuation exceeds 20%.

#### 4. Development of NNP potentials

The environment in nuclear reactors may cause irradiation damage to materials, resulting in changes of materials properties. Molecular dynamics methods are used to simulate radiation damage behavior. However, the accuracy of molecular dynamics method strongly depends on the accuracy of the potential function. The widely used empirical potential functions sometimes struggle to provide sufficient accuracy and reliability in irradiation damage simulations. Therefore, it is necessary to develop a highly accurate potential function. First principles calculations can provide higher accuracy but are limited by calculation speeds and applicable system sizes. With the advancement of artificial intelligence technologies, machine learning methods have been employed to learn potential energy data obtained from first-principles calculations, enabling the construction of Neural Network Potentials (NNP). This approach improves computational accuracy and efficiency while expanding the range of applicable systems.

To validate and evaluate the reliability of NNP method, this study tried to reproduce the empirical potential of BCC Fe using NNP method. Considering the high computational cost of generating data through first-principles calculations, a dataset was efficiently generated using the Mendelev potential of BCC Fe. The dataset consists of over 100 structures which included vacancies and SIAs. The dataset was used for machine learning to train the NNP. Furthermore, the structure of the neural network was optimized by adjusting parameters such as the number of layers, nodes, and epochs. This study explored and improved machine learning based NNP training methodologies and aims to apply them to the development of Fe-Mn-Ni-Si potentials, for RPV steels. In the future, this

research is expected to provide a foundation for the development of highly accurate and reliable interatomic potentials for irradiation damage research.

#### 5. Process Informatics of SiC Coating by CVD

In recent years, silicon carbide composites (SiC/SiC) have attracted attention as structural materials for accident tolerant fuel (ATF) and other gas-cooled and molten salt type small modular reactors (SMR) due to their excellent high temperature strength properties. However, it is known that the corrosion of SiC by high temperature and high pressure water is accelerated by irradiation, and it is essential to develop coating technology. For this problem, successful coating of dense and high-purity alumina and SiC on the surface of SiC/SiC planar specimens by the chemical vapor deposition (CVD) method has been reported. The major advantage of the chemical deposition method is that theoretically any complex geometry can be coated as long as the gas (gas phase raw material) can reach the surface. However, good quality film cannot be obtained unless the deposition parameters such as concentration, temperature, and flux of the raw material gas are optimized for each part or area. Therefore, the objective of this study is to search for optimal deposition parameters by machine learning of data obtained from experiments and fluid simulation data such as gas concentration, temperature, and flow velocity for each part in order to apply the CVD method to a three-dimensional structure.

So far, machine learning models have been created to predict each of the four parameters: film thickness, hardness, Young's modulus, and shear strength, for the four input parameters of temperature, density, pressure, and velocity gradient. Prediction curves were obtained by learning the results of experiments and fluid simulations. Since the number of data is not sufficient at this time, we plan to expand the data to search for optimal process conditions in the future.

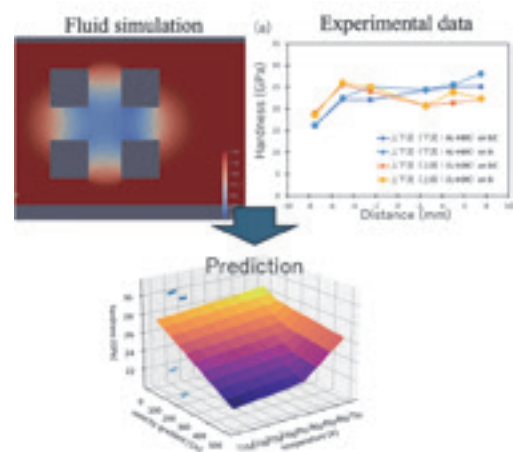


Fig.2 Prediction by machine learning

## Collaboration Works

森下和功, Chen Yuting, 学生, 国立研究開発法人量子科学技術研究開発機構・共同研究, 原子衝突カスケード損傷による材料マイクロ組織発達挙動への影響

森下和功, Chen Yuting, 学生, 国立大学法人福井大学原子力工学研究所・委託研究, MnNiSi に特化した計算機実験による MnNiSi クラスタ形成過程のモデル化

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## Optical Nano-science Research Section

K. Matsuda, Professor

K. Shinokita, Assistant Professor (until December, 31)

S. Matano, Assistant Professor (from March, 1)

## 1. Introduction

We are engaged in fundamental and applied research of nano-materials from a viewpoint of optics and material science. Our research aims to explore new physical and chemical phenomena leading to the applications of novel nano-materials including carbon nanotubes, layered transition metal dichalcogenides, perovskites for the efficient utilization of light energy and the development of future optoelectronic devices with ultra-low energy consumption. The followings are main the research achievements in the year of 2024.

## 1. Robotic mechanical exfoliation of two-dimensional semiconductors combined with Bayesian optimization

Recently, atomically thin two-dimensional (2D) materials including graphene, and monolayer transition metal dichalcogenides have attracted much attention in a variety of disciplines due to their electronic and optical properties that do not appear in their bulk crystals. Several methods for fabricating monolayer 2D semiconductors with thicknesses of only a few nanometers are mechanical exfoliation, chemical exfoliation, chemical vapor deposition, and so on. Among these methods, the mechanical exfoliation from bulk single crystals has been widely and frequently employed to fabricate the high quality graphene and monolayer 2D semiconductors.

Nevertheless, the mechanical exfoliation method itself is recognized as a simple process; however, it encounters significant bottlenecks due to the substantial manpower requirements. The process is composed of numerous steps, such as substrate cleaning, exfoliation of bulk single crystals, transfer of small flakes to the substrate, and monolayer detection, which make it challenging to efficiently prepare the large-area monolayer 2D materials over  $100\ \mu\text{m}^2$ . Moreover, the experimental conditions must be carefully selected from a vast number of potential parameters such as types of tape, folding time, peeling velocity, and so on, and the detailed microscopic mechanism of mechanical exfoliation itself has yet to be elucidated. At the present stage, the only ways are to wait for serendipity by repeating the many experimental trials, or to rely on experienced and skilled researchers to find large graphene and monolayer 2D materials. Recently, several advanced methods have been proposed to support the

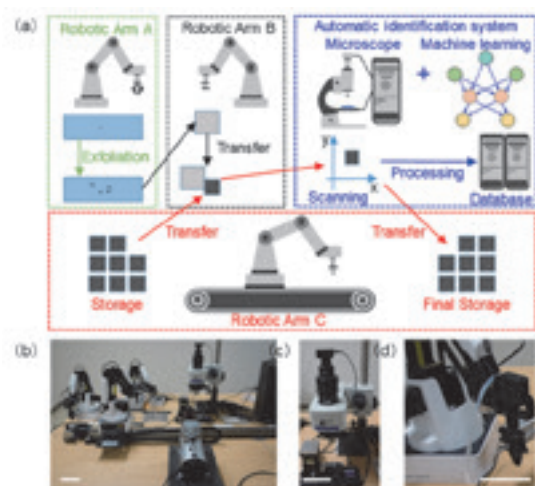


Figure 1 Schematics, functionalities, and photographs of the developed robotic system. (a) Schematic representation of the 2D monolayer exfoliation and searching process. Initially, the single crystals of 2D materials on the blue-tape are repetitively folded by the robotic arm-A. Subsequently, the small flakes of 2D materials are transferred to the Si substrate by the designed stamp of the robotic arm-B. An automatic detection system based on machine learning is employed to identify the 2D monolayers and catalogue their position in a database. Subsequently, the Si substrate with monolayer 2D materials is stored. (b-d) Photographs of (b) the entire system, (c) the optical automatic detection and identification platform, and (d) the blue-tape folding apparatus. The scale bars in the photographs correspond to 10 cm.

fabrication process by using machine learning to identify monolayer 2D materials and robots to automatically stack exfoliated 2D materials. These efforts represent the next generation of research aimed at combining robotic systems and machine learning to efficiently advance all research in the field of 2D materials on a large scale. In this context, it is strongly required to develop the efficient and highly reproducible strategy to fabricate the large-area and high-quality monolayer 2D materials by mechanical exfoliation, which would provide a significant impact on the wide range of research area in 2D materials fundamental research and development.

Figure 1a illustrates the schematics of the developed robotic fabrication and searching system for high-speed and reproducible mechanical exfoliation and detection of monolayer 2D semiconductors. The system is composed of robotic mechanical exfoliation, transfer from the mechanical exfoliation to detection,

and automatic searching and identification of monolayer 2D semiconductors equipped with a microscope. Figures 1b-d show the optical images of the whole robotic system we have developed, the optical automatic identification platform, and the folding device, respectively.

We attempted to efficiently explore the optimized experimental conditions for mechanical exfoliation by combining the developed robotic system with Bayesian optimization (BO), because the mechanical exfoliation is a simple process; however, it involves a huge number of experimental conditions. In BO, it is necessary to set parameters that have a significant impact on the results in order to efficiently explore a large parameter space, and the parameter space was set based on experiments conducted with the developed robotic system. Moreover, we systematically varied and selected the parameters that had a greater effect on the results of mechanical exfoliation. The type of blue-tape, number of blue-tape folding, peeling velocity, and number of transfer onto the blue-tape were selected, resulting in a total of 12,000 experimental conditions. Such a huge number of experimental conditions makes the tasks difficult for optimization without the support of data science approaches such as BO.

Several initial conditions should be provided to construct an appropriate model function for the BO algorithm. The initial ten experimental conditions are selected from all the experimental conditions (12,000 experimental conditions) using the D-optimization criterion to ensure that the characteristics among the parameters are not constant. Considering the results of these initial conditions, the appropriate kernel function<sup>45</sup> is used to construct the model function. Moreover, the acquired number and integrated total area of monolayer flakes were treated as indicators. However, these are not suitable for the BO algorithm due to their high variance even under the same experimental conditions. A new index that indicates the quality of mechanical exfoliation results needs to be designed according to the target, i.e., efficiently acquiring many monolayer flakes larger than  $100 \mu\text{m}^2$  suitable for the fabrication of devices and vdW heterostructures.

We introduced the new evaluation index as a large exfoliated area performance (LEAP). The LEAP is an index that indicates how large and many monolayer WSe<sub>2</sub> can be efficiently obtained with a threshold areal size of  $100 \mu\text{m}^2$ . The LEAP is composed of the value of monolayer appearance probability (MAP: the percentage of monolayers produced that exceed  $100 \mu\text{m}^2$ ) and large exfoliated area score (LEAS: the average size of monolayers that can be fabricated), corresponding to indices for the number and area of monolayer flakes, as described below,

$$\text{LEAP} = a_1 \frac{n}{N} + a_2 \frac{S}{N \times 100}$$

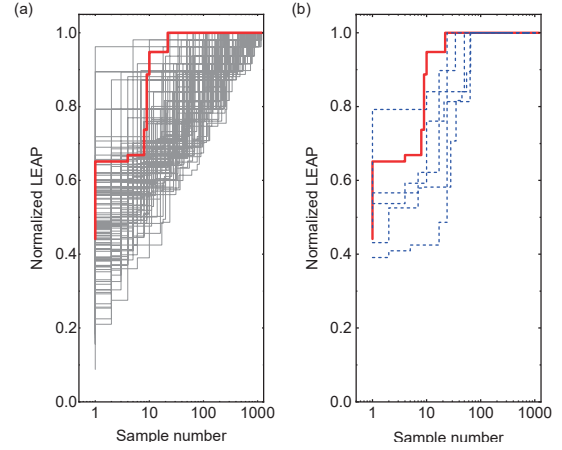


Figure 2 Convergence performances in Bayesian optimization and random simulation. (a) Comparison of convergence performance between Bayesian optimization and random simulation. Grey lines and red line show the simulated traces by the random simulation, and the Bayesian-optimization, respectively. (b) Blue lines show the fastest five traces that reached to convergence in the random simulations. The score of LEAP are normalized to compare.

where the first term and second term correspond to MAP and LEAS, and  $a_{1(2)}$  are coefficient of MAP and LEAS, respectively, and  $N$  and  $n$  indicate the total number of WSe<sub>2</sub> monolayers larger than  $10 \mu\text{m}^2$  and  $100 \mu\text{m}^2$ , respectively. Also,  $S$  indicates the total area of WSe<sub>2</sub> monolayers larger than  $10 \mu\text{m}^2$ .

We simultaneously compared the results of BO with predicted random simulation in order to show the effectiveness of BO in the mechanical exfoliation process. Figure 4a shows a simulated benchmark of calculated LEAP by selecting the parameters in the framework of random simulation and BO. In the random simulation, 1,200 randomly selected conditions are chosen from all experimental conditions of 12,000, and 100 patterns are prepared to compare how quickly the maximum score of LEAP is reached to the score derived in BO. The predicted score in the random simulations is calculated based on a model function constructed in BO. Moreover, the maximum LEAP score in each simulation pattern is normalized to compare the number of trials required to reach the maximum score. The LEAP in BO reaches the maximum score after only 23rd trials, as shown in Figure 2a, while the LEAP in random simulation reaches the maximum score after an average of 485 trials. Figure 2b shows the evolution of predicted LEAP in BO and random simulation for the selected fastest five patterns, as a function of trials. These results clearly show that the Bayesian algorithm is much more effective and a superior optimization than the random algorithm for the optimization of mechanical exfoliation process. These results suggest that a robotic system using the BO algorithm for mechanical exfoliation would be useful for efficient fabrication of many other monolayer 2D materials.

## Collaboration Works

松田一成, University of Bordeaux (フランス), 単一ナノ物質における先端分光

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### 1. Grant-in-Aid for Scientific Research

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## Chemical Reaction Complex Processes Research Section

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## 1. Introduction

In this research section, we study electrochemistry and materials science. We also apply them to the development of new metal production methods, new metal plating processes, and new rechargeable batteries.

In this fiscal year, we have researched a new terbium production method using molten salt electrolysis, a tungsten film plating process using molten salts, and potassium-ion batteries using ionic liquids.

## 2. Development of New Tb Production Method Using Molten Salt Electrolysis

Terbium (Tb) is added to neodymium magnets used in motors for electric vehicles (EVs) and hybrid electric vehicles (HEVs) to prevent deterioration of their magnetic properties at high temperatures. Tb metal added to neodymium magnets is currently produced by metallothermic reduction. As a novel method to produce Tb metal, we focused on the electrochemical reduction of Tb(III) ions using a liquid Zn electrode, followed by volatile separation and vacuum melting to obtain metallic Tb. In this fiscal year, we investigated the electrochemical formation of Tb–Zn alloys at the liquid Zn electrode in molten LiF–CaF<sub>2</sub>–TbF<sub>3</sub> at 1123 K.

Cyclic voltammetry was performed in molten LiF–CaF<sub>2</sub>–TbF<sub>3</sub> (0.30 mol%) at 1123 K. At the Mo flag electrode, a reduction current and the corresponding oxidation current peak were observed at approximately 0.2 V (vs. Li<sup>+</sup>/Li), which were attributed to the deposition and dissolution of Tb metal, respectively. At the liquid Zn electrode, the reduction current increased at approximately 1.1 V. Since the potential was considerably more positive than the Tb metal deposition potential, the reduction current is likely due to the formation of a Tb–Zn alloy. To confirm the electrochemical formation of the Tb–Zn alloy, galvanostatic electrolysis was performed in molten LiF–CaF<sub>2</sub>–TbF<sub>3</sub> (1.0 mol%) at 1123 K using a liquid Zn electrode at  $-100 \text{ mA cm}^{-2}$  for 125 min. Fig. 1 shows the cross-sectional SEM image of the sample after electrolysis [1]. The compositions at points (1) and (2) were determined by EDX analysis and exhibited Tb:Zn ratios of 21:79 and 16:84 mass%, respectively. These results indicate that a liquid Tb–Zn alloy was successfully formed by galvanostatic electrolysis at  $-100 \text{ mA cm}^{-2}$  using a liquid Zn electrode.

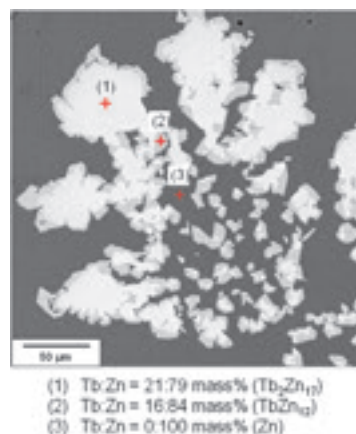


Fig. 1. Cross-sectional SEM image with the EDX analysis results of the sample after galvanostatic electrolysis in molten LiF–CaF<sub>2</sub>–TbF<sub>3</sub> (1.0 mol%) at 1123 K [1].

## 3. Development of W film Plating Process Using Molten Salt Electrolyte: Effect of O<sup>2-</sup> Ion Concentration on the Crystal Structure of W Films

Tungsten (W) is a metal with superior properties, such as heat resistance, high strength, and low thermal expansion. Therefore, there are many applications. In particular, W is expected to be used as divertor material in nuclear fusion reactors. However, its hardness and brittleness make it difficult to process into complex shapes and conventional tungsten processing methods are costly. Thus, the electrodeposition in molten salts was explored as an alternative processing method [2]. We have succeeded in electroplating extremely smooth tungsten films by electrodepositing  $\beta$ -W, which is a metastable phase [3]. We have also investigated the relationship between the crystal structure of electrodeposited W and the oxygen content in the film, and reported that 1.82–6.65 at% oxygen is present in electrodeposited  $\beta$ -W films [4]. We predicted that the existence of O<sup>2-</sup> ion in molten salt has an influence on the crystal structure. To investigate the effect of O<sup>2-</sup> ion, W electrodeposition was performed in molten CsF–CsCl–WCl<sub>6</sub> at 823 K before and after the addition of Li<sub>2</sub>O.

W was electrodeposited in molten CsF–CsCl–WCl<sub>6</sub> (1.0 mol%) at 1.2 V vs. Cs<sup>+</sup>/Cs for 2 min and in molten CsF–CsCl–WCl<sub>6</sub> (1.0 mol%)–Li<sub>2</sub>O (1.0 mol%) at

$-20 \text{ mA cm}^{-2}$  for 75 min. Fig. 2 shows the appearance and SEM images of the electrodeposited sample. The sample obtained before the addition of  $\text{Li}_2\text{O}$  (Fig. 2(a)) was grey in color, and granular deposits were observed on the surface. On the other hand, the sample obtained after the addition of  $\text{Li}_2\text{O}$  (Fig. 2(b)) had a black metallic luster, and no granular deposits were observed on the surface. XRD analysis confirmed that the sample obtained before the addition of  $\text{Li}_2\text{O}$  was  $\alpha$ -W, and that the sample obtained after the addition of  $\text{Li}_2\text{O}$  was  $\beta$ -W. From the above, it is suggested that the surface structure and crystal structure of the electrodeposited W film are affected by the existence of oxide ions.

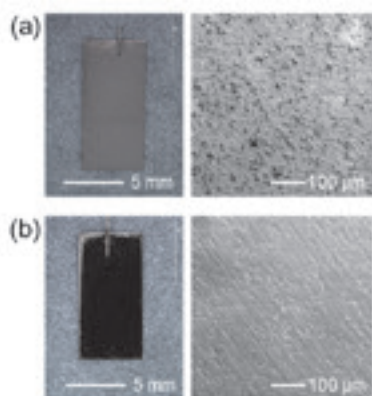


Fig. 2 Optical and SEM images of the samples obtained by (a) potentiostatic electrolysis at 1.2 V for 2 min in molten  $\text{CsF}$ – $\text{CsCl}$ – $\text{WCl}_6$  (1.0 mol%) and (b) galvanostatic electrolysis at  $-20 \text{ mA cm}^{-2}$  for 75 min in molten  $\text{CsF}$ – $\text{CsCl}$ – $\text{WCl}_6$  (1.0 mol%)– $\text{Li}_2\text{O}$  (1.0 mol%) at 823 K.

#### 4. Development of Potassium-ion Batteries Using Ionic Liquid Electrolytes

Towards the further spread of renewable energy resources such as solar and wind power, it is necessary to install electric load leveling systems combined with large-scale batteries. Although lithium-ion batteries are being used in electric vehicles, there are remaining risks of supply instability of scarce resources such as lithium, cobalt, and nickel. Moreover, flammable and volatile organic solvents used in electrolytes may induce serious accidents of large-scale batteries. Thus, we have developed potassium-ion batteries with ionic liquid (IL) electrolytes because potassium resources are abundant in the Earth's crust and ILs possess high safety of nonflammability and negligible volatility [5]. Instead of cobalt and nickel used in lithium-ion batteries, we have focused on iron- and manganese-based positive electrode materials [6,7].

Fig. 3 shows charge–discharge performance of  $\text{K}_{0.46}\text{MnO}_2$  positive electrode using  $\text{K}[\text{FSA}]$ – $[\text{C}_3\text{C}_1\text{pyrr}][\text{FSA}]$  IL electrolyte (FSA = bis(fluorosulfonyl)amide,  $\text{C}_3\text{C}_1\text{pyrr}$  = *N*-methyl-*N*-propylpyrrolid-

dinium) at 298 K. The  $\text{K}_{0.46}\text{MnO}_2$  electrode exhibits stable discharge capacities of 55–60  $\text{mAh g}^{-1}$  in the initial 50 cycles. Although the capacity gradually decreases to 50.3 and 39.4  $\text{mAh g}^{-1}$  after 100 and 400 cycles, respectively, no significant increase of voltage polarization is confirmed in discharge curves, indicating the feasibility of  $\text{K}_{0.46}\text{MnO}_2$  positive electrode in the IL electrolyte.

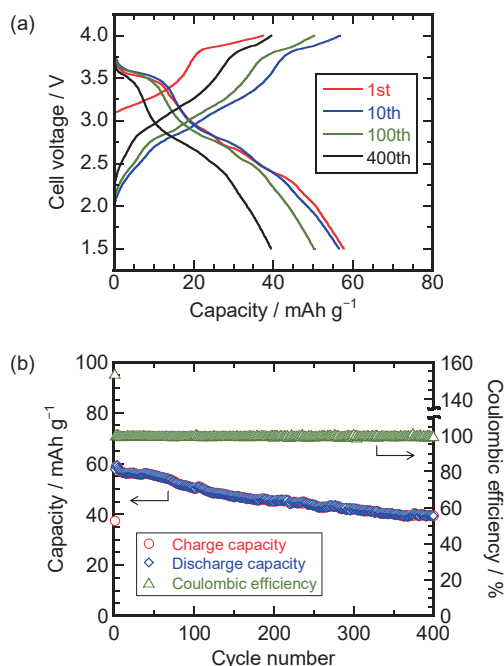


Fig. 3. (a) Charge–discharge curves and (b) cycling properties of  $\text{K}_{0.46}\text{MnO}_2$  positive electrode at 100  $\text{mA g}^{-1}$  [7]. Electrolyte:  $\text{K}[\text{FSA}]$ – $[\text{C}_3\text{C}_1\text{pyrr}][\text{FSA}]$  (molar fraction:  $x(\text{K}[\text{FSA}]) = 0.20$ ). Temperature: 298 K.

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## Molecular Nanotechnology Research Section

H. Sakaguchi, Professor  
T. Kojima, Associate Professor  
S. Nobusue, Assistant Professor

### 1. Introduction

Nanotechnology is essential for highly efficient energy use. Our group studies the basics of assembling small molecules into advanced materials and devices in energy sector with high efficiency. We have already developed several unique techniques which are totally new molecular assembling methodologies such as ‘Electrochemical Epitaxial Polymerization’ and ‘Two-Zone Chemical Vapor Deposition (2Z-CVD)’, which enable the production of molecular wires on metal surfaces from small molecules. By using these techniques, organic electronic devices such as field-effect transistors and organic solar cells will be developed.

The main research achievements in Molecular Nanotechnology Research Section in 2024 are described below.

### 2. Vectorial On-Surface Synthesis of Edge-functionalized Graphene Nanoribbon

Graphene nanoribbons (GNRs) have shown great potential for electronics, optoelectronics, and photonics. These properties strongly depend on their width and edge structure. Therefore, precisely controlled width and edge structure is required for desired properties. Bottom-up synthesis of GNRs is one of the suitable methods to satisfy these requirements because of the definition of their edge structures and widths by the shape of precursors. Atomically precise synthesis of armchair-edged GNRs has already been achieved under ultra-high vacuum (UHV) condition. However, symmetric GNRs via UHV deposition method composed of unfunctionalized hydrocarbons have limited applications, making them difficult to apply in organic electronic devices. To develop devices, high-yield production of assembled GNR films, separation and device fabrication are required.

We have developed 2Z-CVD to produce densely packed, parallelly aligned self-assembled GNRs on Au(111) under low vacuum condition of 1 Torr from halogenated polycyclic aromatic hydrocarbon (PAH) precursors. This technique successfully produced a series of armchair-edged GNRs in high yield. The attractive features of this method originate from an independent temperature control of the radical-generation

process (zone 1) and the growth process (zone 2), which afforded GNRs in high yield without using UHV conditions. The precursor was sublimated to the Au(111) substrate in zone 2 by passing through zone 1 in a quartz tube. The mechanism is supposed to involve radical generation in zone 1, polymerization of the radicals on the Au(111) substrate in zone 2, and subsequent dehydrogenation to form GNRs.<sup>1</sup>

Additionally, we have successfully synthesized cove-edged GNR by combining a newly designed precursor with the 2Z-CVD method.<sup>2</sup> These precursor molecules have a Z-shaped structure, where two *p*-terphenyls connect at a central hinge. They exhibit rigidity and flexibility, deforming on a metal substrate to transform from achiral to chiral molecules that recognize and assemble. They subsequently undergo a polymerization reaction, resulting in generation of homochiral polymers. Then, it was converted into cove-shaped GNRs with dehydrogenation reaction at 500 °C in high yield. We named "dynamic chirality" the phenomenon of molecular deformation on a metal substrate, resulting in chiral conformation from achiral one.

Recently, GNRs having an asymmetrically functional substitution at each edge have attracted much attention due to the realization of ferroelectric or ferromagnetic properties predicted by theory.

However, it has been difficult to produce them because of the decomposition of functional substitutions at high-temperature process during the dehydrogenation reaction. To address the drawback, we have developed brand new methods, “Vectorial on-surface synthesis” and “Molecular-Vapor-Assisted Low-Temperature Growth (MVLTG)”.

“Vectorial on-surface synthesis” is a method for creating stereoregular controlled polymers by extending the “dynamic chiral” principle from symmetric to asymmetric shapes. After producing precursor polymers using the 2Z-CVD method with asymmetric Z-shaped precursor, hydrogen acceptor was fed into the reactor (evacuated quartz tube), which promoted dehydrogenation reaction resulting in giving asymmetrical GNRs without decomposition of functional substitution at a lower temperature.<sup>3-5</sup>

### 3. Electrochemical on-surface synthesis of a strong electron-donating graphene nanoribbon catalyst

On-surface synthesis of edge-functionalized graphene nanoribbons (GNRs) has significant attention. However, on-surface synthesis of GNR having functional substitutes by ultra-high vacuum deposition method is difficult because of the decomposition of these substituents at high temperature of 300~500 °C, as we mentioned above.

To overcome these challenges as another method, we developed an on-surface electrochemical approach that employs redox reactions of asymmetric precursors at an electric double layer, where a strong electric field is concentrated at the liquid-solid interface. With this method, we successfully synthesized layer-by-layer growth of electron-donating GNRs on electrodes at temperatures below 80 °C without compromising functional groups.<sup>6</sup>

Moreover, we demonstrated that applying high voltage triggers a previously unknown heterochiral dicationic polymerization. The resulting electrochemically synthesized GNRs exhibit some of the highest electron-donating properties reported to date, enabling exceptional catalytic activity in silicon etching that outperforms that of noble metals. Additionally, these GNRs show superior photoconductive properties. Our technique opens up new opportunities for the development of various edge-functionalized GNRs with tailored electronic and catalytic properties.

### 4. Janus graphene nanoribbons with localized states on a single zigzag edge

We designed an asymmetric Z-shaped precursor molecule to synthesize asymmetric zigzag-edged GNRs using vectorial on-surface synthesis based on the principle of "dynamic chirality". In this design, one side of the Z-shaped structure is a diphenyl unit, while the other side is a methylphenanthrene unit, forming a zigzag edge structure, which is expected to be the molecules to deform into a shape suitable for reactions on a metal substrate, and to assemble in one direction while recognizing each other. The asymmetric Z-shaped precursor was partially aligned in one direction and polymerized to form a precursor polymer in an atactic manner, consequently occurring via their dehydrogenation at high temperatures, resulting in asymmetric zigzag-edged GNRs. The structure of the synthesized GNRs was confirmed through atomic-level analysis using scanning tunneling microscope (STM) and atomic force microscope (AFM), revealing that the molecules were polymerized in one direction as intended, resulting in GNRs that had zigzag edges on only one side.

Additionally, the density of state mapping using scanning tunneling spectroscopy (STS) demonstrated the

localization of electron spins at the zigzag edge. Furthermore, we conducted theoretical calculations on the electron spin density of asymmetric zigzag-edged GNRs assuming ferromagnetism, with results that matched the experimental, establishing that these asymmetric zigzag-edged GNRs are the ferromagnetic GNRs. We named the newly developed ferromagnetic GNRs "Janus GNRs (JGNRs)" inspired from the two-faced god from Greek mythology. Moreover, we demonstrated that the different magnetic modes of JGNRs, such as ferromagnetism, ferrimagnetism, and antiferromagnetism, can be controlled by adjusting the spacing of the protrusions in the coved structure.<sup>7</sup>

### 5. Photo-Assisted Bottom-Up Synthesis of Orange Phosphorus

A tubular strand of phosphorus composed of vectorially aligned pentagons has been theoretically predicted as a new allotrope of phosphorus with a polar structure, expecting potential applications. However, it has not been successfully synthesized yet due to the difficulty of creating isolated strands to avoid inter-chain bonding. We produced an allotrope named "orange phosphorus" using a photo-assisted synthesis from an amorphous film of solution-processable Na<sub>2</sub>P<sub>16</sub> precursors.<sup>8</sup> A green laser irradiation initiated the phase transition of precursors, inducing chemical reactions like topochemical polymerization and rearrangement, creating a 1D chain of orange phosphorus. 3D electron diffraction crystallography showed that the molecular structure of orange phosphorus consists of one-dimensional polar pentagonal-tubes made up of [P<sub>8</sub>]P<sub>2</sub>[ repeat units. Orange phosphorus demonstrates excellent piezoresistivity due to its high strain-sensitive 1D chain structure, showing strain-induced Raman shifts. Its gauge factor exceeds those of 2D materials such as black phosphorus and transition metal dichalcogenides. These findings indicate that orange phosphorus has great potential for use in strain sensor applications.

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## Biofunctional Chemistry Research Section

E. Nakata, Associate Professor

P. Lin, Assistant Professor

**1. Introduction**

A transition to renewable energy technologies requires new chemistry to learn from nature. For almost 3 billion years, nature has developed fantastic solutions to convert solar energy into chemical energy and to use it in an exceptionally efficient way. Our challenge is to understand nature's efficient bioenergetic processes and to design bio-inspired energy utilization systems. The research interests of our group focus on the design of biomacromolecules and their assemblies for molecular recognition, catalysis, and signal transduction in water, the solvent of life. We take synthetic, organic chemical, biochemical and biophysical approaches to understand biological molecular recognition and chemical reactions. Proteins and protein/nucleic acid assemblies are explored to realize the biomimetic function of biological systems, such as visualization of cellular signals by fluorescent biosensors, directed self-assembly of peptides and proteins to build up nano-bio materials, tailoring of artificial receptors and enzymes based on the complex of biofunctional molecules such as nucleic acid, peptides and/or proteins, and reconstitution of the functional assemblies of receptors and enzymes on the nanoarchitectures. The following are the major research achievements in the fiscal year 2024.

**2. Self-Assembled Fluorophore-Based Probe for Efficient Detection of Endogenous Nitroreductase Activity in *Escherichia Coli***

Fluorescent probes are functional molecules whose fluorescent properties are transformed as a response to specific stimuli. Understanding the mechanisms of these transformations is essential for the design of these stimuli-responsive fluorescent probes. A rational design strategy has been developed to construct stimuli-responsive supramolecular cluster fluorescent probes. They operate by a new mechanism called self-assembly induced lactone formation (SAILac) to control the fluorescence properties of SNARF, an asymmetric xanthene fluorophore. Here, to expand SAILac applicability, the structure-activity relationship of the fluorophore scaffold is studied. SNARF-OBn( $pNO_2$ ), designed as nitroreductase-reactive fluorescent probe based on the SAILac mechanism, is selected as the initial structure. As the result

of the structure-activity relationship studies, a new nitroreductase-reactive fluorescent probe, Rhodol-OBn( $pNO_2$ ), is created, having a superior signal-to-noise (S/N) ratio with higher reactivity toward nitroreductase than the original probe. By using Rhodol-OBn( $pNO_2$ ), the activity of endogenous nitroreductase in *Escherichia coli* is successfully detected.

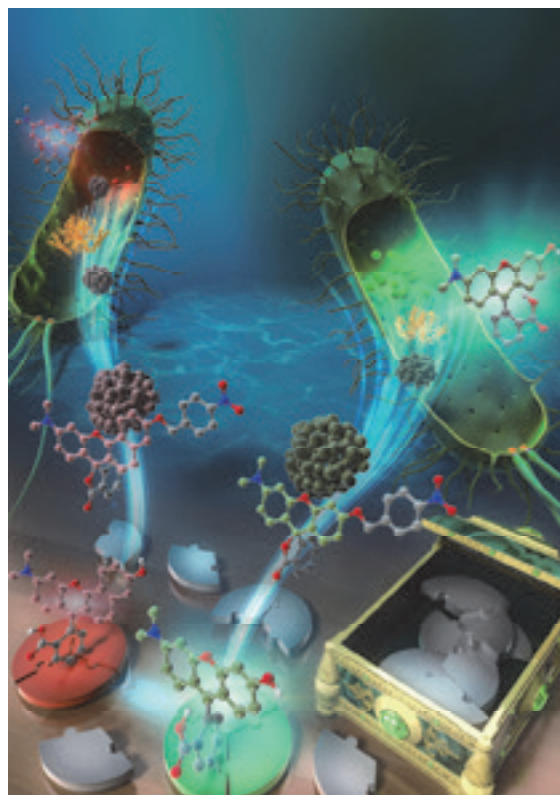


Fig. 1 An illustration of the optimization of the self-assembled fluorophore based probe for efficient detection of endogenous nitroreductase activity in *E. coli*.

**3. A Practical Approach for Polarity and Quantity Controlled Assembly of Membrane Proteins into Nanoliposomes**

Biological membranes achieve selectivity and permeability through protein transporters and channels. The design of artificial compartments with permeable membranes is essential to facilitate substrate

and product transfer in enzymatic reactions. In this study, an *E. coli* outer membrane protein OmpF fused to a modular adaptor was integrated onto a DNA origami skeleton to control the number and polarity of the OmpF trimer. DNA origami skeleton-guided nanoliposomes reconstituted with functional OmpF exhibit pH-responsiveness and size-selective permeability. This approach highlights the potential to construct artificial compartments that incorporate membrane proteins of defined number and polarity, allowing tunable substrate fluxes.

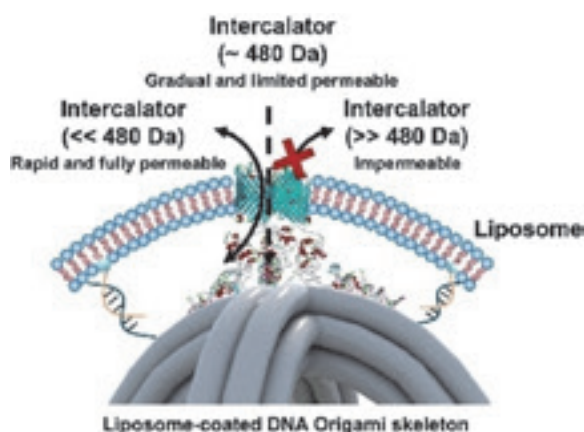


Fig. 2 An image of liposome-coated DNA skeleton with modular adaptor-fused transporter (ZF-OmpF), which incorporated in the liposome.

#### 4. The roles of high-density water layer on the DNA scaffold surface in the modulation of enzyme reactions

It is known experimentally that enzymatic reactions are often accelerated when the enzymes are assembled on the scaffold of DNA nanostructures. However, the exact mechanism by which this acceleration occurs remains unclear. Here, we study the reactions of enzymes with different catalytic mechanisms assembled on a DNA scaffold with various substrates. Analysis of the hydration properties of the substrates using our accurate statistical mechanics theory classifies the substrates into two groups that behave as hydrophilic and hydrophobic solutes, respectively. The reaction of the enzyme on the DNA scaffold is accelerated with a hydrophilic substrate but decelerated with a hydrophobic substrate. We propose a mechanism of acceleration or deceleration in which, due to the formation of a high-density layer of water near the DNA surface with high negative charge density, the concentration of a substrate with high energetic affinity for water within the layer becomes higher than that near a free enzyme, whereas that of a substrate with low energetic affinity becomes lower within the layer. This study provides chemical and physical insights

into a general case of biocatalysts, where the rates of chemical reactions occurring at the interface of biomolecules in aqueous environments can differ substantially from those in the bulk solution due to variations in the local concentration of a given ligand.

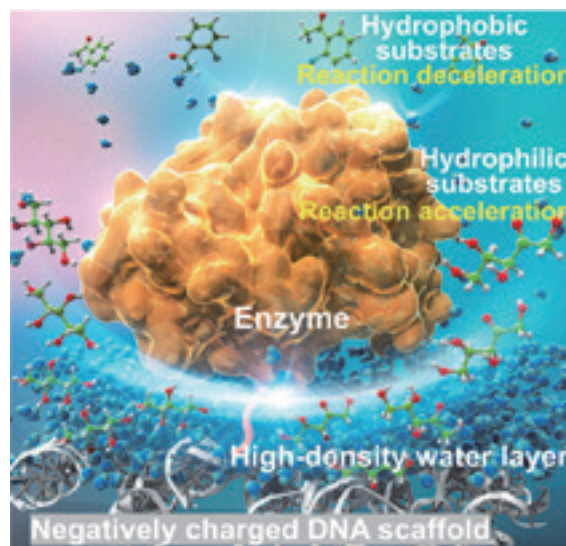


Fig. 3 An illustration of the roles of high-density water layer on the DNA scaffold surface in the modulation of enzyme reactions with hydrophilic substrates or hydrophobic substrates.

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中田栄司, University of British Columbia (カナダ), Enzyme evolution

中田栄司, 東京農業大学, 学術変革領域研究 B・復元細胞機能学

中田栄司, 東京科学大学, 学術変革領域研究 B・復元細胞機能学

中田栄司, 東京大学, 学術変革領域研究 B・復元細胞機能学

中田栄司, 東京科学大学, 学術変革領域研究 A・超越分子システム

中田栄司, 名古屋工業大学, 学術変革領域研究 A・超越分子システム

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## Structural Energy Bioscience Research Section

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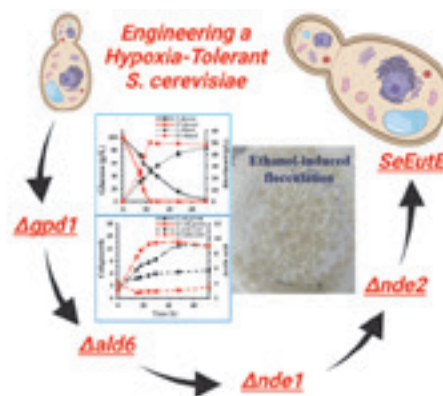
## Introduction

We investigate how biomolecules such as proteins (including enzymes) and functional nucleic acids (DNA and RNA) operate at atomic resolution using structural biology approaches with NMR. By determining both static and dynamic structures, supported by the development of novel methodologies, we elucidate the mechanisms underlying their functions. We also apply structural biology to analyze enzymes involved in degradation of woody biomass at atomic resolution. This analysis contributes to the development of methods for extracting energy and valuable compounds from woody biomass, which can serve as raw materials for a wide range of products. Through these efforts, we aim to support the paradigm shift from oil-based refineries to biorefineries. The following are the main research achievements of 2024.

### 1. Engineering a hypoxia-tolerant *Saccharomyces cerevisiae* for efficient ethanol production through co-utilization of glucose and acetic acid

Improving the robustness of microbial cell factories is essential for advancing bioethanol production. *Saccharomyces cerevisiae* often encounters fermentation stress from acetic acid, a by-product of both yeast metabolism and lignocellulosic biomass pretreatment, which inhibits growth and reduces fermentation efficiency. To overcome these challenges, we engineered a hypoxia-tolerant *S. cerevisiae* strain capable of rapid ethanol production while co-utilizing glucose and acetic acid under oxygen-limited conditions. Using CRISPR-Cas9, we sequentially deleted four key genes (*GPD1*, *ALD6*, *NDE1*, and *NDE2*) involved in glycerol synthesis and NADH oxidation, while introducing the acetylating acetaldehyde dehydrogenase gene from *Salmonella enterica* (*SeEutE*). The resulting strain, E5, achieved more than a 343% faster fermentation rate than the parent strain (C1) when grown under hypoxic conditions with 10% glucose and 0.4% acetic acid. E5 consumed approximately 25% of the acetic acid, reached 98% of the theoretical ethanol yield, and exhibited a 9% higher fermentation rate under hypoxic conditions than under hyperoxic conditions. Notably, flocculation in E5 was induced by ethanol accumulation, beginning as ethanol levels increased

and reaching approximately 3%. By the end of fermentation, 75% of the cells were flocculated, likely enhancing both stress tolerance and fermentation performance. This flocculation occurred without intentional overexpression of flocculin genes, suggesting indirect induction mechanisms linked to metabolic engineering. These results present a novel strategy for optimizing *S. cerevisiae* as a cell factory for bioethanol production, providing a promising approach to efficiently co-ferment glucose and acetic acid while maintaining high performance under hypoxic conditions, with potential applications in both first- and second-generation bioethanol processes.

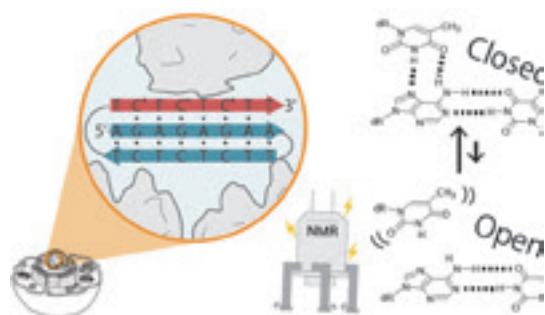


**Figure 1.** Engineering a hypoxia-tolerant *Saccharomyces cerevisiae* for rapid ethanol production via co-utilization of glucose and acetic acid and redox-enhanced flocculation.

### 2. Molecular crowding distinctly modulates base-pair dynamics in DNA triplex structures

This study sheds light on how molecular crowding (MC), a key feature of the intracellular environment, modulates the base-pair opening and closing (BPOC) dynamics of DNA triplex structures. DNA triplexes, composed of Hoogsteen base pairs (HBPs) and Watson–Crick base pairs (WCBPs), play crucial roles in gene regulation and genomic stability. However, how crowded environments affect their dynamics at the base-pair level has remained unclear. Using advanced NMR techniques, we determined, for the first time, the opening ( $k_{\text{open}}$ ) and closing ( $k_{\text{close}}$ ) rate constants of individual base pairs in a DNA triplex under MC conditions mimicked by Ficoll PM 70 and

PEG 200, representing excluded-volume effects and reduced water activity, respectively. Our findings reveal contrasting effects of these crowders. Ficoll PM 70 stabilized the triplex by prolonging the lifetime of closed base pairs, increasing Gibbs energies ( $\Delta G_{\text{open}}^{\circ}$ ) and thermal stability. In contrast, PEG 200 destabilized the triplex by shortening closed-state lifetimes and extending open state lifetimes near the strand ends, reducing  $\Delta G_{\text{open}}^{\circ}$  and thermal stability. Notably, PEG 200 induced residue-specific effects, suggesting localized base-pair openings that may create transient binding sites for regulatory proteins. These results highlight how DNA triplex dynamics respond to changing intracellular environments, potentially contributing to the regulation of gene expression and genomic stability during processes such as the cell cycle and cellular stress.



**Figure 2.** Base-pair opening and closing dynamics of DNA triplex structures under molecular crowding conditions.

### 3. Substrate preference of the anti-HIV factor APOBEC3C and the inhibitory effects of the HIV-1 Vif-human E3 ubiquitin ligase complex on its activity

APOBEC3 (A3) family proteins are cytidine deaminases that convert deoxycytidine to deoxyuridine in single-stranded (ss) DNA. APOBEC3C (A3C) restricts HIV-1 replication by introducing mutations into the viral reverse-transcribed DNA. In infected cells, the viral infectivity factor (Vif) forms the V $\beta$ BCC complex, consisting of CBF $\beta$  and the E3 ubiquitin ligase components Elongin B, Elongin C, and Cullin 5, which promotes the ubiquitination and degradation of A3 proteins. Our previous studies demonstrated that V $\beta$ BCC can also inhibit APOBEC3G (A3G) activity independently of degradation, but its effects on A3C remained unclear. In this study, we evaluated A3C deaminase activity using a uracil-DNA glycosylase assay and examined its inhibition by V $\beta$ BCC. A3C showed the highest activity at pH 5.5 and displayed a preference for target sequences (TC) located near the 5' or 3' ends of ssDNA rather than the center. Furthermore, increasing concentrations of V $\beta$ BCC progressively inhibited A3C activity. Ongoing analyses are investigating the

interactions between A3C, ssDNA, and V $\beta$ BCC to clarify the mechanism of inhibition. These findings reveal key features of A3C enzymatic activity and its regulation by HIV-1 Vif, enhancing our understanding of viral strategies to evade host defenses.

### 4. Enhancement of APOBEC3A deaminase activity by the HIV-1 Vif-human E3 ubiquitin ligase complex

APOBEC3A (A3A) is a cytidine deaminase that introduces mutations into single-stranded (ss) DNA and contributes to innate antiviral defense. We have previously demonstrated that the HIV-1 Vif-human E3 ubiquitin ligase complex (V $\beta$ BCC) inhibits the deaminase activities of APOBEC3B, 3F, 3G, and 3C. Unexpectedly, in the case of A3A, we found that V $\beta$ BCC enhances its deaminase activity. Using a uracil-DNA glycosylase assay, we confirmed that A3A deaminase activity increases in the presence of V $\beta$ BCC. Furthermore, fluorescence polarization experiments revealed that A3A, ssDNA, and V $\beta$ BCC form a complex, suggesting a direct interaction among these components. We are currently investigating the molecular mechanism underlying this unique enhancement of A3A activity by V $\beta$ BCC. These findings may provide new insights into the diverse regulatory roles of the Vif complex on different APOBEC3 family members and offer clues to understanding the complex interplay between HIV-1 and host defense mechanisms.

### 5. Applying an RNase inhibitor cocktail to extend the available time for in-cell NMR experiments on RNA

The intracellular environment is highly crowded with macromolecules, influencing RNA structure and interactions. Although in-cell NMR spectroscopy enables atomic-resolution analysis of RNA in living human cells, its application is challenging due to rapid RNA degradation. To address this issue, we introduced an RNase inhibitor cocktail into cells along with an RNA aptamer and successfully obtained in-cell NMR spectra of the RNA aptamer, which binds strongly to the HIV-1 Tat protein, both in the presence and absence of a Tat-derived peptide. The inhibitor effectively suppressed RNA degradation, extending the RNA's lifetime and allowing the acquisition of intact spectra at physiological temperatures. The resulting spectra provided structural insights into RNA before and after ligand binding without requiring chemical modifications. Notably, the inhibitor was essential for detecting the peptide-free aptamer, which appears to be highly RNase-sensitive. This cost-effective approach enhances in-cell NMR applications for RNA, offering new opportunities to investigate cellular processes and develop RNA-based therapeutics.

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## Financial Support

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## Self-Assembly Science Research Section

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### 1. Introduction

In recent years, DNA origami has emerged as an innovative technique for constructing materials ranging from nano to micrometer scale, with sub-nanometer precision.<sup>1</sup> This method is widely used in various material science, chemical, and biomedical applications.<sup>2</sup> Despite this, the broad applicability of DNA origami is limited by stability issues. DNA origami structures typically melt below 60°C, break when deposited on mica or scanned by force-based methods such as atomic force microscopy (AFM),<sup>3</sup> disintegrate in deionized water and protein-denaturing conditions, and are susceptible to digestion by cellular enzymes.<sup>4</sup>

A few strategies have been employed to enhance the stability of DNA origami under specific conditions.<sup>5</sup> However, there is no reliable method for achieving the desired stability. Recently, we have developed near-quantitative and native nick ligation methods for DNA origami, which show promise for enhancing stability.<sup>6</sup> While these methods have been tested in vitro, the in-cell stability remains unexplored. In addition, the ability to anchor enzymes on DNA origami structures and control their spatial arrangement is crucial for enhancing enzymatic activity, particularly for biomass-related reactions. However, DNA origami naturally adopts a twisted conformation due to its design with 10.67 base pairs per helical turn, which introduces unwanted conformational distortion that disrupts the positioning of enzymes and hampers the efficiency of cascaded enzymatic reactions. This twist in DNA origami is expected to significantly affect enzyme kinetics, limiting its potential for catalysis applications.

This study focused on addressing these structural challenges by employing DNA intercalators to flatten the DNA origami structures, thus improving their performance in biomolecular applications. Also, this research aimed to understand how structural modifications, such as ligation and intercalation, influence enzyme activity and small molecule binding. Additionally, it aimed to develop strategies to stabilize DNA origami for cascaded enzymatic reactions, particularly in biomass-related processes. Furthermore, this study explored the potential of DNA origami as a drug carrier by examining its interactions with small

molecules. The selective binding of specific drugs based on size and other properties is a compelling area of study. Most drug carriers rely on the covalent linkage of drugs to the carrier, requiring extensive chemical modifications and often resulting in low drug loading.<sup>7</sup> Therefore, developing a drug carrier based on non-covalent binding with high drug loading is highly desirable. Finally, it also aims to establish a size-dependent biomolecular sensing platform.

### 2. Stable DNA nanomaterials

The initial research focused on addressing the structural instability of DNA origami. Two ligation methods were explored: 1) Enhanced enzymatic ligation by dimethyl sulfoxide (DMSO): This method effectively improved the stability of 2D DNA origami, achieving over 90% ligation efficiency. 2) Chemical ligation using CNBr: This non-enzymatic approach was also successful, with ligation efficiencies of over 80% in both 2D and 3D DNA origami structures, offering a faster reaction time compared to the enzymatic method.<sup>4,6</sup> While these ligation methods addressed some stability issues, the inherent conformational twist of DNA origami remained a challenge. The next step in the research was to explore the flattening of these twisted structures using DNA intercalators, which are known to unwind duplex DNA.

### 3. Effect of intercalators on origami structure and function

DNA intercalator [Ex: ethidium bromide (EtBr)] was used to test for its ability to relax the twisted DNA origami structures. Intercalators bind to nucleic acids by inserting between the base pairs of DNA, unwinding the helix, and reducing the twist. Intercalators like ethidium bromide were found to effectively flatten the DNA origami, allowing for more uniform and planar structures. This flattening is critical for improving the relative orientation and inter-enzyme distance in cascaded enzymatic reactions. Steady-state fluorescence studies provided insights into the binding dynamics of the intercalator with DNA origami. Intercalation enhanced the fluorescence of the molecules, indicating successful binding to the DNA.

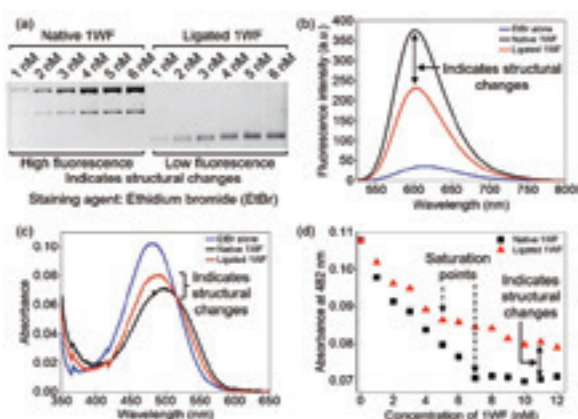


Figure 1. a) Agarose gel electrophoresis of native and ligated 1WF. Staining agent: EtBr. b) Fluorescence spectra of EtBr alone, EtBr with native and ligated 1WF. c) Absorption spectra of EtBr alone, EtBr with native and ligated 1WF. d) Plot of concentration of 1WF vs absorbance at 482 nm ( $\lambda_{max}$  of EtBr alone).

Experiments involved running native and CNBr-ligated 1WF origami at varying concentrations through an agarose gel. As shown in Figure 1a, native samples displayed relatively high fluorescence, whereas ligated samples exhibited markedly weaker fluorescence, indicating that ligation alters base stacking and intercalative binding. Steady-state fluorescence spectroscopy further supported these findings (Figure 1b). While the addition of native 1WF to EtBr significantly increased fluorescence, the same amount of ligated 1WF resulted in notably lower fluorescence enhancement, suggesting ligation-induced changes in the binding pocket. Upon intercalative binding, the absorbance of EtBr decreases with a significant bathochromic shift in the absorption maximum. This phenomenon was used to test the difference in the binding pocket of native and ligated origami (Figure 1c). Native origami showed a greater reduction in absorbance compared to ligated samples. The concentration-dependent absorbance at 482 nm also highlighted significant differences between native and ligated origami (Figure 1d). Similar results were obtained with DMSO-assisted near-quantitative ligation by ligase. While base stacking and intercalative binding were the focus, similar alterations are anticipated for inter-helical distances and groove binding. The study also examined the impact of DNA ligation on the intercalation process. Ligation-induced structural rigidity reduced the available space for intercalators to bind, suggesting that ligation and intercalation affect in a manner that could be harnessed for controlling molecular binding in drug delivery applications.

The time-resolved fluorescence and fluorescence anisotropy measurements will be carried out to get further insight on the binding affinity and structural changes in DNA origami upon intercalation. Additionally, small-angle X-ray scattering (SAXS) will be carried out to obtain direct structural information, so as to

confirm that intercalation led to significant flattening of the DNA origami. The next phase of the research will focus on immobilizing enzymes relevant to biomass-related processes on the stabilized, planar DNA origami. By fixing enzymes in close proximity on the origami scaffold, this study aimed to facilitate cascaded enzymatic reactions, which are crucial for converting biomass into valuable products like bioethanol or biobutanol. In addition to enzyme immobilization, DNA origami will be explored as a potential drug delivery system.

## 4. Conclusions

This research represents a significant advancement in the field of DNA origami, addressing key challenges related to stability, functional diversity, and application in enzyme immobilization and drug delivery. By developing novel ligation and intercalation strategies, the study has successfully created stable, planar DNA origami structures that are ideal for precise enzyme positioning in cascaded enzymatic reactions. These advances have the potential to revolutionize the use of DNA origami in biotechnology, particularly in biomass conversion processes. Additionally, the research highlights the potential of DNA origami as a versatile drug delivery system. The ability to selectively bind and release small molecules opens new avenues for targeted therapies and diagnostic tools. The insights gained from this study contribute to our understanding of how DNA's structural dynamics influence its interactions with small molecules, which is valuable for designing more effective drug delivery systems and biosensors. Future research will focus on further refining ligation techniques, exploring alternative condensing agents for 3D DNA origami, and optimizing drug loading and release dynamics. The findings from this study have broader implications for the development of DNA-based nanostructures for applications in nanomedicine, materials science, and molecular diagnostics.

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A.D. Rajeeve, V.T. Veetil, P.K. K. Namboori, R. Yamuna,\* A. Rajendran,\* Cucurbit[6]uril-stabilized copper oxide nanoparticles: Synthesis, potent antimicrobial and in vitro anticancer activity, *J. Mol. Liq.*, **2024**, 415, Part A, 126323.

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## Environmental Microbiology Research Section

T. Hara, Program-Specific Professor

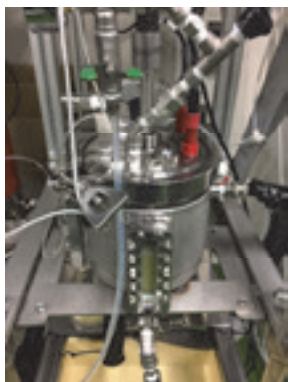
Y. Takatsuka, Program-Specific Associate Professor

## 1. Introduction

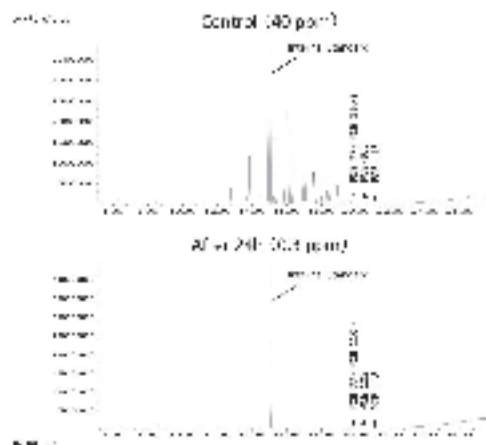
The relationship between energy resource consumption and environmental protection is crucial to developing a sustainable society. Despite our heavy reliance on fossil energy, there is concern that greenhouse gas emissions are disrupting the harmony of the global environment. Additionally, environmental pollution continues to be the shadow of civilization's progress due to the energy consumption of fossil fuels. One of the solutions is to develop a practical method that uses 'enzymes' derived from environmental microorganisms with high energy utilization efficiency in catabolism. Meanwhile, we are also working on sustainable agricultural techniques, which are the source of life energy. We are collaborating with academics, biotechs, and university start-ups globally to network research towards the social implementation of our technologies.

### 2-1. Two-compositely microbial catalysts efficiently degraded polychlorinated biphenyls.

We have developed a composite microbial catalyst that can efficiently degrade polychlorinated biphenyls (PCBs), well-known pollutants found widely in the environment. Biphenyl dioxygenase (BDO) plays a crucial role in the degradation of PCBs. It incorporates two oxygen atoms into the aromatic ring of PCB, which then induces aromatic ring cleavage. In more technological detail, our laboratory has designed a composite catalytic enzyme consisting of two BDOs



**Figure 1: The composite BDOs-microbial catalyst was tested in a dedicated experimental bioreactor with an oxygen microbubble generation device.**



**Figure 2: Gas chromatography-quadrupole mass spectrometer data show the degradation of PCBs by the composite BDOs-microbial catalyst.**

with different substrate specificities. We have also forged a bioreactor that generates oxygen microbubbles to enhance the enzymatic activities of BDOs. With these innovations, we have constructed a practical system that degrades 99.3% of 40 mg L<sup>-1</sup> of domestic major commercial PCBs (Kanechrol KC-300 and KC-400 from KANEKA Corp.) in 24 hours. This result meets the waste disposal measure defined by the Ministry of the Environment Government of Japan (Figure 2).

### 2-2. Several bacterial species associated with PCB dechlorination were genetically identified at PCB-contaminated sites.

We have been developing an artificial enzyme that can dechlorinate PCBs by two-electron reduction. To do this, we collected sediments from freshwater pit pools contaminated with PCBs in the Yodogawa riverside in the Osaka area and investigated whether bacteria associated with PCB dechlorination exist.

According to 16S rRNA gene phylogenetic analysis, it has been observed that *Dehalobacter* sp. and *Desulfitobacterium* sp. are present in that specific location. Wang and He (Environ Sci Technol, 2013) have reported that "*Deharobacter*" can dechlorinate penta- and hexa-chlorinated biphenyls while

"*Desulfotobacterium*" can dechlorinate tetra-chlorinated biphenyls that are hydroxylated at the para position.

We successfully prepared the medium for growing two specific bacterial species and developed a suitable cultivation method. In addition, we observed that these bacterial species can reduce PCBs in an artificial model of a polluted environment. Even today, after a decade of starting this investigation, we continue to observe their long-term effects to confirm the accuracy of our results.

### 3. A new protein secreted by *Rhizoctonia solani* suppresses filamentous fungi growth.

*Rhizoctonia solani* is a filamentous fungus belonging to the phylum Basidiomycota. This fungus is well-known for its ability to infect and cause severe diseases in many crops, such as rice sheath blight, which significantly impacts paddy-rice production. The exact mechanism by which this phytopathogenic fungus infects plants has yet to be fully understood. However, studies have shown that when wheat bran is added to the growth medium, this fungus secretes glycosidases that digest plants and fungi cell walls. Our team has discovered a new protein secreted from *R. solani* when cultured with wheat bran as a solid medium. This protein has a molecular weight of approximately 10 kDa and exhibits antifungal properties against filamentous fungi. This protein inhibited the growth of *Fusarium fujikuroi*, another phytopathogenic filamentous fungus belonging to the phylum Ascomycota. It was suggested that this protein also inhibited conidium formation and germination of *F. fujikuroi*.

After conducting an amino acid sequence analysis, it was found that this protein's partial amino acid sequence suggests an unknown function. Additionally, the full-length amino acid sequence of the protein was deduced after investigating the whole genome sequence of the *R. solani* strain that produces it. The BLAST search results on this sequence also suggested that the protein has an unknown function.

According to the sequence analysis of the cDNA that encodes this protein, the complete amino acid sequence of the protein was found to be 122 residues. The mature protein's amino acid sequence, which excludes the assumed signal peptide sequence, was 88 residues in length. Based on this information, the estimated molecular weight of this protein was 9648.24. Furthermore, this antifungal protein has made us aware of unique structural features in its primary amino acid sequence that have never been reported. The protein consists of 88 residues and has a double-repeat structure of 41 residues, with a high homology of 92% across the central 6 amino acid residues. The functional role of such repetitive sequences still needs to be better understood. We have named this antifungal protein "Double-Repeating Homologous Sequence Antifungal Polypeptide (DRHS-AFP)." We

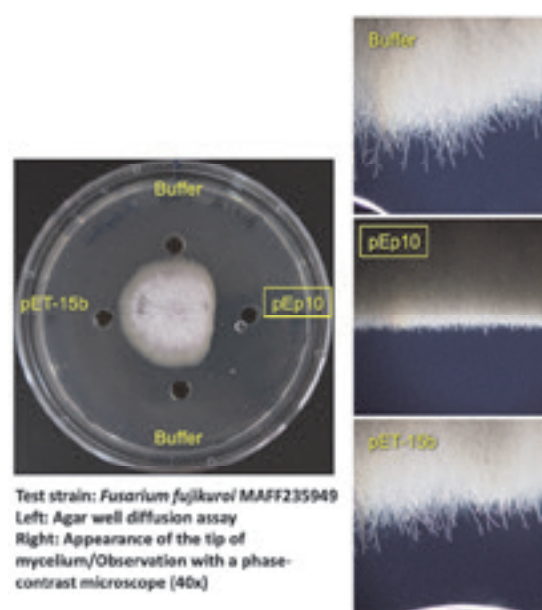
have already succeeded in producing recombinant DRHS-AFP (Figure 3) and are conducting detailed investigations into its functions, focusing on its antifungal activity.

### 4. Research into preventing coffee tree fungal diseases that may be induced by global warming is underway.

The "2050 problem" refers to the impending challenges faced by the global supply of coffee beans. The region best suited for coffee cultivation, known as the "coffee belt," lies around the equator between 25 degrees north and 25 degrees south latitude. It is projected that up to 50% of this coffee belt could be lost due to plant diseases worsened by climate change. Coffee arabica is highly sensitive to these changes. It is significantly impacted by a disease called Coffee Leaf Rust, which is caused by the fungus *Hemileia vastatrix*.

As of 2022, the Lao People's Democratic Republic is the 14th largest coffee-producing country in the world. Arabica coffee is primarily grown on the Bolaven Plateau, which is around 1,200 meters above sea level in Champasak Province, accounting for 60% of the nation's total coffee production.

To address these challenges, we have formed an international collaborative research team that includes the National University of Laos (NUOL), the Laos National Forestry Research Institute (NAFRI), Chiang Mai University, and Pibulsongkram Rajabhat University (PSRU). Our research focuses on controlling *H. vastatrix* in the Bolaven Plateau. This fiscal year, we have provided internship research guidance to NUOL faculty staff and, in collaboration with PSRU faculty, established a genetic identification system for *H. vastatrix* at NAFRI on the Bolaven Plateau.



**Figure 3. The anti-filamentous fungal activity of genetically recombinant DRHS-AFP.**

## Collaboration Works

原 富次郎, 高塚由美子, Lamont Doherty Earth Observatory, Columbia University (アメリカ), ポリ塩化ビフェニル類を分解する微生物とその由来酵素

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原 富次郎, 高塚由美子, Pibulsongkram Rajabhat University (タイ), コーヒーノキさび病真菌のゲノム解析と培養方法の開発

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## Financial Support

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原 富次郎, 基盤研究(C), 高塩素置換型ポリ塩化ビフェニル類の還元的脱塩素化を大気下で実現させる (分担金)

高塚由美子, 基盤研究(C), 高塩素置換型ポリ塩化ビフェニル類の還元的脱塩素化を大気下で実現させる

### 2. Others

原 富次郎, 東洋ガラス(株), 環境微生物の探索と機能解明の研究のため

原 富次郎, (株)竹中工務店, 環境微生物の探索と機能解明の研究のため

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## Integrated Research Center for Carbon Negative Energy Science

T. Nohira, Professor  
 K. Kondo, Associate Professor  
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 T. Yamamoto, Assistant Professor  
 Y. Norikawa, Assistant Professor  
 S. Chuaychob, Program-Specific Assistant Professor

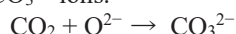
### 1. Introduction

In order to achieve carbon neutrality by 2050, we need to develop new energy systems that include active carbon dioxide fixation processes in addition to "zero-emission" technologies. Our research center is conducting research on such carbon negative technologies. To be specific, we are working on the conversion of carbon dioxide into useful materials by using renewable energy and biomass, etc.

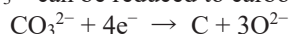
In this fiscal year, we have investigated the conversion of CO<sub>2</sub> into carbon material by molten salt electrochemical process. We have also studied enzymatic conversion of woody biomass for the production of chemical products from renewable carbon resources. In addition, we investigated CO<sub>2</sub> fixation reaction which is facilitated by an enzyme called ribulose 1,5-bisphosphate carboxylase/oxygenase (RuBisCO).

### 2. Conversion of CO<sub>2</sub> into Carbon Material by Molten Salt Electrochemical Process

As one of the Carbon dioxide Capture and Utilization (CCU) technologies, the electrochemical synthesis of carbon in molten salts is attracting much attention [1]. By using molten salts containing O<sup>2-</sup> ions, CO<sub>2</sub> can be captured as CO<sub>3</sub><sup>2-</sup> ions.



Then, CO<sub>3</sub><sup>2-</sup> can be reduced to carbon as follows.



Many allotropes of carbon, for example, diamond, amorphous carbon, graphite, carbon nanotubes, and carbon nanofibers have been electrodeposited in molten salt. We focused on graphite because its demand is expected to increase in the future as a negative-electrode material for lithium-ion batteries. We first explored combinations of molten salts that could be used at high temperatures.

Fig. 1 shows the theoretical decomposition voltages of Li<sub>2</sub>CO<sub>3</sub>, CaCO<sub>3</sub> and BaCO<sub>3</sub> to C and CO calculated using HSC Chemistry [2]. For CaCO<sub>3</sub>, CO generation is preferential at temperatures higher than 1073 K. For Li<sub>2</sub>CO<sub>3</sub> and BaCO<sub>3</sub>, C deposition is preferential even above 1173 K. However, Li<sub>2</sub>CO<sub>3</sub> begins to thermally decompose gradually when the temperature exceeds its melting point of 996 K. Therefore, in this study, we selected BaCO<sub>3</sub> as the source of CO<sub>3</sub><sup>2-</sup> and BaCl<sub>2</sub> as the main constituent salt. In addition, NaCl and KCl were added to lower the melting point. In this fiscal year, the

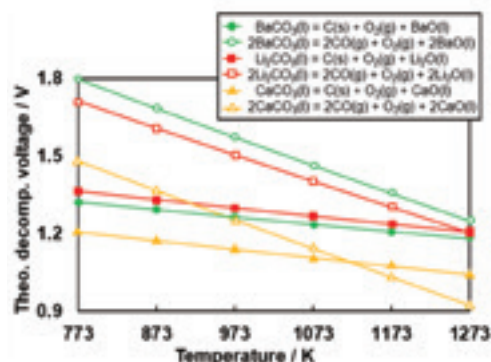


Fig. 1 Temperature dependency of theoretical decomposition voltages of Li<sub>2</sub>CO<sub>3</sub>, CaCO<sub>3</sub>, and BaCO<sub>3</sub>.

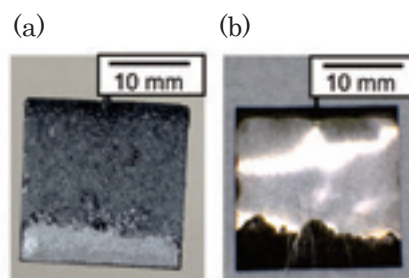


Fig. 2 Optical images of (a) before and (b) after washing the sample prepared by potentiostatic electrolysis at 0.4 V for 30 min in molten NaCl-KCl-BaCl<sub>2</sub>-BaCO<sub>3</sub> (2.0 mol%) at 873 K.

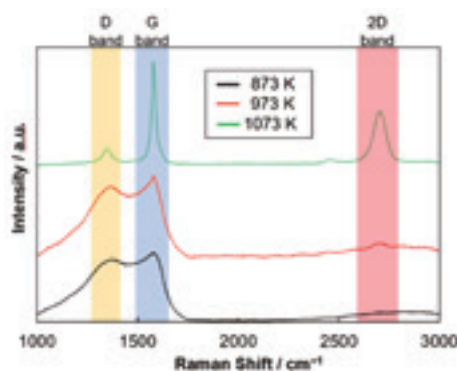


Fig. 3 Raman spectra of the samples prepared by potentiostatic electrolysis at 0.4 V for 30 min in molten NaCl-KCl-BaCl<sub>2</sub>-BaCO<sub>3</sub> (2.0 mol%) at various temperatures.

electrodeposition of carbon from molten NaCl–KCl–BaCl<sub>2</sub>–BaCO<sub>3</sub> was conducted at various temperatures (873–1073 K). Fig. 2 shows the appearances of a sample obtained at 873 K and 0.4 V vs. Na<sup>+</sup>/Na before and after washing. A black deposit was obtained, which was divided into parts that peeled off from the electrode during washing and parts that did not. Fig. 3 shows the results of micro-Raman analysis of the carbon remaining on the electrode surface for the samples obtained at each temperature. At 873 K and 973 K, spectra characteristic of amorphous carbon were observed, while peaks characteristic of graphite were observed at 1073 K. From these results, it is suggested that higher temperatures are advantageous for graphite formation.

### 3. Woody Biomass Conversion by Synergistic Action of Enzymes

Woody biomass is a promising renewable resource for energy production and chemical products. However, practical techniques for converting woody biomass into useful products are still being explored. The components of woody biomass are decomposed through the concerted action of various enzymes from wood-rotting organisms in nature. Cellulose, a major woody biomass component, is hydrolyzed by cellulases. Lytic polysaccharide monooxygenase (LPMO) accelerates hydrolysis through a synergistic action with cellulases. In this fiscal year, we assessed the synergistic effect of cellulase and LPMO on the conversion of natural biomass. The conversion of pulp derived from sugar cane trash was successfully enhanced by the simultaneous use of cellulases and LPMO, achieving the same glucose yield as when only cellulase was used, but with 48% less enzyme dosage. To further improve glucose yield by preventing non-specific adsorption of enzymes to lignin through hydrophobic interactions, we tested the addition of surfactant Tween 20. The addition of 1.4 % Tween 20 increased the conversion degree by 1.2-fold. Additionally, we tested the addition of H<sub>2</sub>O<sub>2</sub> as an oxygen source. H<sub>2</sub>O<sub>2</sub> is known to be a preferred oxygen source for LPMO over O<sub>2</sub>. However, excessive H<sub>2</sub>O<sub>2</sub> can lead to enzyme deactivation. Therefore, we tested the gradual addition of H<sub>2</sub>O<sub>2</sub> throughout the reaction time for sugarcane trash conversion. By optimizing the addition rate of H<sub>2</sub>O<sub>2</sub>, a 1.9-fold increase in conversion degree was achieved compared to the result obtained without H<sub>2</sub>O<sub>2</sub> addition.

### 4. Novel Process for Carbon Fixation Reaction

RuBisCO, the most abundant enzyme on Earth, catalyzes CO<sub>2</sub> fixation through the Calvin–Benson–Bassham pathway in plants, algae, and bacteria, but it has a low fixation rate and only binds ribulose 1,5-bisphosphate (RuBP) substrate.

To expand CO<sub>2</sub> fixation pathways, we engineered *Tk*-RuBisCO from *T. kodakaraensis* KOD1 to utilize novel

substrates derived from natural sugars, creating a Ru-BisCO library for alternative carbon fixation.

We optimized substrate-enzyme interactions by selecting sugar molecules structurally similar to RuBP (Fig. 4a), with ribulose-5-phosphate (Ru5P) (Fig. b) as the first alternative substrate. Through site-directed mutagenesis of *Tk*-RuBisCO's catalytic pocket, we generated mutant variants that were solubly expressed in *E. coli* (Fig. 4c) and purified in comparable yields (Fig. 4d).

While the wild-type enzyme exhibited higher specificity for RuBP (Fig. 5a), some mutants showed CO<sub>2</sub> fixation activity with Ru5P (Fig. 5b), which produces one molecule of 3-PGA and a by-product, compared to RuBP, which produces two molecules of 3-PGA. These results emphasize the potential for a RuBisCO library targeting mutant-substrate interactions to enhance CO<sub>2</sub> fixation.

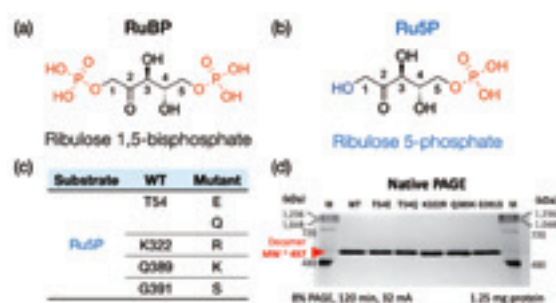


Fig. 4. RuBP (a) and Ru5P (b) substrates. *Tk*-RuBisCO variants for Ru5P (c) and their purity evaluation (d).

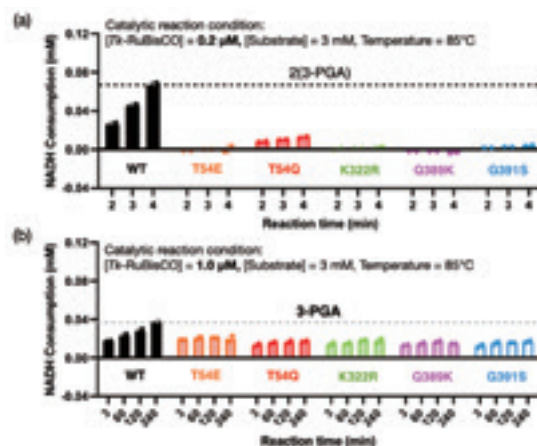


Fig. 5. The CO<sub>2</sub> fixation reactions of *Tk*-RuBisCO WT and mutants with (a) the native RuBP substrate and (b) the alternative Ru5P substrate.

### Acknowledgment

A part of this study was conducted in collaboration with Cosmo Oil Co., Ltd.

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## Financial Support

### 1. Grant-in-Aid for Scientific Research

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野平俊之, 基盤研究(B), めっき被覆と熱間等方加圧による核融合炉高熱負荷機器の修復再生 (分担金)

近藤敬子, 基盤研究(C), リグニンと多糖を分離する酵素の実バイオマスに対する活性および構造機能相関の解析 (期間延長)

近藤敬子, 基盤研究(C), 木質の多糖分解およびリグニン分解に関わる酸化還元酵素間の協奏効果の解析

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野平俊之, 新エネルギー・産業技術総合開発機構, 部素材からのレアアース分離精製技術開発事業

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S. Chuaychob, R. Alkhalaileh, M. Fujihashi, Y. Michimori, Y. Miwa, P. Lin, E. Nakata, H. Atomi, T. Morii, Novel Carbon Fixation Reaction for Enhanced CO<sub>2</sub> Utilization, 日本化学会第 105 春季年会, 関西大学, 2025.3.26-.29

R. Alkhalaileh, S. Chuaychob, M. Fujihashi, Y. Michimori, Y. Miwa, P. Lin, E. Nakata, H. Atomi, T. Morii, Design of RuBP Derivatives for the Modulation of Carboxylase Specificity, 日本化学会第 105 春季年会, 関西大学, 2025.3.26-.29

## **3-2. AWARD**



## Nishikawa Prize (Foundation for High Energy Accelerator Science)

### Quantum Radiation Energy Research Section Heishun Zen (Associate Professor)

Associate Professor Heishun Zen was awarded the Nishikawa Prize from the Foundation for High Energy Accelerator Science on March 5th, 2025 for his contribution to the development of a mid-infrared free-electron laser (FEL) for advanced research. The Nishikawa Prize is annually given to recognize individuals or a small group of people who have demonstrated outstanding originality and internationally acclaimed achievements in research on high-energy accelerators and related technologies. The prize was established in 1989 to dedicate to the memory of Tetsuji Nishikawa.

In his award lecture, which was held on March 5th, 2025, at Arcadia Ichigaya, he presented his research work on the development of mid-infrared FEL at Kyoto University. He has established a novel beam loading compensation scheme named cavity detuning and achieved the world's highest extraction efficiency of oscillator FELs, few-cycle pulse generation, and nonlinear compression. He also established a control system of the FEL and intensively supported user experiments of the FEL.



## Outstanding Oral Presentation Award in the EMSES 2025 (16th Eco-Energy and Materials Science and Engineering Symposium)

### Quantum Radiation Energy Research Section Ju Yoon Hnin Bo (D3)

The 16th Eco-Energy and Materials Science and Engineering Symposium (EMSES 2025), which was sponsored by the Kyoto University Foundation for the Promotion of Education and Research, was held from January 8th to 10th at Kyoto University. This event provides a forum for researchers, engineers and industry experts to discuss recent development, new ideas and breakthroughs in Eco-Energy and Materials Sciences and Engineering technologies.

Ju Yoon Hnin Bo (D3) made an oral presentation on the topic of "Investigation of Phonon-induced Magnetic Domain Dynamics as Neel Temperature Approaches in Nickel Oxide" in the generation and application of high-power radiation sources section and received the Outstanding Oral Presentation Award.

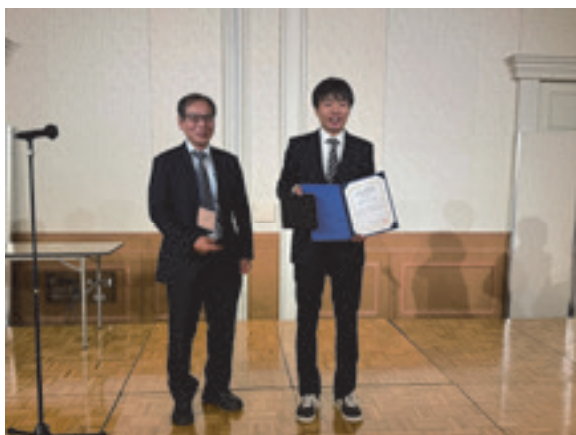


## JSPF Award for Early Career Research Contributions (Ito Sanae Special Award)

**Complex Plasma Systems Research Section  
Fumiyoshi Kin (Assistant Professor)**

Assistant Professor Fumiyoshi Kin was awarded Early Career Research Contributions (Ito Sanae Special Award) of the Japan Society of Plasma Science and Nuclear Fusion Research on November 18th, 2024. This award is annually given to a researcher who is under 35 years old and has achieved outstanding academic results in the field of plasma and nuclear fusion research. He was awarded this prize on the achievements of “Experimental study of avalanche transport and profile formation in torus plasmas”.

In his award lecture, held on November 28th, 2024, at Tower Hall Funabori in Tokyo, he presented experimental findings on electron heat fluctuations that propagate ballistically in the radial direction and contribute to the formation of the temperature profile, including stiffness, internal transport barriers and dome-shaped profiles.



## Excellent Presentation Award in the 61st National Heat Transfer Symposium of Japan

**Functional Materials Science and Engineering  
Research Section  
Akiteru Takahashi (M2)**

The 61st National Heat Transfer Symposium of Japan was held on May 29<sup>th</sup>-31<sup>st</sup>, 2024, in Kobe, Japan. This event provides young researchers and students an opportunity to present their works on heat transfer and related engineering.

Akiteru Takahashi (M2) made a poster presentation at the symposium with the topic of “Measurements of exciton thermal radiation from high-purity semiconducting carbon nanotube thin films”. His presentation was highly evaluated, and he received the Excellent Presentation Award. His research clarified exciton effects in thermal radiation from high-purity semiconducting carbon nanotube thin films, providing a basis for realizing highly efficient thermal energy conversion technologies using carbon nanotubes.



## Merit Award in the Global Research Immersion Program for Young Scientists (Grips)

Functional Materials Science and Engineering  
Research Section  
Mioko Kawakami (D2)

The Grips program is hosted by four Chinese universities near Yangtze river delta in China, including Zhejiang University. This program aims to cultivate a community and foster a group of aspiring future scientists who can encourage diverse perspectives and experiences among international students.

Mioko Kawakami (D2) participated in the Grips program at Zhejiang University from June to August 2024, and gave a poster presentation on the topic of “Floating Catalyst Chemical Vapor Deposition for Boron Nitride Nanotube Synthesis” in the Academic Poster Competition at the end of the program. Her presentation was highly evaluated, and she received the Merit Award which is given to about top 15% posters, from the Grips Committee.



## Young Scientist Poster Award/Nanoscale Horizons Award in the 67<sup>th</sup> Fullerenes-Nanotubes-Graphene General Symposium

Functional Materials Science and Engineering  
Research Section  
Akiteru Takahashi (M2)

The 67th Fullerenes-Nanotubes-Graphene General Symposium was held on September 1st–3rd, 2024 in Kochi, Japan. This conference provides researchers and students an opportunity to present their recent studies on nanomaterials science.

Akiteru Takahashi (M2) made a poster presentation at the symposium with the topic of “Observation of exciton thermal radiation from single-chirality carbon nanotube membranes”. His presentation was highly evaluated, and he received the Young Scientist Poster Award and the Nanoscale Horizons Award. His research clarified optical properties in single-chirality carbon nanotube membranes at elevated temperatures including their exciton thermal radiation properties for the first time, which provides an important basis for highly efficient thermophotovoltaic energy conversion technologies.



## Excellent Poster Certificate in the 14th A3 Symposium of Emerging materials: Nanomaterials for Energy and Electronics

Functional Materials Science and Engineering  
Research Section  
Zhirui Liu (D1)

The 14th A3 Symposium of Emerging Materials: Nanomaterials for Energy and Electronics was held on October 25th–29th, 2024 in Jincheng, China. This conference provides researchers and students an opportunity to present their recent studies on nanomaterials science.

Zhirui Liu (D1) made a poster presentation at the symposium with the topic of “Exciton binding energy in single-chirality carbon nanotube membranes probed by two-photon excitation spectroscopy”. His presentation was highly evaluated, and he received the Excellent Poster Certificate. His research clarified fundamental exciton properties in aggregated carbon nanotubes which will be crucial for their applications as energy functional materials for high-temperature thermal optics and photonics.



## Excellent Student Poster Award in the 9th Photonics Workshop

Functional Materials Science and Engineering  
Research Section  
Mioko Kawakami (D2)

The 9th Photonics Workshop was held on November 14<sup>th</sup>–15<sup>th</sup>, 2024 in Okinawa, Japan. This workshop provides researchers and students an opportunity to present their recent studies on photonics.

Mioko Kawakami (D2) made a poster presentation at the workshop with the topic of “Design and fabrication of near-infrared perfect absorbers using carbon nanotube thin films”. Her presentation was highly evaluated, and she received the Excellent Student Poster Award. Her research provides a new concept to fabricate perfect absorbers and emitters in the near infrared region with a very simple structure using carbon nanotubes, promising for high-performance wavelength selective emitter necessary for realizing high efficiency thermophotovoltaic devices.



## Symposium Poster Award in the 15th International Symposium of Advanced Energy Science

Functional Materials Science and Engineering  
Research Section  
Mioko Kawakami (D2)

The 15th International Symposium of Advanced Energy Science was held on December 10<sup>th</sup>-13<sup>th</sup>, 2024, jointly by the Institute of Advanced Energy, Kyoto University and Joint Usage/Research Center for Zero-Emission Energy Research. This event provides young researchers and students an opportunity to present their works on energy science related to zero-emission energy.

Mioko Kawakami (D2) made a poster presentation at the symposium with the topic of “Carbon nanotube-based 4-layer all-dielectric near-infrared perfect absorber”. Her presentation was highly evaluated, and she received the Symposium Poster Award. Her research provides a new concept to fabricate perfect absorbers and emitters in the near infrared region with only a 4-layer structure using carbon nanotubes, promising for high-performance wavelength selective emitter necessary for realizing high-efficiency thermophotovoltaic devices.



## Symposium Poster Award in the 15th International Symposium of Advanced Energy Science

Functional Materials Science and Engineering  
Research Section  
Zhirui Liu (D1)

The 15th International Symposium of Advanced Energy Science was held on December 10<sup>th</sup>-13<sup>th</sup>, 2024, jointly by the Institute of Advanced Energy, Kyoto University and Joint Usage/Research Center for Zero-Emission Energy Research. This event provides young researchers and students an opportunity to present their works on energy science related to zero-emission energy.

Zhirui Liu (D1) made a poster presentation at the symposium with the topic of “Aggregation effect of single-structure carbon nanotubes on the exciton binding energies”. His presentation was highly evaluated, and he received the Symposium Poster Award. His research clarified that exciton binding energy in carbon nanotubes can be maintained very high even in the aggregated conditions, which is crucial for their applications as energy functional materials for high-temperature thermal optics and photonics.

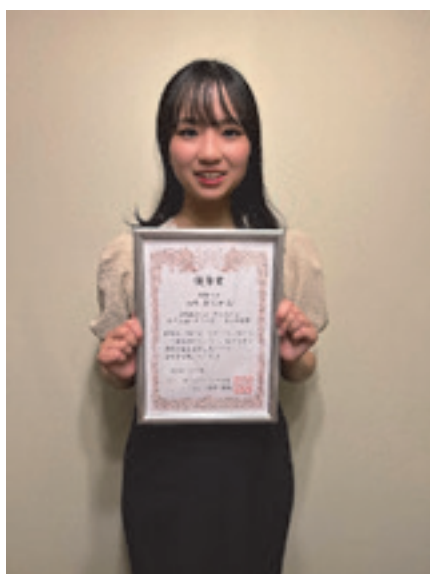


## Best Poster Award at the 98th Workshop of Materials Tailoring Society

Chemical Reaction Complex Processes  
Research Section  
Hinako Yamamoto (M1)

The 98th Workshop of Materials Tailoring Society, which organized by Materials Tailoring Society, was held on 1st–3rd August, 2024 at Karuizawa, Japan. This symposium provides an opportunity for students who will lead the next generation to present their research.

Hinako Yamamoto (M1) attended this workshop and received the best poster award for her poster presentation on the topic of “Effect of  $F^-$  ion concentration on electrodeposition of Ti in molten  $LiCl-LiF$ ”.

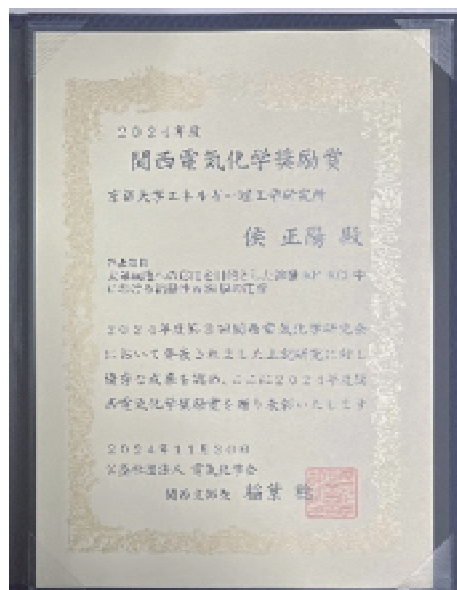


## Encouragement Award in the 3rd Kansai Electrochemical Research Association

Chemical Reaction Complex Processes  
Research Section  
Zhengyang Hou (D1)

The 3rd Kansai Electrochemical Research Association, which was sponsored by the Kansai Branch of the Electrochemical Society of Japan, was held on 30th November, 2024 at Kyoto University. This event aims to invite researchers in electrochemistry field as lecturers to provide participants with cutting-edge knowledge and offers attending students an opportunity to present their works.

Zhengyang Hou (D1) attended and made a poster presentation on the topic of “Electrodeposition of Crystalline n-Si in Molten  $KF-KCl$  for Application to Solar Cells”. He received Encouragement Award from the Kansai Branch of the Electrochemical Society of Japan.



## Symposium Poster Award in the 15th International Symposium of Advanced Energy Science

Chemical Reaction Complex Processes  
Research Section  
Wataru Moteki (D3)

The 15th International Symposium of Advanced Energy Science was held on December 10–12, 2024. This event provides researchers and students in the field related with energy an opportunity to present their works.

In the symposium, the poster session for young researchers and students was held. Mr. Wataru Moteki (D3) attended and made a presentation on the topic of “Electrodeposition of crystalline Si using liquid metal cathodes in molten KF–KCl: Comparison of Zn and Ga”. He received the Symposium Poster Award.

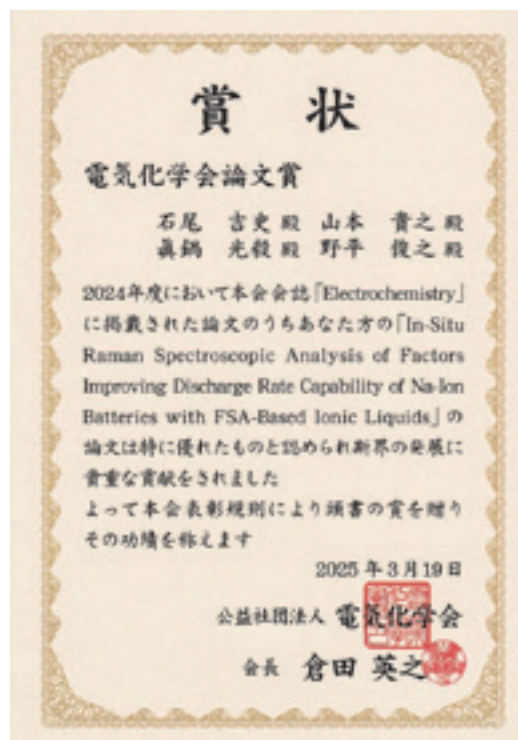


## Excellent Paper Award of The Electrochemical Society of Japan

Chemical Reaction Complex Processes  
Research Section  
Takayuki Yamamoto (Assistant Professor)  
Toshiyuki Nohira (Professor)

Assistant Professor Takayuki Yamamoto and his co-authors received the Excellent Paper Award of The Electrochemical Society of Japan on March 19th, 2025. This award is annually given to authors of excellent papers published in “Electrochemistry”, which is the academic journal of the Electrochemical Society of Japan.

The authors received the award for the paper titled “In-Situ Raman Spectroscopic Analysis of Factors Improving Discharge Rate Capability of Na-Ion Batteries with FSA-Based Ionic Liquids”.



## Best Poster Presentation Award in the 19th Symposium for Young Scientists of the Japanese Society for Biomaterials at Kansai

**Biofunctional Chemistry Research Section  
Mashal Asif (M2)**

The 19th Symposium for Young Scientists of the Japanese Society for Biomaterials at Kansai, was held on 27th, July 2024 in Kyoto. This event provides young researchers and students in the field of biochemistry, biomaterials and its surrounding area an opportunity to present their works.

Asif Masal (M2) attended and made a poster presentation on the topic of “Application of DNA nanostructure-based sensor in monitoring wide-range pH and cathepsin activity”.

She received the Best Poster Presentation Award from the Kansai Branch of the Biomaterials Society of Japan.



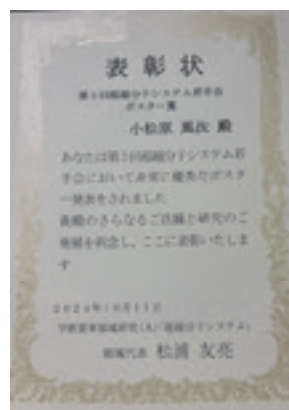
## Best Poster Presentation Award in the 3rd Symposium for young scientists of “Bottom-up creation of cell-free molecular systems: Surpassing nature”

**Biofunctional Chemistry Research Section  
Futa Komatsubara (D2)**

3<sup>rd</sup> Symposium for young scientists of “Bottom-up creation of cell-free molecular systems: Surpassing nature” was held on 11th, October 2024 in Nagoya. This event provides researchers and students in the field of biomolecule-complexes systems and its surrounding area an opportunity to present their works.

Futa Komatsubara (D2) attended and made a poster presentation on the topic of “Construction of nanoliposome supported by DNA nanostructure and the trial of enzyme assembly inside the nanoliposome”.

He received the Best Poster Presentation Award from the symposium supported by Grant-in-Aid for Transformative Research Areas (A).



## Symposium Poster Award at the 15<sup>th</sup> International Symposium of Advanced Energy Science

**Structural Energy Bioscience Research Section  
Tomoki Sakamoto (Researcher)**

The 15<sup>th</sup> International Symposium of Advanced Energy Science was held from December 10 to 12, 2024, jointly organized by the Institute of Advanced Energy, Kyoto University, and the Joint Usage/Research Center for Zero-Emission Energy Research. The symposium provided an opportunity for young researchers and students to present their work in energy science, particularly in the field of zero-emission energy research.

At the symposium, Tomoki Sakamoto presented a poster titled "Investigating the Impact of Molecular Crowding on the Base Pair Opening/Closing Dynamics of DNA Triplex Structures." His presentation was highly evaluated and awarded the Symposium Poster Award.

DNA, the carrier of genetic information, can form triplex structures at specific nucleotide sequences, which have been implicated in neurodegenerative disorders such as Friedreich's ataxia. In the presentation, he reported, for the first time, the determination of the rate constants for base pair opening ( $k_{\text{open}}$ ) and closing ( $k_{\text{close}}$ ) within a DNA triplex under molecular crowding (MC) conditions, using advanced NMR techniques. To simulate different aspects of MC, the study employed Ficoll PM 70 to mimic excluded-volume effects and PEG 200 to reduce water activity. The results revealed contrasting effects of these crowding agents on DNA triplex dynamics. These findings suggest that the base-pair opening/closing dynamics of DNA triplex structures are highly sensitive to changes in the intracellular environment.





## **4. JOINT USAGE/RESEARCH PROGRAM**





## Joint Usage/Research Center Program “Zero Emission Energy Research”

It is an urgent task to find out the best solutions against the energy and environmental problems for ensuring the sustainable society on the earth. The new energy system for this purpose must be environmentally friendly or ecological one. Here, we should consider not only the energy sources but also the efficiency in each phase of energy usage. The former should be good quality and enough quantity. The latter should be considered including the so-called “three Rs (Reduce, Reuse and Recycle)” in the energy system.

- Reduce of energy consumption, environmental pollutants such as greenhouse gas, waste-heat, hazardous waste, etc.
- Reuse of waste heat/energy, etc.
- Recycle fuel, etc.

To realize them, only the extension of the present technology is not enough. Interdisciplinary studies with innovative ideas are indispensable to realize the energy system for the next generation.

We propose a new concept of Zero Emission Energy as a typical model of Advanced Energy. IAE Zero Emission Energy Research aims at the realization of environmentally friendly energy system for sustainable society with minimum emission of environmental pollutants and with maximum utilization of energy and resources. Since FY2011, we had operated a project, “Joint Usage/Research Program on Zero Emission Energy”, which is the program authorized by the MEXT. We started the second term of the Program from FY2016 and the third phase in FY2022. Here, we aim to (1) promote interdisciplinary joint usage/research studies for Zero Emission Energy Science & Technology, (2) promote education & practical training for young researchers and (3) explore future horizons of Advanced Energy System for sustainable development. IAE provides many unique & attractive facilities for Joint Usage/Research not only in the field of advanced plasma & quantum energy but also in the field of soft energy.

Many researchers have participated in this program. In FY2024, there were 81 Joint Usage/Research collaboration subjects (including 5 workshops) on Zero Emission Energy, with more than 300 visiting participants, including graduate and undergraduate students, from 40 universities and institutions all over Japan. Researchers from 8 foreign universities also participated in the program. The results of these collaborations are summarized in the report “IAE Joint Usage/Research Program on Zero Emission Energy 2024”. If you are interested in this collection, please contact the office of Zero

Emission Energy Research.

In addition to the Joint Usage/Research collaborations, we organized “The 15th International Symposium of Advanced Energy Science - Toward the Realization of Advanced and Carbon Negative Energy” from December 10 to 12, 2024. This symposium was held on a larger scale than usual in collaboration with the affiliated Integrated Research Center for Carbon Negative Science (ICaNS). Additionally, the oral sessions were simultaneously streamed on YouTube to accommodate remote participants. This symposium consisted of an oral session, ZE poster session, student poster session, and parallel seminars. The oral session was attended by 250 participants and the parallel seminars by 60 researchers, attracting many participants. In total, 310 scientists and students, including 6 foreign and 9 domestic invited speakers, participated in the symposium. At the symposium poster session, awards were given for outstanding presentations.

We are also operating “Zero Emission Energy Network” to share the knowledge of Advanced Energy and Zero Emission Energy with researchers in the fields of energy science and technology, because world-wide activities for Zero Emission Energy Research are indispensable for the realization of sustainable society.

In FY2024, the intermediate examination by MEXT was conducted for all the Joint Usage/Research Center Programs. Our program gave an “A” evaluation. Since then, we have been continuing the effort to keep this high evaluation with the researchers of the related communities.



Poster of the 15<sup>th</sup> International Symposium

## List of Zero Emission Energy Joint Usage/Research Subjects in FY 2024

(Subject, Principal Researcher, IAE Key Person)

Structural analysis of lignocellulosic biomass by NMR spectroscopy toward decarbonized society, Hiroshi Nishimura, Masato Katahira

Degradation mechanism of high melting point materials for heat exchanger applications, Keisuke Mukai, Juro Yagi

Development of anode/electrolyte interface for advanced Na-ion battery, Hiroki Sakaguchi, Toshiyuki Nohira

Development of Interfacial Strain Relaxation Methods in Multimaterials for Fusion Reactor Components, Ryuta Kasada, Kiyohiro Yabuuchi

Emission properties and photoinduced electron-transfer reactions of photosensitizers bound to the reaction site of enzyme, Hiroshi Takashima, Eiji Nakata

NMR analysis of artificial biomolecules that control the growth of plants or microorganisms, Taiichi Sakamoto, Takashi Nagata

Electrocatalysis of graphene nanoribbons: Utilization for surface processing of silicon and energy conversion, Kazuhiro Fukami, Hiroshi Sakaguchi

Fermentative production using hydrogen sulfide and food processing by-products as energy sources, Minoru Takeda, Masato Katahira

Precise control of mode-selective phonon excitation on energy material, Kyohei Yoshida, Hideaki Ohgaki

Changes in atomic density distribution in tritium breeding material  $\text{Li}_8\text{ZrO}_6$  sintered body due to  $\text{Li}_2\text{O}$  evaporation, Kiyoto Shinmura, Juro Yagi

Structural basis of G-quadruplex recognition by the replication initiator ORC, Shou Waga, Yudai Yamaoki

Energy location of  $\text{Ce}^{3+}$  4f and defect levels in multicomponent garnet oxide crystals determined by photo-induced free carrier plasma transient absorption spectroscopy, Mamoru Kitaura, Heishun Zen

Mechanism of changes in mechanical strength properties of lithium-ion electrolyte due to ionic conduction, Kazuya Sasaki, Juro Yagi

Study on development of compound-based anode for K-ion battery and on compatibility with ionic liquid electrolyte, Yasuhiro Domi, Takayuki Yamamoto

Elucidation of interactions between TLS and long non-coding RNA that regulates liquid-liquid phase separation caused by TLS, Riki Kurokawa, Masato Katahira

Design of Electrode/Electrolyte Interface for All Solid-State Battery by Photo-Induced Chemical Solution Process, Ikuma Takahashi, Juro Yagi

Improved techniques for manipulating magnetized cells, Motonari Uesugi, Hideaki Ohgaki

Innovative Approach for Lignin Utilization: Reactivity Analysis through Selective Stable Isotope Labeling Method, Yasuyuki Matsushita, Masato Katahira

Experimental verification of hydrogen adsorption and desorption behavior for advanced neutron multipliers, Jae-Hwan Kim, Juro Yagi

Development of Fluorophores Directed toward Application in Luminescent Solar Concentrators, Masaki Shimizu, Hiroshi Sakaguchi

Determining the conditions of heat treatments for extending the lifetime of nuclear reactors (3), Yoshitaka Matsukawa, Kiyohiro Yabuuchi

Elucidation of the highly efficient energy production system utilized by intracellular organelle, Reiko Sakaguchi, Eiji Nakata

NMR Structural Analysis of Cell Growth-Related Protein in Complex with Viral Protein-Derived Peptide, Hideki Kusunoki, Takashi Nagata

Study of Hydrogen Isotope Separation Technology by Molten Salt, Hisayoshi Matsushima, Toshiyuki Nohira

Investigation on molecular structure changes of polylactide and polylactide-based materials induced by mid-infrared free electron laser, Sakhorn Rimjaem, Hideaki Ohgaki

Development of the crystalline cellulose degradation system consisting of the psychrophilic fungus-type hybrid enzymes., Masataka Horiuchi, Takashi Nagata

Characterization of E. coli derived G-quadruplexes which can regulate gene expression., Yoichiro Tanaka, Takashi Nagata

Investigation of Fast Charge Extraction in Perovskite Solar Cells with QDs-enhanced Electron Transfer utilizing MIR free-electron laser, Sukrit Sucharitakul, Hideaki Ohgaki

Fluorescent biosensor for visualizing nuclear localization signal of transcription factor Sp1 for regulating metabolic reactions, Shunsuke Tajima, Eiji Nakata

Novel pulsed terahertz source by super-radiance free electron laser oscillator, Kazuyuki Sakaue, Heishun Zen

Study of Chemical Reactions in the processing of super engineering plastics, Jun Fujioka, Heishun Zen

Development of strong superconducting bulk magnets with high shape-flexibility, Takanori Motoki, Hideaki Ohgaki

Python-Based LV Microgrid Planning Strategies: Clustered Topology and PV Hosting Capacity, Vannak Vai, Hideaki Ohgaki

Experimental research on the sophistication of advanced information infrastructure for the operation and maintenance of complex energy systems, Hidekazu Yoshikawa, Kazunori Morishita

Carbon Capture - Bioenergy System Design and Biofuel Readiness Analysis for Urban Communities, Pulungan Muhammad Almaududi, Hideaki Ohgaki

Biochar Production from Cocoa Byproducts for Rural Application, Juniza Md Saad, Hideaki Ohgaki

(Tentative Title) Achieving Carbon-Neutral Organic Coffee Cultivation through Biocontrol, Keonakhone Khounvilay, Hideaki Ohgaki

Proposing a Green Energy Ecosystem through Solar and Wind Energy in Indonesia, Anugerah Yuka Asmara, Hideaki Ohgaki

Life Cycle Assessment of Rural Electrification in Malaysia, Chia-Kwang Tan, Hideaki Ohgaki

Study of damage rate effects on mechanical properties and microstructural evolution in reactor pressure vessel model alloys., Ken-ichi Fukumoto, Kiyohiro Yabuuchi

In-situ measurement of periodic nanostructures on semiconductor surface induced by mid-infrared free electron lasers, Masaki Hashida, Heishun Zen

Description of free-electron laser interaction based on observation of coherent edge radiation, Norihiro Sei, Hideaki Ohgaki

Developing a new optical technique to determine the ratio of hydrogen bubbles to the total evolved hydrogen during water electrolysis, Kota Ando, Takashi Nakajima

Structural studies on hierarchical molecular architectures created in microfluidic devices, Munenori Numata, Eiji Nakata

Neutronics feasibility and compatibility of Li-Br/F/I based molten salt applied for fusion reactors' liquid blanket, Yasuyuki Ogino, Juro Yagi

Physical properties of large-scale structure of atomic layer materials, Susumu Okada, Kazunari Matsuda

Development of Highly Bioactive Zirconia Ceramics and Surface Control Technology, Takeshi Yabutsuka, Kiyohiro Yabuuchi

Basic study on pulse water surface discharge with needle electrodes and its improvement, Hiroto Matsuura, Shinichiro Kado

Exploring Quantum Materials for High-Efficiency and High-Performance Energy Conversion, Satoru Konabe, Yuhei Miyauchi

An artificial-nucleic-acid probe for live-cell imaging of energy metabolism, Shinichi Sato, Eiji Nakata

Development of ratiometric optical thermometry based on thermal properties of photoluminescence in single-walled carbon nanotubes, Shun Aota, Yuhei Miyauchi

Tunneling ionization with ultrafast intense infrared pulses, Ryoichi Hajima, Heishun Zen

Fabrication of multi-scale target using nano-material technology for the structured plasma generation for hydrogen-boron nuclear fusion using high intensity laser, Yasuaki Kishimoto, Hiroshi Sakaguchi

Analysis of insulin ball in mice given infrared free electron laser-irradiated insulin, Kazuhiro Nakamura, Heishun Zen

Optical properties of high-quality two-dimensional heterostructures, Wenjin Zhang, Yuhei Miyauchi

Highly efficient laser-induced photochemical processes by using machine-learning approach combined with quantum optimal control, Yuki Yoshi Ohtsuki, Takashi Nakajima

Development of novel guanine-tethered antisense oligonucleotides, Masaki Hagihara, Eiji Nakata

AFM/EM imaging of intracellular metals with nanostructures constructed via signal amplification systems, Ippei Takashima, Eiji Nakata

Effect of the metal-insulator transition temperature of vanadium dioxide film on its refractive index, Kazuma Isobe, Taishi Nishihara

Development of RNA editing technology to control metabolic enzyme genes, Masatora Fukuda, Eiji Nakata

Development of a New Method for Controlling Thermal Radiation by Quantum Metamaterials, Atsushi Sakurai, Yuhei Miyauchi

Bactericidal effect of Gram active bacteria an infrared free electron laser, Toshizo Toyama, Heishun Zen

Study of spacial property of excitons in atomically thin layered materials, Masaru Sakai, Kazunari Matsuda

Development of 3-dimensional radiative distribution measurement system using incoherent digital holography in Heliotron J., Hayato Kawazome, Shinichiro Kado

Whole genome analysis and culture method development of Thai coffee leaf rust fungus, Rampai Kodsueb, Yumiko Takatsuka

Systematic study of selective desulfation phenomena in glycosaminoglycans using infrared free electron laser, Takashi Honda, Heishun Zen

High-temperature oxidation properties of oxide dispersion strengthened alloy powder in argon atmosphere, Noriyuki Iwata, Kiyohiro Yabuuchi

Developing the technique to monitor the spatial size distribution of radioactive micro/nano fragments during laser decontamination, Atsushi Kosuge, Takashi Nakajima

Application of Model Inclusive Learning to Fusion Plasma Science - Equilibrium Reconstruction of Plasma -, Yasuaki Kuroe, Shinji Kobayashi

Study and experiment of the high-energy electron generation by the high-power laser-irradiation to the structured target, Ryutaro Matsui, Kazunari Matsuda

Development of New Semiconductor Power Control Devices Aiming for Carbon Neutrality, Kensho Okamoto, Kazunori Morishita

Analysis of direct energy conversion method using charge separation by cyclotron motion, Nobuhiro Nishino, Shinichiro Kado

Study on ultrafast measurement of relativistic electromagnetic fields, Masato Ota, Heishun Zen

Investigation of Intrabacterial Calcification due to FEL Irradiation on Human Oral Resident Bacteria, Tetsuro Kono, Hideaki Ohgaki

Radial Correlation Analysis on Edge Plasma Turbulence in a Toroidal Plasma and its Dependence on Plasma Configuration, Yoshihiko Nagashima, Shigeru Inagaki

Study on the mechanism of direct conversion of cellulosic materials into glucose under microwave irradiation conditions, Sadatsugu Takayama, Juro Yagi

Study on emission process and evaluation of light outputs for novel scintillator and dark-matter search using the one electron beam, Shunsuke Kurosawa, Hideaki Ohgaki

Distributed Workshop on "Physics and control of non-linear and non-equilibrium plasma based on the concept of broad-band energy science", Yasuaki Kishimoto, Kazunari Matsuda

The 3rd International Symposium of Biofunctional Chemistry: Towards the understanding of biological energy systems, Reiko Sakaguchi, Lin Peng

The Japanese Society for Biomaterials, Kansai Block 2024, Tetsuya Adachi, Eiji Nakata

4th Switzerland-Japan Biomolecular Chemistry Symposium (SJBCS2024), Nobutaka Fujieda, Eiji Nakata

## **5. COLLABORATION WORKS IN THE LABORATORY FOR COMPLEX ENERGY PROCESSES**



# Collaboration Works in The Laboratory for Complex Energy Processes

## 1. Introduction

The laboratory was established for research on advanced energy by the collaborative projects among the researchers in the Institute of Advanced Energy to promote joint activity of our knowledge and wisdom to find solutions to these interdisciplinary energy/environmental problems. From such a viewpoint, the research targets of the laboratory are focused on two specific fields, (i) "advanced studies of science and technology on plasma energy and quantum energy" and (ii) "innovative studies of nano-bio functional materials for power generation". For this purpose, two sections (A2 and A3 mentioned below) are founded. In addition, A1 section promotes international or domestic collaborative research and assists activities such as academic meetings and seminars. In the fiscal year of 2024, strong advancement of the collaboration was achieved.

Close connection between related research fields in the institute have yielded unique and interesting outcomes from the collaboration. The laboratory takes charge of organizing and promoting the cooperative research project as a center of research activity in the Institute. The research teams were formed by mostly young generation staffs and students in the institute lead by associate professor or assistant professor, and participated in specific projects to carry out their subjects. The cooperative research activities will be published in a publication edited in the laboratory at the end of the fiscal year. Management of the technical staffs for large scale equipment are also under the responsibility of the laboratory.

### A1 Division of International and Industrial Partnership

This division promotes international collaborative research on advanced energy to lead the field of energy science and technology as a worldwide pioneer. For this purpose, the symposium and the workshop organized by institution member are supported. Following disappearance of the threat of COVID-19, meetings or exchanges were energetically held.

### A2 Division of Plasma and Quantum Energy Research

This section promotes studies on advanced plasmas and quantum energy for realizing future energy systems, integrating plasma energy science and advanced energy material research. In particular, based on the results obtained in our related groups, we aim at extending the research fields and contributing to human society by utilizing the existing key devices such as Heliotron J, MUSTER and inertial electrostatic confinement (IEC) device, which have been developed in the institute.

### A3 Division of Soft Energy Science Research

This division promotes studies on emergent materials and systems for realizing next generation soft energy system. In particular, functional nano- and bio-materials to efficiently utilize solar energy and bio-energy are studied by integrating laser science, nanotechnology, and bio-technology. We aim at extending our research fields by utilizing the existing devices such as System for Creation and Functional Analysis of Catalytic Materials, SEMs, SPM, NMRs including 800 MHz LC-MS/NMR, Solar Simulator, KU-FEL and various laser systems.

## 2. The cooperative research program

In the fiscal year of 2024, two categories were set up: (1) "Cooperative Research" for cross sectional research and (2) "Sprouting Research" for challenging research proposal by Assistant and Associate Professors. The submitted proposals were evaluated by the selection committee organized by a center director, a program chair and three division chairs. Three "Cooperative Research" proposals and four "Sprouting Research" proposals were approved. The number of research subjects is listed in Table 1 according to the division. A brief summary of the cooperative research subjects carried out in FY2024 is shown in the next page.

**Table 1 Number of the accepted research subjects according to the division**

Category			Total
A1	A2	A3	
5	3	4	12

The individual research subjects are as follows.

### **Supporting Activities on International and Industrial Collaborative Research**

#### **A1**

#### **“Organization of the seventh research seminar on RNA research, and presentation and exchange of information in the seminar”**

- M. Katahira, T. Nagata (Institute of Advanced Energy, Kyoto University)
- R. Kurokawa, R. Yoneda (Saitama Medical University)
- T. Yamashita (Hoshi University)
- N. Shiina (National Institute for Basic Biology)
- S. Ishigaki (Nagoya University)
- T. Oyoshi (Shizuoka University)
- T. Mannen (Ritsumeikan University)
- A. Takeuchi (Ehime University)
- T. Ohyama (RIKEN)
- K. Furugori (Yokohama City University)
- T. Nobeyama (Kyoto University)

#### **“Support to the Eco-Energy and Materials Science and Engineering Symposium 2025 (EMSSES2025)”**

- H. Ohgaki, M. Katahira, K. Nagasaki, E. Nakata (Institute of Advanced Energy, Kyoto University)
- S. Pivsa-Art, S. Pavasupree, K. Bhumkittipich, B. Plangklang, S. Niamlang (Rajamangala University of Technology Thanyaburi)
- T. Sagawa, T. Yabutsuka (Graduate School of Energy Science, Kyoto University)
- H. Kamitakahara (Graduate School of Agriculture, Kyoto University)
- Y. Aso, K. Yamada (Kyoto Institute of Technology)
- M. Tanaka, H. Tarao (National Institute of Technology Kagawa College)

#### **“The 31st FEL and High-Power Radiation Workshop”**

- H. Ohgaki, H. Zen (Institute of Advanced Energy, Kyoto University)
- T. Hara, T. Kii (RIKEN)
- T. Kanai (Institute for Molecular Science)
- R. Hajima, M. Kando, K. Kawase (KPSI, QST)
- M. Hashida (Tokai University)
- M. Kato (Hiroshima University)
- N. Sei (AIST)
- J. Fujioka (Tokyo University of Science)
- Y. Hayakawa (Nihon University)
- Y. Takashima (Nagoya University)
- S. Miyamoto (Osaka University)
- K. Yoshida (Kumamoto Industrial Research Institute)

#### **“4th Switzerland-Japan Biomolecular Chemistry Symposium (SJBCS2024)”**

- E. Nakata, A. Rajendran, P. Lin, C. Surachada (Institute of Advanced Energy, Kyoto University)
- S. Futaki, M. Imanishi, Y. Kawaguchi (Institute for Chemical Research, Kyoto University)
- I. Hamachi (Graduate School of Engineering, Kyoto University)
- A. Steinauer, B. Fierz (EPFL)
- Liang, A.D., P. Rivera-Fuentes (University of Zurich)
- M. Frei, D. Hilvert, J. Bode (ETH Zurich)
- C. Cao, S. Hoogendoorn, N. Winssinger, S. Matile (University of Geneva)
- T. Ward (University of Basel)
- H. Suga, K. Sugihara (The University of Tokyo)
- K. Matsuura (Tottori University)
- K. Kinbara, M. Kamiya (Institute of Science Tokyo)
- H. Abe (Nagoya University)
- S. Tsukiji (Nagoya Institute of Technology)
- Y. Hori (Kyushu University)
- R. Watanabe (RIKEN)
- P. Laurino (Okinawa Institute of Science and Technology)
- N. Fujieda (Osaka Metropolitan University)
- H. Ishida (Kansai University)

#### **“5th Symbio Community Forum Lecture 2024 - Japan’s Energy Policy: Seventh Strategic Energy Plan”**

- K. Morishita (Institute of Advanced Energy, Kyoto University)
- H. Yoshikawa, S. Yoshikawa, T. Morii, M. Shioji, K. Mishima (Kyoto University)
- M. Abe (Graduate School of Energy Science, Kyoto University)
- A. Gofuku (Okayama Prefectural University)
- K. Ishihara (Office of Institutional Advancement and Communications, Kyoto University)
- J. Nitta (Arcadia Systems Inc.)
- T. Matsuoka (Utsunomiya University)
- H. Iwakiri (Department of Education, University of the Ryukyus)
- T. Terai (The Institute of Applied Energy)
- K. Akimoto (RITE)

## Cooperative Research

### A2

#### **“Nitride/Oxide double coating using dielectric barrier discharge”**

- J. Yagi, S. Kobayashi, S. Inagaki (Institute of Advanced Energy, Kyoto University)
- T. Ishii, Y. Ito (Graduate School of Energy Science, Kyoto University)

### A3

#### **“Optimization of Synthesis of functional molecules from carbon dioxide”**

- E. Nakata, J. Yagi, S. Onishi (Institute of Advanced Energy, Kyoto University)

#### **“Flattening DNA Origami by Intercalators and its Application for Enzymatic Reactions: Steady-State and Time-Resolved Fluorescence Analyses”**

- A. Rajendran (Institute of Advanced Energy, Kyoto University)

## Sprouting Research

### A2

#### **“Simultaneous measurement of electron density and electron temperature fluctuations in Heliotron J”**

- F. Kin (Institute of Advanced Energy, Kyoto University)

#### **“Reproducing Aurora Green Emission in Laboratory Plasma”**

- S. Kado (Institute of Advanced Energy, Kyoto University)

### A3

#### **“Woody biomass degradation in membrane bioreactor using lytic polysaccharide monooxygenase”**

- K. Kondo (Institute of Advanced Energy, Kyoto University)

#### **“Electrochemical synthesis of MXene compounds using molten salts”**

- T. Yamamoto, K. Kawaguchi (Institute of Advanced Energy, Kyoto University)

# The Laboratory Seminars

## Laboratory Seminars

The Laboratory promotes topical academic seminars in order to strengthen the research activities in each research section and to enhance the mutual cooperation among a lot of academic fields. In the fiscal year of 2024 the aims and progress reports of eight cooperative researches were presented and discussed, as summarized below. The Laboratory also planned a symposium on April 4, 2025 for presentation of the cooperative research results in FY2024.

Institute of Advanced Energy, Kyoto University

**(1) July 12, 2024**

**Daniel A. Scherson**

**“In situ Spectroscopy: Energy Storage and Electrosynthesis Applications”**

Department of Chemistry, Case Western Reserve University

**(5) October 4, 2024**

**E. Nakata**

**“Optimization of Synthesis of functional molecules from carbon dioxide”**

Institute of Advanced Energy, Kyoto University

**(2) August 30, 2024**

**F. Kin**

**“Turbulent transport in magnetically confinement fusion plasmas and studies in Heliotron J”**

Institute of Advanced Energy, Kyoto University

**(6) October 4, 2024**

**K. Kondo**

**“Application of oxidative enzymes for woody biomass decomposition”**

Institute of Advanced Energy, Kyoto University

**(3) August 30, 2024**

**S. Kado**

**“Auroral emission spectroscopy and its laboratory reproduction trial”**

Institute of Advanced Energy, Kyoto University

**(7) December 20, 2024**

**A. Rajendran**

**“Stable DNA nanomaterials for practical applications”**

Institute of Advanced Energy, Kyoto University

**(4) September 13, 2024**

**J. Yagi**

**“Activities in Advanced Atomic Energy section for the development of the advanced breeding blanket system in fusion reactors”**

**(8) December 20, 2024**

**T. Yamamoto**

**“Toward the development of rare-metal-free batteries”**

Institute of Advanced Energy, Kyoto University

## **6. INTEGRATED RESEARCH CENTER FOR CARBON NEGATIVE SCIENCE**



## 2024 Activities

### Introduction

The Integrated Research Center for Carbon Negative Science (ICaNS) was established in August 2022 to promote carbon negative science research toward the realization of a carbon neutral society in 2050. Currently, the balance between carbon dioxide emissions and absorption has been disrupted, resulting in an excess of carbon dioxide emissions and a serious impact on the earth in the form of climate change. Restoring the balance is difficult with "zero emission" technologies alone and requires the development and implementation of more active carbon dioxide fixation processes, so-called "carbon negative" technologies. The Center will work to develop such new carbon dioxide fixation technologies in collaboration with the Graduate School of Engineering and Graduate School of Energy Science at Kyoto University. It will also work to develop human resources for "carbon negative science," which has not been done before.

In the fiscal year (FY2022), three major research projects were launched: 1) Solar Energy Utilization for CO<sub>2</sub> Capture and Conversion, 2) Conversion of CO<sub>2</sub> into Useful Substances, 3) Biological Utilization of CO<sub>2</sub>. This fiscal year (FY2024), continuing from the previous year, we promoted these priority research projects and established an educational system for "carbon negative science". In addition, an international seminar on carbon negative energy science was held, and the center's laboratories were upgraded and experimental equipment was installed.

### 1. Solar Energy Utilization for CO<sub>2</sub> Capture and Conversion

The objective of this group is to establish novel science and technology for efficient solar energy utilization required for capturing CO<sub>2</sub> and/or converting CO<sub>2</sub> into valuable materials. In FY2024, the following studies were primarily conducted in this group: Studies on developments of carbon nanotube-based high-performance selective solar absorber for solar energy harvesting and wavelength-selective thermal emitter for high efficiency thermophotovoltaic energy conversion; novel energy conversion and generation technologies based on quantum optical science; on-surface electro-chemical synthesis of a strong electron-donating graphene nanoribbon catalyst; electrochemical performance of laser-textured electrodes for efficient hydrogen evolution; light-induced nitrogen fixation; and conversion of pollutants and biomass to value-added chemicals.

### 2. Conversion of CO<sub>2</sub> into Useful Substances

This project group aims to convert CO<sub>2</sub> into useful substances. We are particularly interested in electrochemical methods of conversion. By using high-temperature molten salts as electrolytes, for example, CO<sub>2</sub> could be converted into a wide variety of valuable carbon materials, such as diamonds, carbon nanotubes, and graphite. If aqueous solutions, organic solvents, or ionic liquids are used as electrolytes at relatively low temperatures, CO<sub>2</sub> can be converted into methane, ethylene, and other materials. In FY2024, various types of carbon were electro-deposited using CO<sub>2</sub> as the raw material in molten chloride salts at temperatures of 500 to 900 °C. It was found that amorphous carbon was likely to be obtained at low temperatures, and that graphite could be obtained under certain conditions at high temperatures. In addition, although only in small quantities, diamond was also obtained as another carbon allotrope.

### 3. Biological Utilization of CO<sub>2</sub>

Research in this project focuses on bio-related methods, materials and enzymes with the goal to contribute to Carbon Negative Science. In FY2024, studies that were carried out include the following: the development of tools to better understand the biological cell and its energy conservation, and technology to enhance and/or prolong the activity of enzymes, particularly those related to CO<sub>2</sub>-fixation. Engineering of the CO<sub>2</sub>-fixation enzyme and enhancement of substrate specificity have been carried out to enable the production of valuable molecules from CO<sub>2</sub>, thereby improving CO<sub>2</sub> fixation through a novel reaction. Development of membranes, reactors and processes to enhance biomass utilization and establish efficient biorefineries have been performed. New microbial enzymes or metabolic pathways that can utilize CO<sub>2</sub> or prevent its release have been identified. Our collaborative research has been continued in pursuit of our shared goals and to contribute to the development of a bio-based society.

### 4. Education Activity

The content of "Carbon Negative Energy" was incorporated into the existing undergraduate course "Advanced Energy Science". As for graduate-level lectures, "Carbon Negative Energy" was incorporated into "Socio-Environmental Energy Science I and II" in the Graduate School of Energy Sciences, as in the previous fiscal year. The concept of "Carbon Negative Energy" was also promoted to visitors to the institute.

## 5. Other Activities



Fig. 1-1 A dehumidifier.



Fig. 1-2 An X-ray photoelectron spectroscopy.



Fig. 1-3 An experimental bench.

ray photoelectron spectroscopy (XPS), and an experimental bench were installed, as shown in Figs 1-1, 1-2, and 1-3.

We further upgraded Laboratory 1-5, Waiting Room 1-2, Program-Specific Associate Professor's Room and Program Specific Assistant Professor's Room in the main building of the Uji Campus. As for experimental equipment, a dehumidifier, an X-

## **ICaNS Events**

June 25: The 1st Steering Committee meeting was held.

December 13: Symposium Session of Exploring Carbon Negative Energy Science was held in 15th International Symposium of Advanced Energy.

December 12: Symposium on Exploring Carbon Negative Energy Science 2024 was also held as a parallel seminar of the 15th International Symposium of Advanced Energy.

December 21: The 2nd Steering Committee meeting was held.

February 22: The 3rd Steering Committee meeting was held.



## **7. PROJECTS WITH OTHER UNIVERSITIES AND ORGANIZATIONS**



## NIFS Bilateral Collaboration Research Program on Heliotron J

The Heliotron J group at IAE, Kyoto University has joined the Bilateral Collaboration Research Program managed by National Institute for Fusion Science (NIFS) since FY2004. This unique collaboration program promotes bilateral research between NIFS and research institutes or research centers of universities that have facilities for nuclear fusion research. Under this collaboration scheme, the facilities operated in the different universities are open to all fusion researchers just as joint-use facilities of NIFS.

The main objective of the research in our Heliotron J group under this joint research program is to investigate experimentally/theoretically the transport and stability of fusion plasma in the advanced helical magnetic field and to improve the plasma performance through advanced helical-field control in Heliotron J. Picked up are the following seven key-topics; (1) magnetic configuration control for energy confinement, (2) production and confinement of high-density NBI plasmas and high-beta plasmas with novel fuelling methods, (3) electron thermal turbulent transport with self-criticality, (4) control of MHD and its physical mechanism, (5) edge plasmas in advanced helical systems, (6) production of energetic (MeV) electrons by non-resonant microwaves, and (7) development of new plasma diagnostics and analysis methods.

Two results from this collaboration research in FY2024 are shortly reported below. The annual report for all the collaboration subjects in this program will be published by NIFS.

**Formation of internal transport barrier in NBI plasmas:** Controlling heat transport is important to achieve high performance in magnetic confinement fusion plasmas. In stellarator/heliotron plasmas, a high electron temperature plasma with a steep electron temperature gradient (electron temperature transport barrier; e-ITB) formation in the plasma center has been observed at electron cyclotron heating (ECH). This is called CERC (Core Electron-Root Confinement) because a strong positive radial electric field (electron root) is formed near the core. Under such conditions, it is known that a characteristic strong positive radial electric field ( $E_r$ ) is formed.

In this study, we found that the heat transport was improved in the inner region by applying high intensity gas puffing (HIGP) to neutral beam injection (NBI) heating. We have measured the temperature profile with a Thomson scattering system and a charge exchange spectroscopy and evaluated the radial electric field by simulation. The plasma is heated with balanced NBI at an injection power of 280-290 kW. The gas puffing is abruptly switched off by applying a high-intensity gas puffing (HIGP) for a short period of

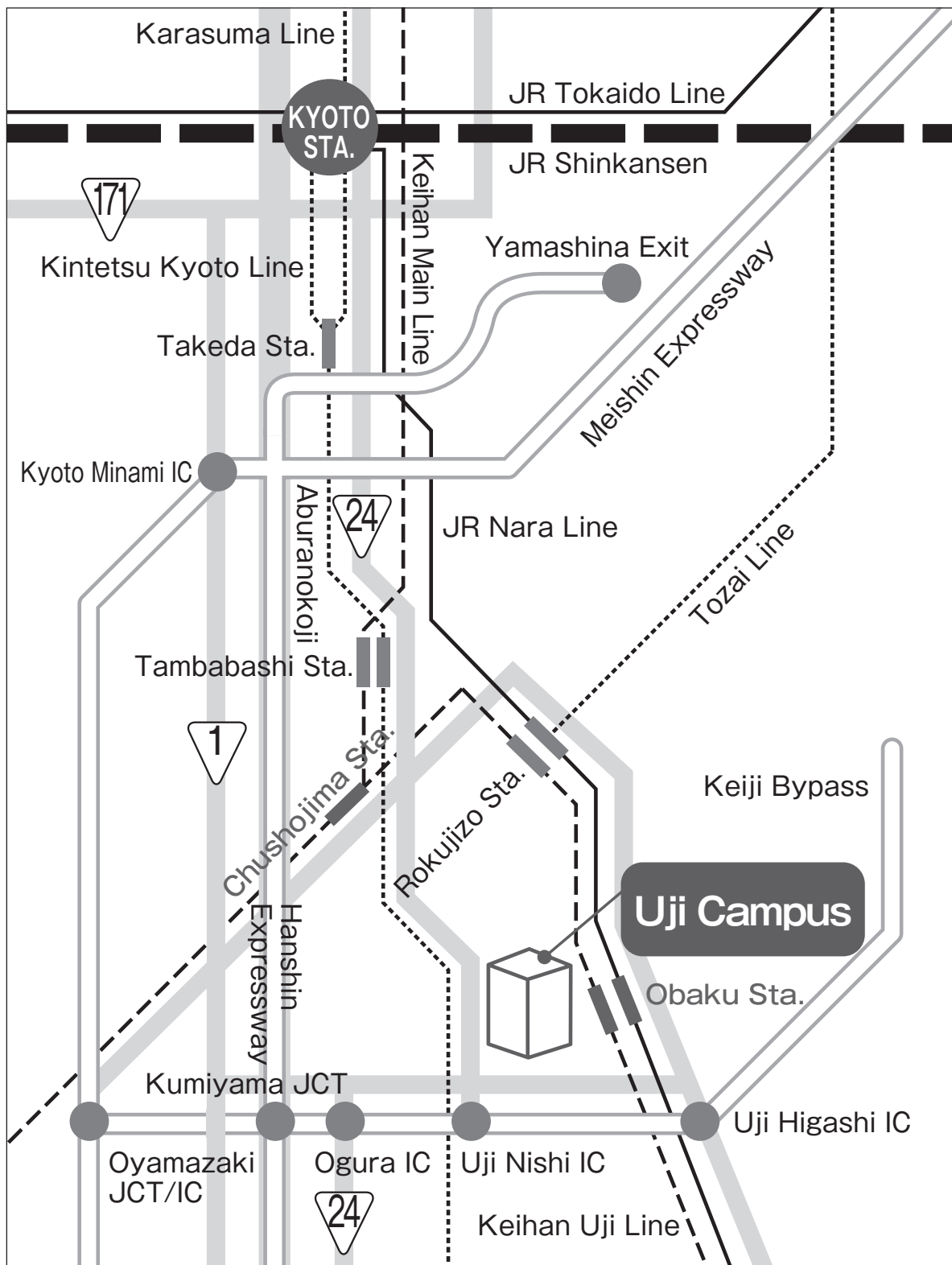
10 ms. After the HIGP injection, the Te profile shrinks and becomes more peaked. It then recovers to form an e-ITB structure with core Te of 370 eV. The difference is remarkable compared to the usual gas puff (GP) experiment. On the other hand, the ion temperature profile also sharpens with a similar time variation, but to a small degree.

The radial electric field calculated using the DKES PENTA code shows (i) a negative sign and (ii) no difference in the radial electric field compared to the normal gas puffing experiment, even in the peaked profile, suggesting that confinement is improved by a physical process different from that of CERC. We are currently investigating the relationship with lower-order rational surfaces.

**L-H transition experiment:** The H-mode transition, which improves plasma confinement performance in fusion plasmas, has been studied extensively. During the L-H transition, an increase in ExB shear flow and a decrease in turbulence amplitude have been observed in stellarator/heliotrons as well as in tokamaks. On the other hand, it has been frequently observed that the pedestal density gradient increases during the H-mode transition, but the temperature gradient remains unchanged. The lack of a temperature transport barrier suggests weak correlation between heat and particle transport in stellarator/heliotrons. The situation is similar to I-modes in tokamaks.

In this study, electron density and electron temperature fluctuations during the L-H transition of heliotron J have been measured simultaneously with a Doppler reflectometer and an electron cyclotron emission (ECE). The L-H transition occurs in high-density plasmas ( $n_e > 1.5 \times 10^{19} \text{ m}^{-3}$ ) and is characterized by a change in the radial electric field, a decrease in the turbulence amplitude, and an increase in the pedestal density gradient only. The same characteristics as in previous studies have been observed: a change in radial electric field, a decrease in turbulence amplitude, and an increase only in the pedestal density gradient. Electron temperature fluctuation is observed at the frequency of about 15 kHz before the L-H transition. The density fluctuation amplitude measured with the reflectometer also oscillates at the same frequency, suggesting that this mode is nonlinearly coupled with the background turbulence. This mode also has a radial correlation length over the region,  $0.2 < \rho < 1.0$ . After the L-H transition, the amplitude of the density fluctuation decreases while the electron temperature fluctuation remains unchanged.

## 8. HOW TO GET TO THE IAE





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