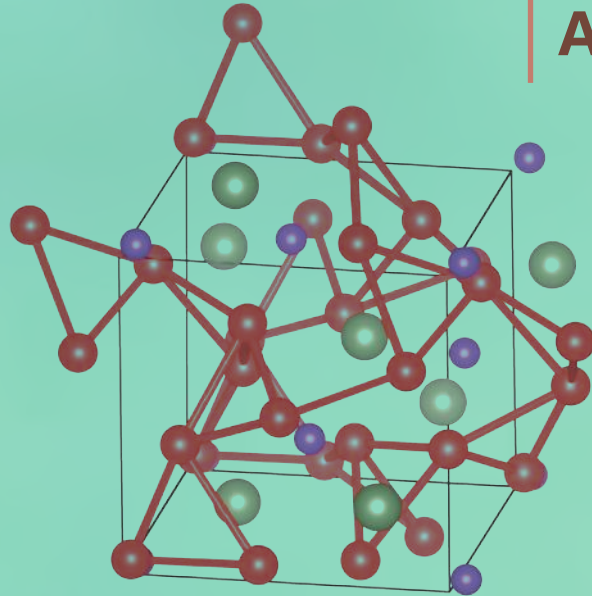


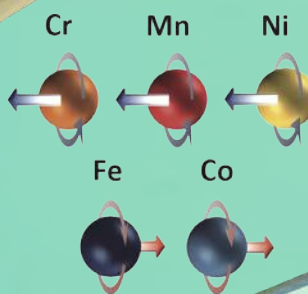
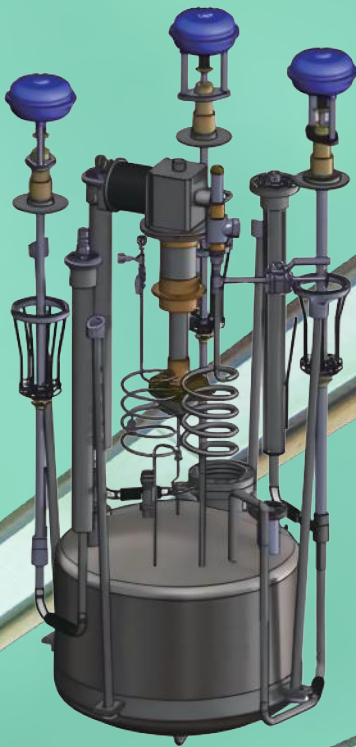
NSRRC

Activity Report

2024



National
Synchrotron Radiation
Research Center



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About the National Synchrotron Radiation Research Center

Located in Hsinchu Science Park, the National Synchrotron Radiation Research Center (NSRRC) is a non-profit research organization, funded by Taiwan's National Science and Technology Council (NSTC). NSRRC currently operates two synchrotron light sources: the Taiwan Light Source (TLS) and the Taiwan Photon Source (TPS). As of December 2024, NSRRC provides users worldwide with 42 beamlines including two Taiwanese contract beamlines at SPring-8. The TLS, of beam energy 1.5 GeV and circumference 120 m, is the first third-generation synchrotron light source facility established in Asia and the third globally. It comprises 22 beamlines and over 50 experimental stations, and began user operation in 1993. The TPS, of circumference 518.4 m, is equipped with a low-emittance synchrotron storage ring and a booster synchrotron of energy 3 GeV. It has a capacity for about 40 beamlines, with 18 currently open to users. As one of the brightest mid-energy synchrotron light sources in the world, the TPS not only accelerates scientific advancements but also broadens the application of synchrotron radiation across diverse research fields. In 2024, NSRRC's experimental facilities served more than 2,900 users. For more information about the NSRRC, please visit www.nsrcc.org.tw.

NSRRC

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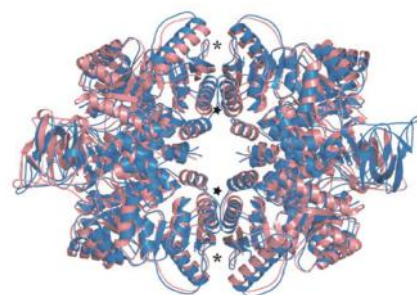
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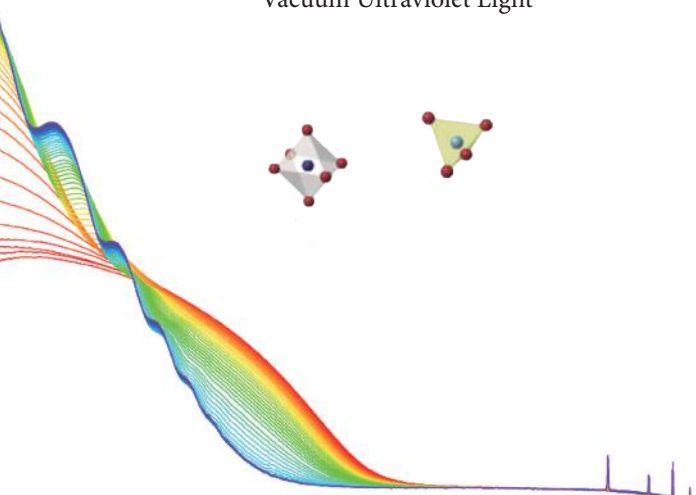
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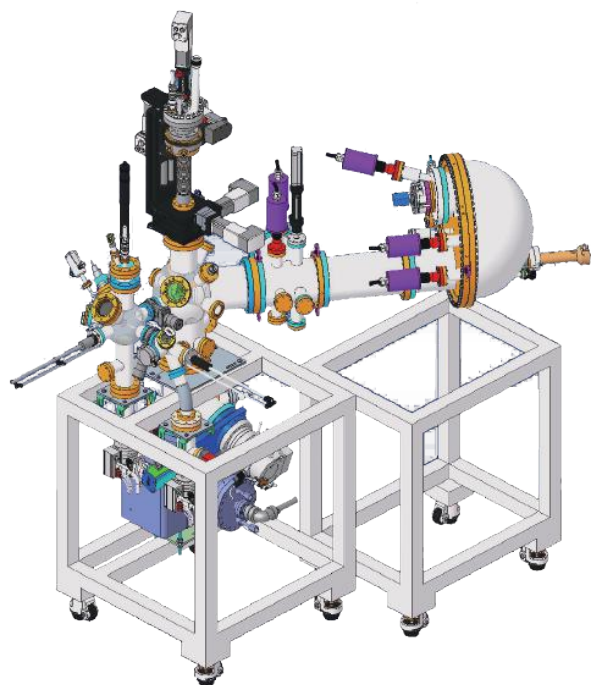
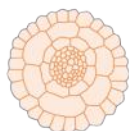
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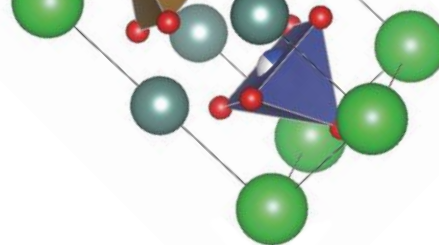
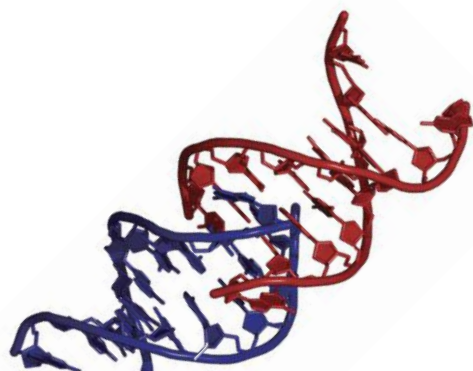


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Message from the Director

Reflecting on 2024, I am filled with a sense of pride, gratitude, and motivation for all we've accomplished. As we continue to expand and enhance our research facilities, significant progress has been made, marking a pivotal moment in both our operations and the scientific achievements.

In terms of achievements, 2024 saw our user community reach a new milestone of 2,929 users, with 1,830 experimental runs and 13,861 user visits. These efforts have contributed to the publication of 619 SCI research papers, of which 58% have an impact factor higher than that of *Phys. Rev. Lett.*, underscoring the critical role that NSRRC plays in advancing scientific discovery.

In 2024, a series of alarming earthquakes caused beam disruptions at the Taiwan Light Source (TLS) and the Taiwan Photon Source (TPS). After the magnitude 7.1 Hualien earthquake on April 3, our accelerators were restored to normal and stable operation within just a few hours. We are grateful for the support and concerns expressed by other synchrotron radiation facilities during this period. Although these events resulted in a slight decrease in annual availability—99.4% for the TLS and 97.7% for the TPS—the overall performance remains highly satisfactory.

Looking ahead, NSRRC is committed to expanding the capabilities of TPS. Currently, 18 beamlines are operational, with plans to increase to 26 by 2029. This expansion will include a dedicated beamline for industrial applications, fostering deeper collaboration with industry partners. We have also launched a call for letters of interest for new beamlines, and an Infrared Microspectroscopy Beamline has been selected. The addition of this new infrared beamline will further broaden the spectral range of the TPS and strengthen the research in the fields of biomedicine and semiconductors.

As these advancements unfold, we also take time to reflect on the legacy of the TLS. Inaugurated in 1993, TLS is not only an exemplification of Taiwan's ability to develop cutting-edge technologies in ultra-high vacuum technologies, precision magnets, and instrumentation, but also a cornerstone of scientific research in Asia. With over 225,000 user visits and more than 7,000 SCI publications, TLS's impact is undeniable. However, age-related issues are beginning to emerge, with key components experiencing wear and the discontinuation of their production. NSRRC foresaw this situation more than a decade ago, leading to the construction of the TPS, designed to replace and surpass the TLS, particularly in X-ray capabilities. The time for this transition is approaching.

Moreover, while NSRRC has earned international recognition for the high-quality user support, maintaining this expertise with limited manpower has become increasingly challenging. Following a thorough evaluation of economic considerations and user research needs, the Board of Trustees has resolved to operate TLS until the end of 2028, with partial operation possible in 2029 if needed. As TPS beamlines are phased in, TLS beamlines will gradually be scaled down, with all users ultimately transitioning to the TPS.

NSRRC is committed to supporting users throughout this transition. We will continue offering training courses and workshops to help users maximize the potential of the new TPS facilities and broaden their research opportunities. By embracing these changes, we will ensure our user community remains at the forefront of global scientific research.

Looking to the future, NSRRC will continue pushing the boundaries of scientific frontiers, advancing accelerator technologies, and driving innovation in experimental techniques. Our goal is not just to collect data but to transform the data into actionable insights that can help solve the world's fundamental challenges and support industries in overcoming critical issues.



Chia-Hung Hsu
Director

Preface

NSRRC is a research facility that provides powerful light sources and experimental capabilities not readily available in in-house laboratories to facilitate scientific advances and foster innovation. Thanks to the efforts of all users and our hard-working staff, 2024 continues to be a very productive year. To give an overview of the diverse activities that took place throughout this year, the editorial committee has carefully selected a range of research publications using the experimental facilities offered in Taiwan Photon Source (TPS), Taiwan Light Source (TLS), Taiwan Contract Beamlines at SPring-8, and the neutron facilities to illustrate NSRRC's contributions. One thing worth mentioning here is the growth of contributions from TPS. While such a trend is expected due to the construction of TPS phase-III beamlines entering its fourth year, and the continuation of TLS-to-TPS transition, I would like to express my sincere thanks to those efforts that are not necessarily reflected in terms of scientific publications.

In the Research Highlights of this volume, we feature 31 fascinating research topics across sections of Physics and Materials Science, Chemical Science, Soft Matter, Life Science, Energy Science, Environmental and Earth Sciences, and Neutron Science. In Facility Development and Status, we present four articles that explore the operational status and advancements in accelerator technology, including the optimization of the undulator system, performance of the energy-saving initiatives, and the innovative helium phase separator; additionally, two newly opened experimental facilities at the TPS—Soft X-ray Nanoscopy Beamline and Tender X-ray Spectroscopy Beamline—are introduced with detailed descriptions.

Facts and Figures provides a snapshot of NSRRC's operations, offering key statistics on administrative aspects, user engagement, beamline list, major events, and outreach activities. The Appendix includes lists of NSRRC publications resulting from the use of NSRRC beamlines, or related to instrumentation, accelerator facility, and neutron project, as well as student dissertations and theses, further highlighting NSRRC's impact in the science community.

One goal the editorial committee hopes to achieve is to channel research happening at NSRRC to a broader readership. This task is a challenge itself. Enjoy reading.



Der-Hsin Wei
Deputy Director



Research Highlights



Physics and Materials Science

This year has seen groundbreaking research in physics and materials science, leading to advancements in magnetism, quantum materials, high-entropy oxides, and phosphors. These discoveries drive innovations in spintronics, superconducting qubits, and next-generation electronics. One study explores circular dichroism in Ni_3TeO_6 , an observed giant X-ray natural circular dichroism effect linked to parity-time symmetry breaking and Berry curvature, revealing the characteristic of altermagnetism and evidence for coupling between spin and chirality. Another study solves a puzzle in ferrimagnetic alloys, demonstrating how Mn-moment switching and Fe-moment increase influence Curie temperature in $\text{Gd}_6(\text{Mn}_{1-x}\text{Fex})_{23}$. In 2D quantum materials, researchers highlight the potential of ultrathin β -Sn as a 2D topological nodal line semimetal, advancing quantum computing. High-entropy spinel oxide ($\text{Fe, Co, Ni, Cr, Mn})_3\text{O}_4$ films exhibit strain-induced crystalline magnetic anisotropy due to Mn 3d orbital changes. Rare-earth phosphor research reveals stable Eu-doped BaAl_2O_4 despite non-uniform doping, enhancing LED technology. Lastly, nanometer-thick aluminum films grown *via* molecular beam epitaxy with an Al_2O_3 capping layer demonstrate high crystallinity and stable superconducting properties, which are essential for quantum circuits. These studies exemplify cutting-edge research and shape future technological advancements. (by Cheng-Maw Cheng)

Discovery of Giant X-ray Natural Circular Dichroism in an Antiferromagnet with Time-Reversal Symmetry

A structurally chiral, polar, and collinear antiferromagnet with effective time-reversal symmetry and no net spin moments is shown to exhibit a giant X-ray natural circular dichroism.

Circular dichroism is the difference in the absorption of right- and left-handed circularly polarized (RCP and LCP, respectively) light. It is a valuable spectroscopic tool used for studying the magnetic properties and structural chirality of molecules, proteins, and solids. For example, a typical ferromagnet with a net magnetic moment represents a broken time-reversal symmetry state and shows an X-ray magnetic circular dichroism (XMCD) signal that provides the spin- and orbital-magnetic moments of constituent atoms. Although an antiferromagnet (AFM) is not expected to show an XMCD signal as it does not have a net magnetic moment, some studies have shown that an XMCD signal can be observed in AFM materials with non-collinear or chiral spin structures. More recently, the so-called “altermagnets” with no net spin moments have also shown XMCD, which were understood as originating from broken time-reversal symmetry. In contrast, AFM materials with conserved time-reversal symmetry but broken space inversion symmetry can exhibit X-ray natural circular dichroism (XNCD) without an applied magnetic field.^{1,2}

It is known that XNCD originates from the interference between electric-dipole (E1) and electric-quadrupole (E2) transitions.² However, its magnitude is orders of magnitude lower than those of typical XMCD signals. In a path-finding study led by Di-Jing Huang (NSRRC) and conducted in collaboration with scientists from Taiwan, the United States, Japan, and Germany, researchers have now discovered a giant XNCD in X-ray absorption spectroscopy (XAS) of a collinear AFM Ni_3TeO_6 that exhibits time-reversal symmetry. The authors used spatially resolved ($\sim 1 \mu\text{m}$) XAS to study Ni_3TeO_6 that possesses a polar and chiral crystal structure with a collinear magnetic structure.³ The results also showed that the observed giant XNCD changes its sign between domains of opposite crystal chirality with the magnetic structure keeping time-reversal symmetry.

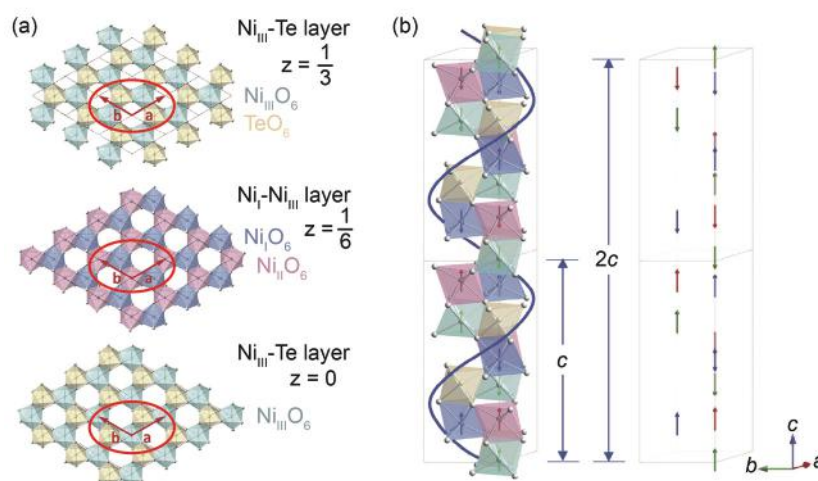


Fig. 1: Crystal and spin structures of Ni_3TeO_6 . (a) The crystal structure of Ni_3TeO_6 consists of ferromagnetically coupled Ni_IO_6 and $\text{Ni}_{III}\text{O}_6$ honeycomb layers alternating with the $\text{Ni}_{III}\text{O}_6$ and TeO_6 honeycomb layers. (a) and (b) are in-plane unit-cell vectors. The structural unit cell comprises six honeycomb layers stacked along the c -axis. (b) Chiral crystal structure of Ni_3TeO_6 . The blue helix represents the handedness of the crystal chirality. The spins represented by arrows are aligned along the c -axis, showing the collinear antiferromagnetic structure of Ni_3TeO_6 .⁵ The magnetic unit cell consists of two structural unit cells, that is, 12 honeycomb layers stacking along the c -direction. The structural unit-cell length c and the magnetic unit-cell length $2c$ are indicated by double-headed arrows. [Reproduced from Ref. 3]

Nickel tellurite, Ni_3TeO_6 , crystallizes in a modified corundum structure and is known to exhibit a large optical rotation associated with chiral and polar domains.⁴ The three Ni sites and one Te site in the structure form two kinds of honeycomb layers consisting of edge-shared NiO_6 and TeO_6 octahedra stacked along the c -axis, generating the polar crystal structure. The ferromagnetically coupled Ni_IO_6 and $\text{Ni}_{III}\text{O}_6$ honeycomb layer alternates with the $\text{Ni}_{III}\text{O}_6$ and TeO_6 honeycomb layer, as shown in **Fig. 1(a)**.

Ni_3TeO_6 shows an AFM transition with a Néel temperature of $T_N = 62 \text{ K}$, from a paramagnetic state to a commensurate collinear AFM state with Ising anisotropy,⁵ as schematically shown in **Fig. 1(b)**. This is confirmed by momentum scans of resonant X-ray magnetic scattering (RXMS) with an incident X-ray beam spot size of $\sim 1 \mu\text{m}$, measured as a function of temperature (**Figs. 2(a) and 2(b)**, see next page) at positions A and B, respectively, as shown in **Fig. 3(a)** (see next page). The results show that the intensity of the $(0, 0, 1.5)$ peak appears below T_N and steadily increases with decreasing temperature. These results of temperature-dependent RXMS at the Ni L_3 -edge XAS (**Figs. 2(a)–2(c)**)

and magnetic susceptibility measurements (Fig. 2(d)) are consistent with the collinear AFM structure of Ni_3TeO_6 . The observed AFM transition is marked by a sharp cusp in the parallel-to- c -axis component χ_{\parallel} of the magnetic susceptibility. These results also agree with the results of temperature-dependent X-ray linear dichroism (XLD) measured at TPS 41A and plotted in Fig. 2(c).

Ni_3TeO_6 is actually considered to be a unique material, as it exhibits both chirality and polarity. However, because of its polar $R3$ structure, polarity aligns with chirality, and this makes it impossible to observe different polarities within the same chiral domain concurrently. The authors then circumvented this difficulty by carrying out spatially resolved temperature-dependent XAS and XNCD mapping over an area of $90 \times 90 \mu\text{m}^2$ with a resolution of $\sim 1 \mu\text{m}$ in order to characterize the domain structure, as shown in Figs. 3(a) and 3(c). The authors could distinguish two types of chiral domains by performing Ni L_3 -edge XAS measurements at TPS 41A using circularly polarized X-rays.

Most importantly, the authors observed a relatively large XNCD for two distinct types of domains for temperatures below $\approx T_N$, as shown in Fig. 3(b), despite the absence of both an external magnetic field and remanent magnetization. The measured dichroic spectra, *i.e.*, the RCP

– LCP difference spectra, show a maximum magnitude of XNCD of approximately 20% of the Ni L_3 -edge XAS peak, much larger than the previously reported one of 2%,⁶ thus indicating a giant XNCD in Ni_3TeO_6 . The observed giant XNCD exhibits a non-linear dependence on the X-ray intensity, showing a positive peak at $\omega_{\text{RT}} + 0.3 \text{ eV}$ at position A and a negative peak at $\omega_{\text{RT}} + 0.7 \text{ eV}$ at position B. The dichroic maps shown in Fig. 3(c) demonstrate a sign change between the domains of opposite chirality. The color contrast between the domains increases with temperature, and it becomes strongest around T_N and invisibly weak at room temperature. The XNCD intensities at A and B plotted as functions of temperature in Fig. 3(d) show a clear enhancement below $\approx T_N$.

The magnitude of the observed XNCD in Ni_3TeO_6 is comparable to the XMCD of $3d$ transition metals but is much larger than the usual XNCD resulting from the E1–E2 interference. This is indicative of a new mechanism going beyond the E1–E2 interference, likely arising from a uniform “effective magnetic field” active in Ni_3TeO_6 . From a symmetry perspective, moving X-ray photons in a chiral crystal structure can induce an effective magnetic field along their propagation direction. The authors propose a theoretical model to explain the giant XNCD, which is attributed to a broken PT symmetry, where P is the parity and T is time reversal. In particular, the authors could

show from their model that the XNCD cross-section of L-edge XAS is proportional to the integration of Berry curvature over momentum at a fixed energy.³ In accordance with the XMCD sum rule, the energy integration of XNCD is proportional to the integration of Berry curvature across the entire momentum space, leading to an orbital moment. Thus, the chiral structure of Ni_3TeO_6 ensures a non-zero average orbital magnetization responsible for the “effective magnetic field” and the giant XNCD. Further, the observed XNCD shows a strong temperature dependency, just like the XMCD from a ferromagnet across its magnetic phase transition, revealing the characteristic of altermagnetism and evidence for coupling between spin and chirality. All these properties of Ni_3TeO_6 establish the first example of a new class of magnetic materials that can exhibit circular dichroism with time-reversal symmetry. (Reported by Ashish Chainani, Jun Okamoto and Di-Jing Huang)

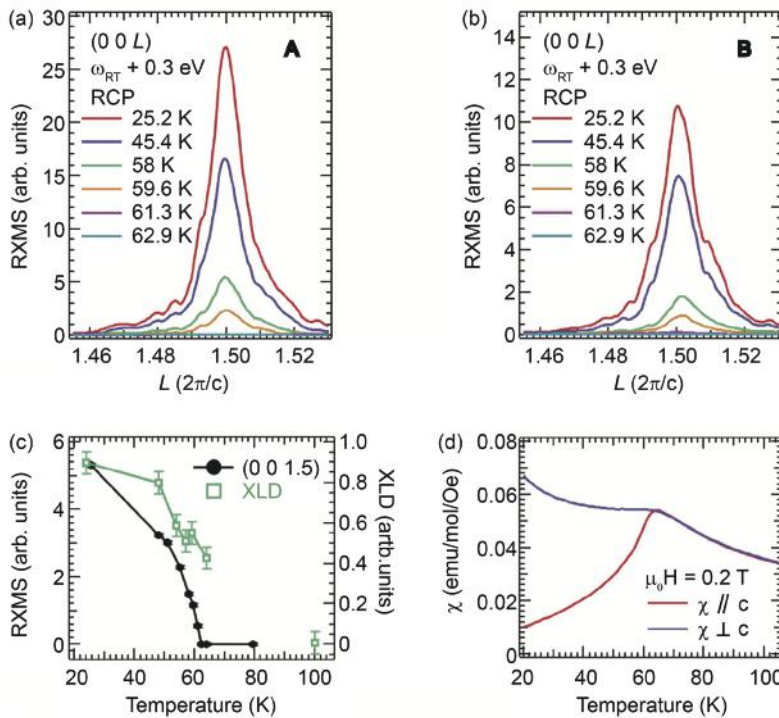


Fig. 2: Magnetic transition of Ni_3TeO_6 . (a,b) RXMS intensities at $q^*=(00L)$ in the reciprocal space scanned along L with RCP at the photon energy of $\omega_{\text{RT}}+0.3 \text{ eV}$ as a function of temperature measured at positions A and B, respectively. Here, ω_{RT} is the Ni L_3 -edge XAS peak energy, and A and B belong to opposite chiral domains shown in Fig. 3(a). (c) Temperature dependence of XLD and the $(0, 0, 1.5)$ peak of RXMS at the Ni L_3 edge plotted across the Néel transition. (d) DC magnetic susceptibility for magnetic field parallel and perpendicular to the c axis taken with $\mu_0 H = 0.2 \text{ T}$. [Reproduced from Ref. 3]

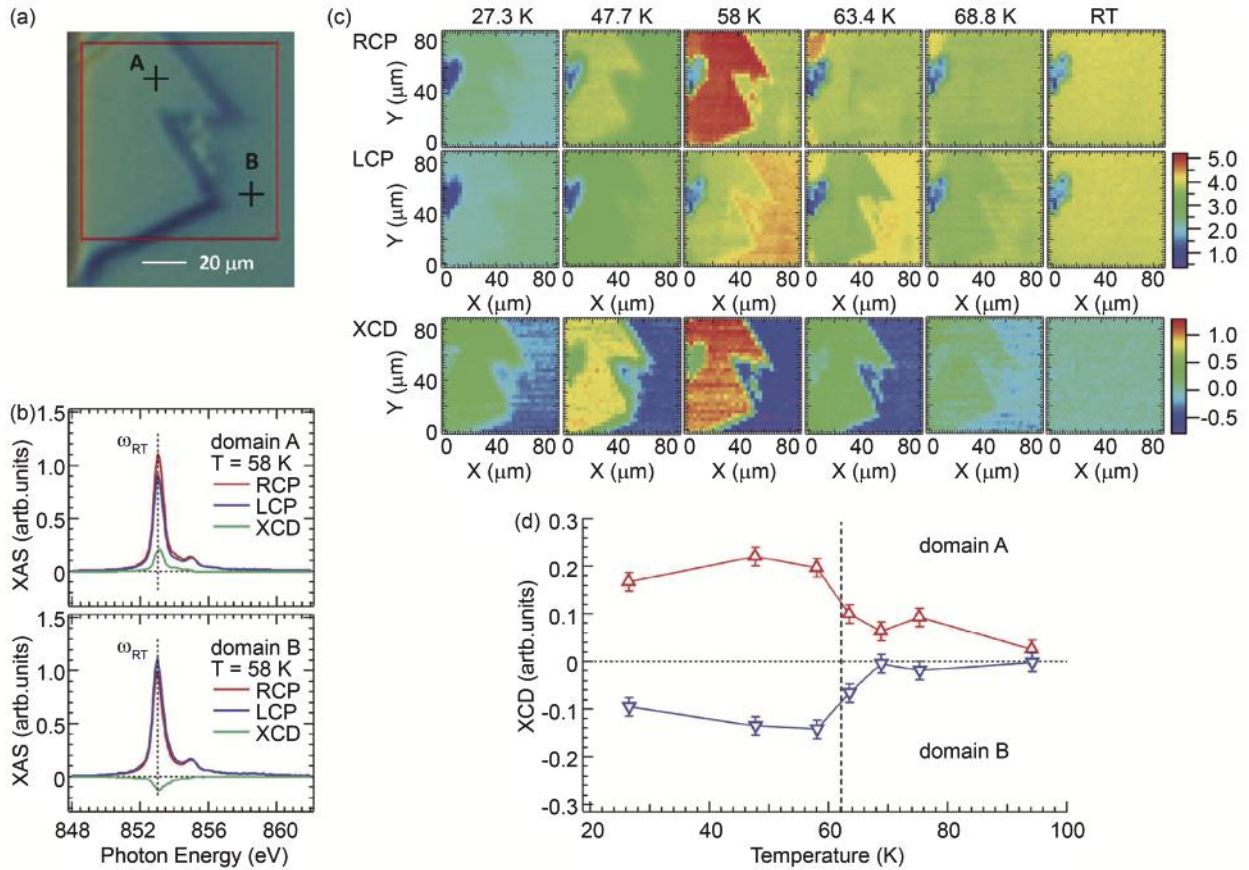


Fig. 3: Temperature-dependent XNCD of Ni_3TeO_6 . (a) Image of the Ni_3TeO_6 sample recorded with a polarized-light microscope. Horizontal and vertical edges are parallel to the $[0\ 1\ \bar{1}\ 0]$ and $[2\ \bar{1}\ \bar{1}\ 0]$ axes, respectively. The red-square box shows a $90 \times 90 \mu\text{m}^2$ area on which the XAS and XCD images were taken. Positions A and B are in two different domains of opposite crystal chirality. (b) XAS spectra taken at positions A and B with RCP and LCP and difference spectra, *i.e.*, X-ray circular dichroism spectra, at 58 K measured by the fluorescence-yield method. Circular dichroism signals at A and B are positive and negative, respectively. (c) XAS images of Ni_3TeO_6 in the $90 \times 90 \mu\text{m}^2$ area measured at a photon energy of $\omega_{\text{RT}} + 0.7 \text{ eV}$ (photon energy of ω_{RT} at RT) with RCP (top) and LCP (middle) and RCP – LCP difference images (bottom) at various temperatures. (d) XCD intensity normalized to the L_3 XAS peak intensity, defined by $2(\text{RCP} - \text{LCP})/(\text{RCP} + \text{LCP})$, at positions A and B plotted against temperature. The dichroism changes its sign between A and B. [Reproduced from Ref. 3]

This report features the work of Jun Okamoto, Di-Jing Huang and their collaborators published in *Adv. Mater.* **36**, 2309172 (2024).

TPS 41A Soft X-ray Scattering

- XAS, XNCD, RXMS
- X-ray Natural Circular Dichroism, Antiferromagnetic Transition, Materials Science, Condensed-matter Physics

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Element-Specific Curie Temperatures and Heisenberg Criticality in Ferrimagnetic $\text{Gd}_6(\text{Mn}_{1-x}\text{Fe}_x)_{23}$

Temperature-dependent X-ray magnetic circular dichroism solves a long-standing puzzle of non-monotonic Curie temperatures coupled to monotonic composition-controlled magnetization.

Many rare-earth (R)–transition-metal (M, M') ternary alloys of the type $\text{R}_a(\text{M}_{1-x}\text{M}'_x)_b$ exhibit non-monotonic ferrimagnetic Curie temperatures (T_C) coupled to monotonic composition-controlled magnetization. Its origin has remained an important long-standing puzzle in the absence of studies investigating their temperature (T)-dependent element-specific magnetism. To answer this question, a team of scientists from the NSRRC, Taiwan, and Institut Jean Lamour, France, conducted a systematic study of the ferrimagnetic series $\text{Gd}_6(\text{Mn}_{1-x}\text{Fe}_x)_{23}$, $x = 0.0–0.75$. The researchers used the experimental techniques of X-ray absorption spectroscopy (XAS), X-ray magnetic circular dichroism (XMCD), and hard X-ray photoemission spectroscopy (HAXPES) in combination with density functional theory calculations, including on-site Coulomb energy (DFT+ U), to determine the answer. For the first time, the researchers successfully applied the Kouvel–Fisher analysis method to element-specific T -dependent XMCD data. The study unambiguously revealed distinct sublattice T_C values and 3-D Heisenberg critical behavior of the ferrimagnetic transition in $\text{Gd}_6(\text{Mn}_{1-x}\text{Fe}_x)_{23}$, as well as clarified the origin of non-monotonic ferrimagnetic T_C coupled to monotonic composition-controlled magnetization.¹

As shown in **Figs. 1(a)–1(f)**, the researchers performed XAS–XMCD measurements at the Dragon beamline **TLS 11A1**, with an applied field of ± 1 T for the series $\text{Gd}_6(\text{Mn}_{1-x}\text{Fe}_x)_{23}$, $x = 0.0–0.75$ after characterization of the

crystal structure and ferrimagnetic T_C values obtained from magnetization measurements. **Figures 1(a)–1(c)** show representative XAS spectra for (a) Gd M-edge and (b) Mn L-edge of the parent compound, as well as (c) the Fe L-edge of $\text{Gd}_6(\text{Mn}_{0.5}\text{Fe}_{0.5})_{23}$ measured at $T = 29$ K. From such measurements of compounds with $x = 0.0–0.75$, the researchers obtained XMCD spectra, as shown for all x in panels (d) to (f). While the Gd M-edge XMCD spectra for all x indicate a total magnetic moment of $\sim 7 \mu_B$, the Mn L-edge XMCD shows a small net magnetic moment ($\sim 0.4 \mu_B$), which switches from parallel to antiparallel with respect to the Gd magnetic moments and becomes $\sim 0.05 \mu_B$ on increasing x . By contrast,

Fe L-edge XMCD shows that Fe magnetic moments are antiparallel with respect to the Gd magnetic moments for all x and increase systematically from $\sim 0.4 \mu_B$ (for $x = 0.2$) to $1.4 \mu_B$ (for $x = 0.75$).

Next, the researchers performed HAXPES measurements at **SP 12U1**, one of the Taiwan-contract Beamlines in SPring-8, for the series and showed that the Gd $3d$ core-level multiple spectra can be analyzed using an intermediate coupling scheme.² By contrast, the Fe $2p$ core-level spectra showed single peaks attributed to typical metals, whereas the Mn $2p$ spectra showed a main peak and shoulder, which could be assigned to Mn up-spin and down-spin sites.

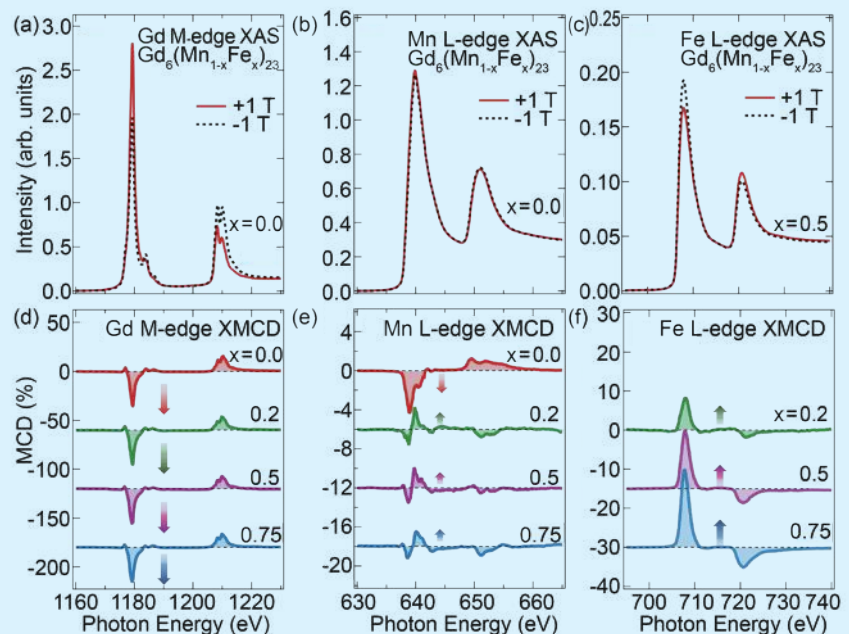


Fig. 1: Representative XAS spectra of $\text{Gd}_6(\text{Mn}_{1-x}\text{Fe}_x)_{23}$ with an applied field of ± 1 T for (a) Gd M-edge ($x = 0$), (b) Mn L-edge ($x = 0$), and (c) Fe L-edge ($x = 0.5$) measured at $T = 29$ K, from which we obtained the XMCD spectra, as shown for all x in panels (d)–(f). XAS–XMCD spectra of (d) Gd M-edge, (e) Mn L-edge, and (f) Fe L-edge of the series $\text{Gd}_6(\text{Mn}_{1-x}\text{Fe}_x)_{23}$ ($x = 0, 0.2, 0.5, \text{ and } 0.75$) measured at $T = 29$ K. Arrows in (d)–(f) show the relative magnitude and orientation of the spins. [Reproduced from Ref. 1]

This observation is consistent with the crystal and magnetic structures of the parent $\text{Gd}_6\text{Mn}_{23}$, as obtained from neutron diffraction studies,³ which indicated two types of Mn “b, d” up-spin sites and two types of Mn “f₁, f₂” down-spin sites, as shown in Fig. 2(a). The Fe and Mn 3s core level HAXPES spectra showed exchange splitting, and a Van-Vleck analysis result indicated magnetic moments that are consistent with neutron diffraction results.²

Next, the researchers conducted bulk-sensitive HAXPES valence band spectroscopy and compared it with DFT+*U* electronic structure calculations. Figure 2(b) shows the comparison between the experimental valence band spectrum and the calculated partial density of states (P-DOS) for Gd 4*f*, Gd 5*d*, Mn 4*s*, and Mn 3*d* and the total DOS for $\text{Gd}_6\text{Mn}_{23}$ obtained from DFT+*U* calculations with on-site Coulomb energies $U_{\text{Mn}}^{\text{DFT}} = 0.75$ eV and $U_{\text{Gd}}^{\text{DFT}} = 6.5$ eV. The optimal values of $U_{\text{Mn}}^{\text{DFT}} = 0.75$ eV and $U_{\text{Gd}}^{\text{DFT}} = 6.5$ eV also yielded magnetic moment values consistent with neutron diffraction results.³ The crucial role of on-site Coulomb energies in the valence band states of transition metals was further clarified by 2*p*–3*d* resonant-PES.⁴ Figure 2(c) shows a plot of the valence band P-DOS for Gd 4*f*, Gd 5*d*, Fe 4*s*, and Fe 3*d* and the total DOS for $\text{Gd}_6\text{Fe}_{23}$ obtained from the DFT+*U* calculations. To compare with experimental data for $x = 0.75$, the calculated total DOS for $\text{Gd}_6(\text{Mn}_{0.25}\text{Fe}_{0.75})_{23}$ was approximated as a sum of 25% $\text{Gd}_6\text{Mn}_{23}$ and 75% $\text{Gd}_6\text{Fe}_{23}$ total DOS, plotted together with the bulk-sensitive HAXPES valence band spectrum in Fig. 2(d). The results indicated a fair match between experimental and calculated results.¹

The researchers conducted *T*-dependent XMCD and bulk magnetization experiments to determine element-specific T_C values and the nature of the ferrimagnetic transition in $\text{Gd}_6(\text{Mn}_{1-x}\text{Fe}_x)_{23}$.

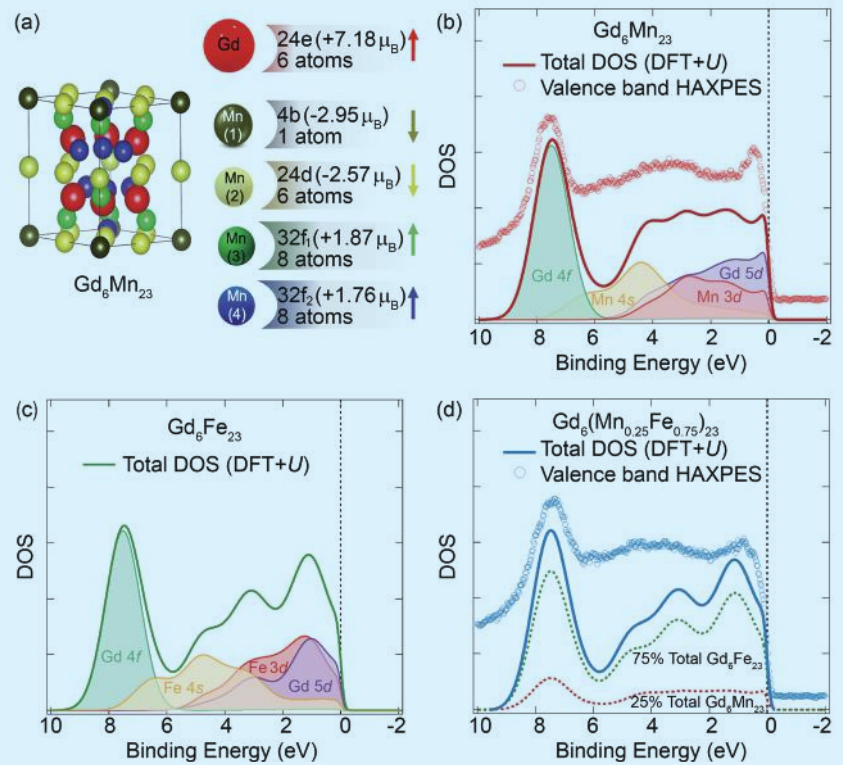


Fig. 2: Crystal structure schematic and comparison of HAXPES valence band with DOS calculations. (a) The crystal structure of $\text{Gd}_6\text{Mn}_{23}$, indicating the magnetic moments from the band structure calculations of the Gd site, Mn “b, d” sites, and Mn “f₁, f₂” sites. (b) The P-DOS for Gd 4*f*, Gd 5*d*, Mn 4*s*, and Mn 3*d* and the total DOS for $\text{Gd}_6\text{Mn}_{23}$ obtained from DFT+*U* calculations with $U_{\text{Mn}}^{\text{DFT}} = 0.75$ eV and $U_{\text{Gd}}^{\text{DFT}} = 6.5$ eV compared with bulk-sensitive HAXPES valence band spectrum. (c) The valence band P-DOS for Gd 4*f*, Gd 5*d*, Fe 4*s*, and Fe 3*d* and the total DOS for $\text{Gd}_6\text{Fe}_{23}$ obtained from DFT+*U* calculations. (d) The valence band total DOS for $\text{Gd}_6(\text{Mn}_{0.25}\text{Fe}_{0.75})_{23}$ is approximated by an additive mixture of 25% $\text{Gd}_6\text{Mn}_{23}$ and 75% $\text{Gd}_6\text{Fe}_{23}$ total DOS, and it is plotted together with the bulk-sensitive HAXPES valence band spectrum. [Reproduced from Ref. 1]

Figure 3 (see next page) shows the *T*-dependent Gd/Fe XMCD results fitted to a power law for all *x*. The data revealed that Gd $T_C = 273.5$ K for $x = 0.0$ and 0.75 and that it was smaller than the bulk T_C values (489 K for $x = 0$; 306 K for $x = 0.75$). In contrast, it decreased to the bulk T_C values of 172 K (for $x = 0.2$) and 135 K (for $x = 0.5$) when Gd T_C decreased to below 273.5 K. A scaling analysis (Fig. 3(b)) indicated a 3D Heisenberg-type transition for all *x* values. To confirm the critical behavior, a more rigorous Kouvel–Fisher analysis was performed for all *x* values, and the results are shown in the bottom row of Fig. 3. The *T*-dependent Mn L-edge XMCD could not be measured as it showed weak XMCD intensity. The results confirmed the 3-D Heisenberg critical behavior of the ferrimagnetic transition in $\text{Gd}_6(\text{Mn}_{1-x}\text{Fe}_x)_{23}$.¹

Finally, a magnetic phase diagram (Fig. 4, see next page) based on element-specific T_C values with total magnetization from XMCD and total bulk magnetization measurements (M_{Tot}^X and M_{Tot}^B , respectively) showed three regions with (i) Mn-sublattice bulk- $T_C >$ Gd-sublattice T_C , (ii) a reduced common- T_C for all sublattices, and (iii) Fe-sublattice bulk- $T_C >$ Gd-sublattice T_C . The results thus revealed that the Mn-moment switching and gradual increase in Fe moment combined to cause non-monotonic T_C values coupled to monotonic magnetization. The study indicates the importance of element-specific T_C values for tuning magnetic properties in $\text{R}_a(\text{M}_{1-x}\text{M}'_x)_b$ ternary alloys. (Reported by Ashish Chainani)

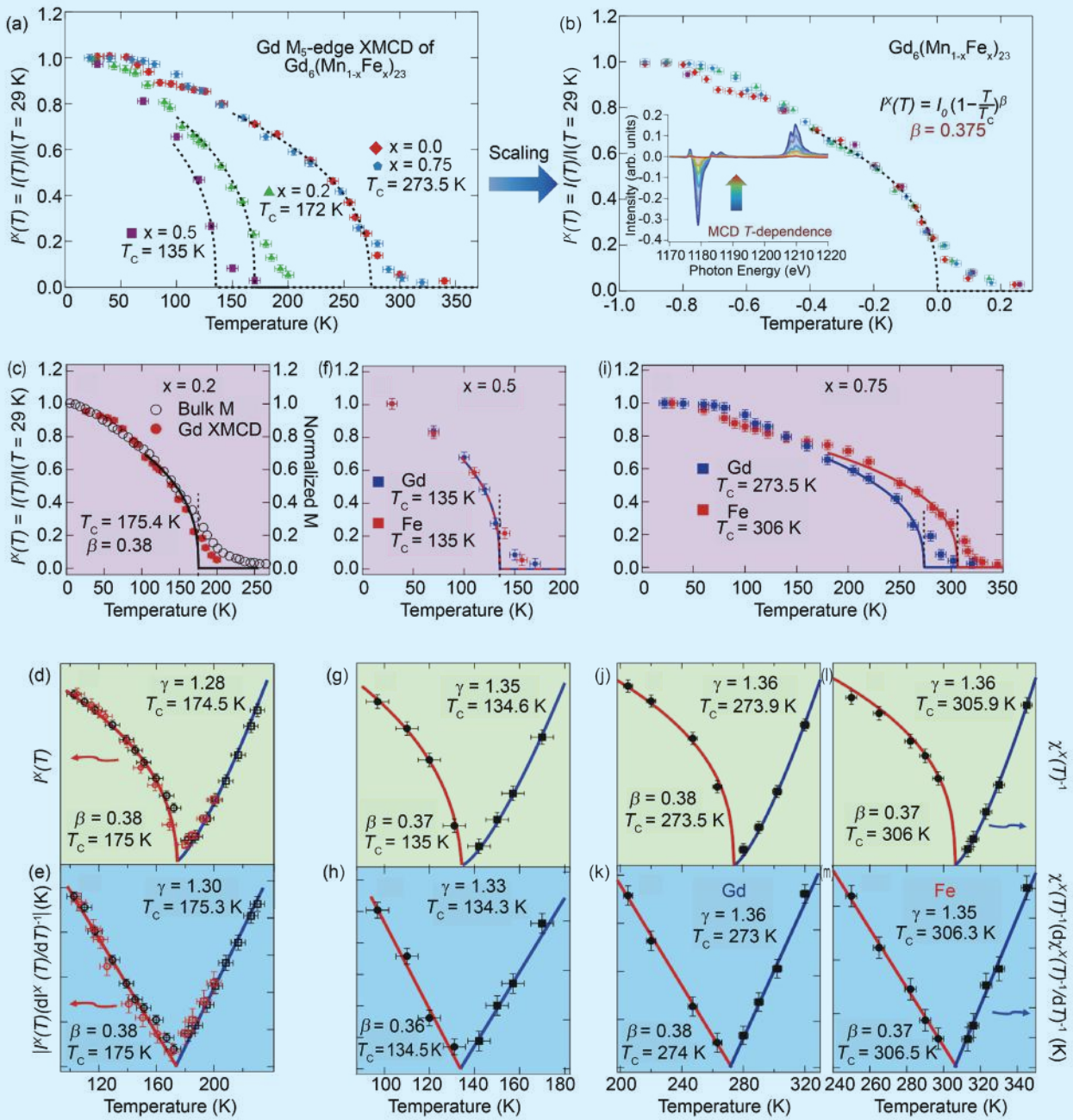


Fig. 3: Element-specific T_C values from power law analyses and Kouvel–Fisher analyses of XMCD. (a) T dependence of the Gd M_{5-} edge XMCD intensity for $Gd_6(Mn_{1-x}Fe_x)_{23}$, fitted by a power law exponent $\beta = 0.38 \pm 0.01$ (dashed black line). (b) Critical scaling behavior for the $Gd_6(Mn_{1-x}Fe_x)_{23}$ series. The inset shows T dependence of Gd M -edge XMCD intensity for $Gd_6(Mn_{0.25}Fe_{0.75})_{23}$. (c) T dependence of the Gd M_{5-} edge XMCD intensity and total bulk magnetization ($M_{tot}^B(T)$) of $Gd_6(Mn_{0.8}Fe_{0.2})_{23}$ fitted by a power law with $\beta = 0.38 \pm 0.01$ (black line). (d) Power-law and (e) Kouvel–Fisher analysis fits for $M_{tot}^B(T)$ (black empty circles). The Gd M_{5-} edge XMCD intensity $I^X(T)$ plotted together (red empty circles) also follows the Kouvel–Fisher analysis for $M_{tot}^B(T)$ near T_C . (f) T dependence of the Gd M_{5-} edge (blue squares) and Fe L_{3-} edge (red squares) XMCD intensity for $Gd_6(Mn_{0.5}Fe_{0.5})_{23}$ fitted to a power law with $\beta = 0.37 \pm 0.01$ (dashed blue-red line). (g) Power-law and (h) Kouvel–Fisher analyses for both Gd M_{5-} edge and Fe L_{3-} edge XMCD intensities plotted together. (i) T dependence of the Gd M_{5-} edge (blue squares) and Fe L_{3-} edge (red squares) XMCD intensity for $Gd_6(Mn_{0.25}Fe_{0.75})_{23}$ fitted by a power law with $\beta = 0.38 \pm 0.01$ (blue and red lines, respectively). (j) Power-law and (k) Kouvel–Fisher analyses for Gd M_{5-} edge XMCD intensities, and (l) power-law and (m) Kouvel–Fisher analyses for Fe L_{3-} edge XMCD intensities. The red and blue lines in (d), (e), (g), (h), (j)–(m) are the fits below and above T_C , respectively. All XMCD results were obtained with an applied field of ± 1 T. [Reproduced from Ref. 1]

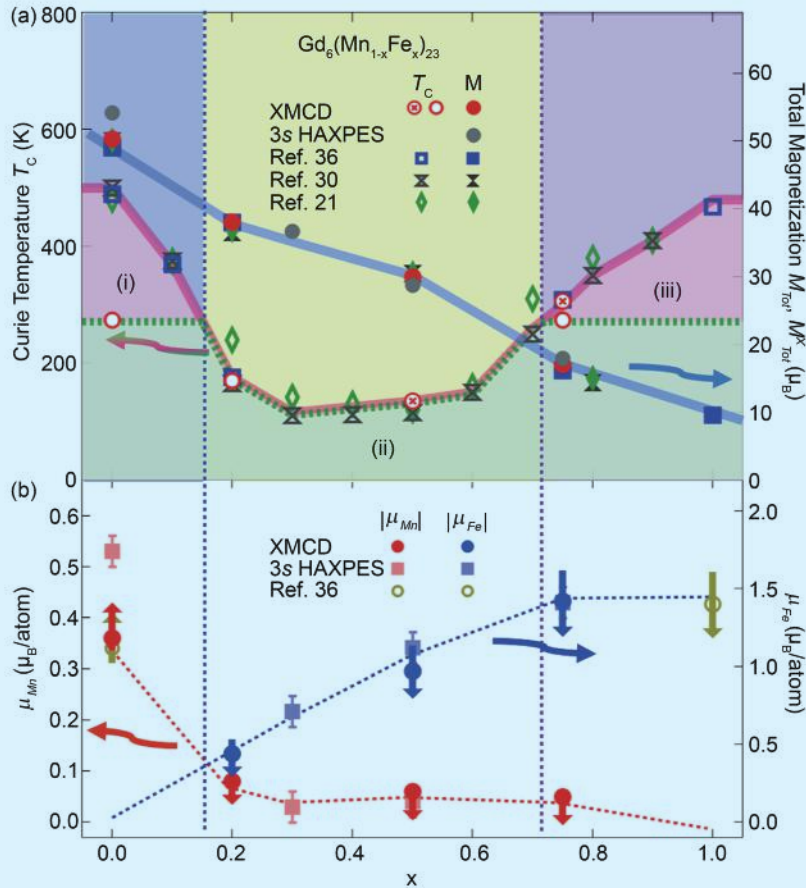


Fig. 4: Magnetic phase diagram of $Gd_6(Mn_{1-x}Fe_x)_{23}$. (a) Summary of element-specific T_C values with XMCD and bulk magnetizations (M_{Tot}^x and M_{Tot}^B) as a function of x . The plots of $T_{C(Gd)}$ (empty red circle \circ) and $T_{C(Fe)}$ (red cross circle \otimes) show three regions: (i) For $0.0 < x \leq 0.15$, bulk T_C is determined by the Mn sublattice, and $T_{C(Gd)} = 273.5 \text{ K} < \text{bulk } T_C$. (ii) For $0.15 < x \leq 0.72$, the Gd and Fe sublattices show the same $T_C = \text{bulk } T_C$. (iii) For $0.72 < x \leq 1.0$, the Fe moments determine the bulk T_C , and $T_{C(Gd)} = 273.5 \text{ K} < \text{bulk } T_C$. (b) Summary of Mn and Fe magnetic moments (μ_{Mn} , μ_{Fe}) as a function of x , showing switching of Mn moments for $x \geq 0.2$ plotted with magnetic moments from 3s HAXPES analysis. [Reproduced from Ref. 1]

This report features the work of Ashish Chainani and his collaborators published in *Commun. Mater.* **5**, 68 (2024) and *Physical Review B* **109**, 035102 (2024).

TLS 11A1 (Dragon) MCD, XAS SP 12U1 HAXPES/Photoemission

- XAS, XMCD, HAXPES
- Ferrimagnetic Transition, Materials Science, Condensed-matter Physics

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Unlocking Dual Topological States in the 2D Limit

A promising way enhances the understanding of 2D topological materials and lays the groundwork for future electronic and superconducting applications.

Two-dimensional (2D) quantum materials have gained attention for their exceptional electronic properties, particularly in spintronics and quantum computing. Among them, 2D topological insulators (TIs) are notable for their protected metallic edge states, which exhibit spin-momentum locking and insusceptibility to backscattering from nonmagnetic impurities. These features make them strong candidates for low-power electronic devices and quantum computing. The discovery of topological nodal line semimetals (TNLSMs), which host one-dimensional nodal lines of band degeneracy in the Brillouin zone, has further expanded the landscape of topological materials. However, experimental realization of 2D TNLSMs remains rare despite numerous theoretical predictions. Stanene, the monolayer allotrope of tin (Sn), has emerged as a promising 2D TI because of its sizable topological band gap ($\sim 0.3 \text{ eV}$) induced by strong spin-orbit coupling (SOC). Unlike silicene and germanene, which possess much smaller SOC-induced gaps, stanene's robust band inversion enables the quantum spin Hall effect at room temperature. Additionally, stanene's electronic properties can be tuned *via* strain

engineering and substrate interactions, making it a versatile platform for investigating topological phase transitions. In contrast, β -Sn, a metallic allotrope of tin with a body-centered tetragonal structure, exhibits superconducting behavior and has potential applications in topological superconductivity. However, the electronic structure of ultrathin β -Sn films remains largely unexplored, leaving its potential as a 2D TNLSM unexamined.

To advance understanding of 2D TNLSMs, Pin-Jui Hsu and Horng-Tay Jeng, both from National Tsing Hua University, and Cheng-Maw Cheng from the NSRRC investigated the electronic structure of monolayer β -Sn grown on a Cu(111) substrate by using scanning tunneling microscopy (STM), angle-resolved photoemission spectroscopy (ARPES), and density functional theory (DFT) calculations. Their study probed the structural and electronic phase transition from honeycomb stanene (α -Sn) to cubic β -Sn as Sn coverage increased. The transition from a 2D TI to a 2D TNLSM was directly observed, revealing the coexistence of type-I and type-III nodal lines in monolayer β -Sn for the first time. Experimental observations aligned well with DFT calculations, confirming that β -Sn exhibits a nearly freestanding electronic structure on Cu(111), an unexpected behavior in a metal-on-metal system. This suggests that β -Sn/Cu(111) provides an ideal platform for studying intrinsic 2D TNLSM properties. Furthermore, integrating superconducting few-layer Sn with topological nodal line monolayer β -Sn could open new avenues for exploring 2D topological superconductivity and Majorana fermions. Comprehensive ARPES experiments at the **TLS 21B1** beamline were carried out on high-quality α -Sn and β -Sn thin films prepared on Cu(111) using molecular beam epitaxy. A single layer of α -Sn was first grown on a Cu(111) substrate at low temperature. STM and low-energy electron diffraction revealed a honeycomb lattice of stanene with a $p(2\times 2)$ supercell. ARPES measurements confirmed the band structure's consistency with reported results. When an equivalent amount of Sn atoms was subsequently deposited onto the stanene/Cu(111) at low temperature, the honeycomb lattice disappeared, and a high-coverage Sn (HC-Sn) cubic β -Sn phase emerged, as observed in the STM images. This structural phase transition from semiconducting honeycomb α -Sn to metallic cubic β -Sn provided a unique opportunity to investigate this topological transition in the band structure of ultrathin β -Sn(001) thin films.

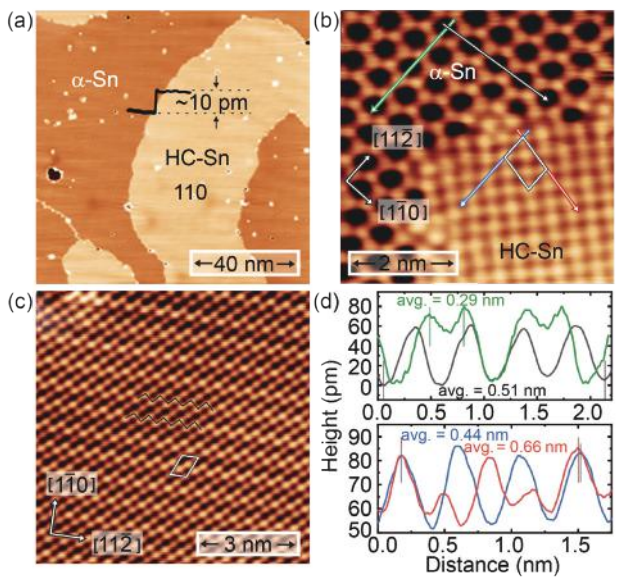


Fig. 1: (a) STM topographic overview of about 0.54 ML Sn grown on Cu(111). (b) Atomically resolved image at the boundary between α -Sn and HC-Sn. The white rhombuses indicate the unit cells of α -Sn ($p(2\times 2)$) and HC-Sn. (c) STM image of HC-Sn on a larger scan area with atomic resolution. The periodic zigzag pattern (black zigzag stripes) has been clearly resolved. White rhombus frame denotes the unit cell. (d) Top panel: black and green curves are topographic line profiles taken along the arrow lines on the α -Sn phase along $[1\ \bar{1}\ 0]$ and $[1\ \bar{1}\ \bar{2}]$ directions, respectively, in (b). Bottom panel: topographic line profiles (red and blue curves) taken along the arrow lines on HC-Sn in (b). [Reproduced from Ref. 1]

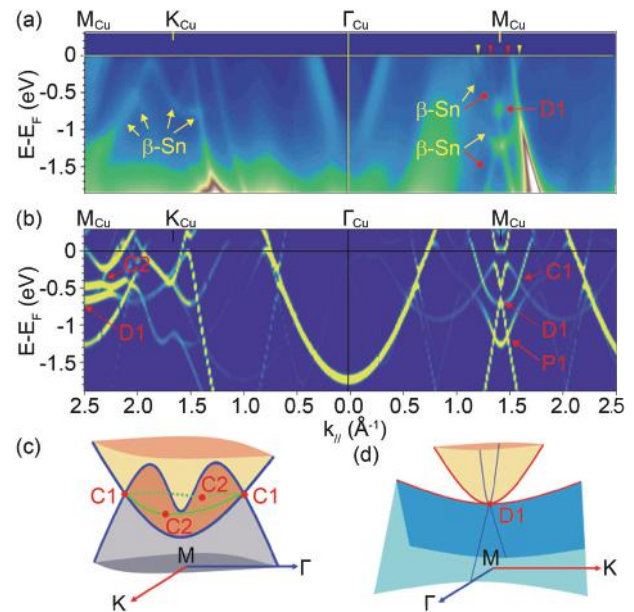


Fig. 2: (a) The band dispersions of single-layer β -Sn/Cu(111) along $M_{Cu}-K_{Cu}-\Gamma_{Cu}-M_{Cu}$ relative to Cu(111) high-symmetry point direction. (b) The calculated unfolding band structure of freestanding monolayer β -Sn along the high-symmetry lines in the BZ of Cu(111). Schematic diagram of type-I (c) and type-III (d) nodal line band crossings. C1 and C2 in (c) and D1 in (d) near the high-symmetry point M_{Cu} are indicated by the red arrows in (a) and (b). [Reproduced from Ref. 1]



STM topographic imaging of Sn thin films grown on Cu(111) at 80 K revealed distinct regions corresponding to α -Sn and β -Sn phases. We identified a honeycomb lattice with a $p(2\times 2)$ supercell in the darker regions characteristic of α -Sn, while the brighter regions exhibited a higher-coverage Sn phase with a denser atomic arrangement. DFT calculations confirmed that the interlayer distance between the top β -Sn layer and the first Cu layer was 2.40 Å, consistent with the STM findings. To explore evolution in electronic structure from low-coverage (LC) to high-coverage (HC) Sn phases on Cu(111), ARPES measurements were conducted. Relative to pristine Cu(111), a linear band dispersion with a Dirac point at 0.62 eV binding energy emerged around the M_{Cu} point for β -Sn/Cu(111) shown in **Fig. 2(a)**, indicating an electronic topological transition. Two wave vectors crossing the Fermi level were identified, along with two electron pockets around the M_{Cu} point: one with significant Fermi level crossing and another deeper in the binding energy region. Additional β -Sn-derived bands, which are confirmed as 2D features through photon energy-dependent ARPES experiments, appeared at the K_{Cu} point. DFT band structure calculations were performed for both freestanding monolayer β -Sn and β -Sn/Cu(111). The unfolded band structure of freestanding β -Sn monolayer closely matched ARPES results, particularly around the M_{Cu} and K_{Cu} points. The band-crossing points C1 and D1 near M_{Cu} were identified as type-I and type-III nodal line semimetal features. The C1 crossing formed a closed ring-shaped type-I nodal line, while the D1 crossing exhibited a type-III nodal line characterized by a cone-saddle intersection. These findings confirm that β -Sn/Cu(111) hosts a dual-nodal line semimetal phase, making it an exciting candidate for further exploration of 2D TNLSMs.

Similar to reported 2D nodal line materials such as CuSe, AgSe, and Cu₂Si, monolayer β -Sn possesses mirror reflection symmetry. In three-dimensional TNLSMs, nodal line projections onto surfaces generate drumhead-like flat surface bands protected by topological invariants. However, in 2D TNLSMs, node lines do not directly protect edge states, complicating their observation. Despite this challenge, β -Sn's coexistence of type-I and type-III nodal lines presents an exceptional case for further research. Few experimental realizations of 2D TNLSMs exist due to stringent symmetry and SOC requirements. While theoretical predictions suggest that external perturbations like strain or electric fields can induce a TNLSM phase in some 2D materials, experimental verification remains scarce. The discovery of type-I and type-III gapless nodal line behaviors in β -Sn/Cu(111) marks a significant step forward. These findings, backed by STM-ARPES-DFT investigations, establish monolayer β -Sn as a novel 2D dual-nodal line semimetal, offering a valuable platform for exploring topological superconductivity and other quantum phenomena.

To summarize, by elucidating topological transition and nodal line behavior in ultrathin β -Sn films, this work enhances the understanding of 2D topological materials and lays the groundwork for future electronic and superconducting applications. The unique properties of β -Sn highlight its potential as a novel material for next-generation quantum devices. (Reported by Cheng-Maw Cheng)

This report features the work of Pin-Jui Hsu, Horng-Tay Jeng, Cheng-Maw Cheng and their collaborators published in ACS Nano 18, 20990 (2024).

TLS 21B1 Angle-resolved UPS

- High-resolution ARPES
- Materials Science, Condensed-matter Physics

Reference

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Single-Element-Driven Crystalline Magnetic Anisotropy in the High-Entropy Oxide (Fe, Co, Ni, Cr, Mn)₃O₄

X-ray absorption spectroscopy, magnetic circular dichroism, and linear dichroism show that crystalline magnetic anisotropy in (Fe, Co, Ni, Cr, Mn)₃O₄ originates from the orbital anisotropy of Mn 3d states due to substrate-induced epitaxial strain.

The design of multicomponent materials or so-called high-entropy alloys and compounds has attracted enormous attention due to their extraordinary ability to tailor functional properties. However, it is not easy to identify the role of each element in terms of their impact on crystal structure and its relation to electronic and magnetic properties in such materials. In a recent study on high-entropy spinel oxide (Fe, Co, Ni, Cr, Mn)₃O₄ epitaxial films, Ying-Hao Chu's team (National Tsing Hua University) successfully grew high-quality films with varying strain states, and then carried out a detailed investigation on the crystal structure and magnetic properties, as well as used electron microscopy and synchrotron spectroscopy techniques to determine element-specific properties. The team discovered that the high-entropy oxide (HEO) thin film under compressive strain shows evidence of crystalline magnetic anisotropy that is derived from a single element.¹ Using a combination of X-ray absorption spectroscopy (XAS), X-ray magnetic circular dichroism (XMCD), and X-ray linear dichroism (XLD), the team showed that the observed crystalline magnetic anisotropy originates from orbital occupation changes in Mn 3d states induced by epitaxial strain in the films.

The team first fabricated high-entropy (Fe, Co, Ni, Cr, Mn)₃O₄ epitaxial films on single-crystal MgAl₂O₄ (MAO) and MgO substrates with a high-quality interface. This resulted in elimination of defects in the HEO film and imposed compressive and tensile strain on HEO films grown on MAO and MgO, respectively. The team then carried out extensive analyses for a detailed understanding of HEO films, including X-ray diffraction (XRD), reciprocal space mapping, Raman spectroscopy, macroscopic magnetic characterization, electron microscopy, and synchrotron spectroscopy. The XRD rocking curves of the HEO (004) peak on MAO and MgO substrates

measured at TLS 17A1 show full width at half maximum (FWHM) of 0.42° and 0.14°, respectively, demonstrating the superior crystallinity of HEO films on these substrates. Reciprocal space mapping (RSM) was then used to show that the HEO film is under an in-plane compressive strain of 0.52% on the MAO substrate, while the HEO film is under an in-plane tensile strain of 0.85% on the MgO substrate. Raman spectroscopy showed four peaks at 527 cm⁻¹ (3F_{2g}), 588 cm⁻¹ (3F_{2g}), 648 cm⁻¹, and 700 cm⁻¹ (A_{1g}). The wavenumbers of 648 and 700 cm⁻¹ indicate the octahedral and tetrahedral centers in HEO. Accordingly, the crystal structure of HEO was classified as a spinel based on results from XRD and Raman spectroscopy. Scanning transmission electron microscopy with electron energy loss spectroscopy further confirmed that the HEO films exhibited a spinel phase.

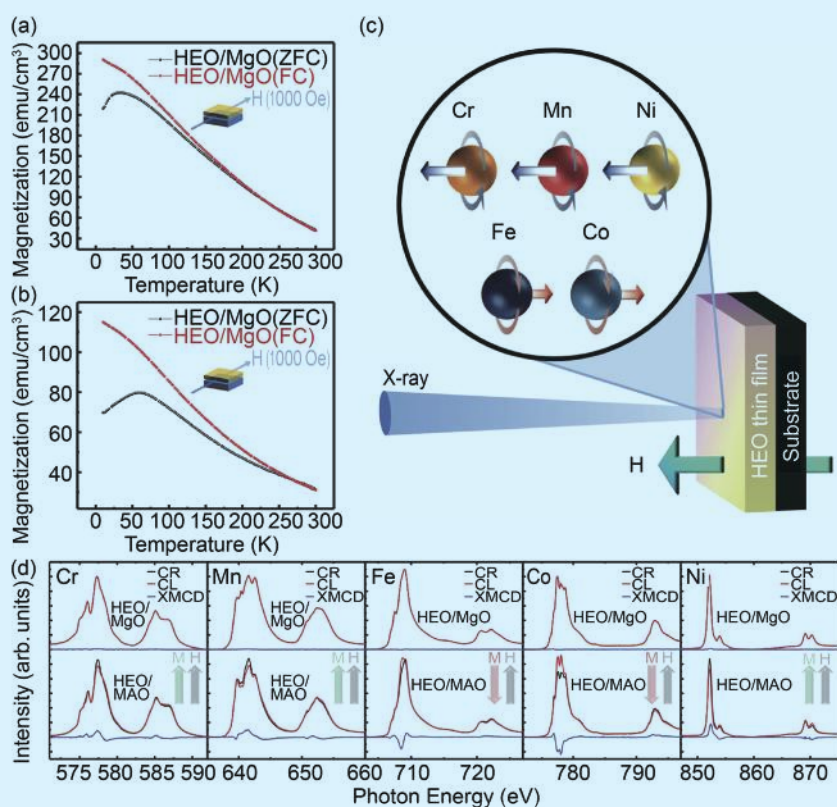


Fig. 1: M(T) and XMCD results. Temperature-dependent magnetization for (a) HEO/MgO and (b) HEO/MAO heterostructures under ZFC and FC processes with a DC magnetic field of 1000 Oe along the in-plane direction. (c) Schematic diagram illustrating the relationship between the applied magnetic field and the direction of the element's magnetic moment. (d) XMCD spectra of each element for the HEO/MgO and HEO/MAO structures. [Reproduced from Ref. 1]

Next, in order to understand the magnetic behavior of HEO films as a function of temperature, zero-field-cooled (ZFC) and field-cooled (FC) magnetization (M_{ZFC} and M_{FC} , respectively) measurements of the HEO/MgO and HEO/MAO heteroepitaxies were carried out as shown in **Figs. 1(a) and 1(b)**. It was found that both samples show a spin-glass-like behavior since considerable deviation was observed between M_{ZFC} and M_{FC} below ~ 280 K.

The team carried out XMCD measurements at **TPS 45A**, with the samples in a remanence state and the incident X-ray normal to the surface at 200 K, as shown schematically in **Fig. 1(c)**. The samples were magnetized with a field of 3 T normal to the sample surface before they were introduced into the chamber. **Figure 1(d)** shows clear XMCD signals for all five transition metals. For the HEO/MAO structure, a positive XMCD signal at the main absorption peak of the Cr, Mn, Ni L_3 edge spectra was observed. In contrast, Fe and Co spectra showed a negative XMCD signal at the L_3 edge. These results indicate that the spin direction of Cr, Mn, and Ni corresponds to the direction of the applied magnetic field (parallel), and that of Fe and Co is opposite to the applied magnetic field (antiparallel), as shown in **Fig. 1(c)**. In addition, a higher-intensity XMCD signal for the HEO/MAO structure and a lower-intensity XMCD signal for HEO/MgO were observed (**Fig. 1(d)**). The team concluded that because of the strong in-plane shape of its magnetic anisotropy, the magnetic moments of the HEO/MGO structure are aligned in-plane,

while the magnetic moments of the HEO/MAO structure are aligned with the out-of-plane direction. This suggests that crystalline magnetic anisotropy dominates over the shape of magnetic anisotropy in the HEO/MAO structure. These results were further confirmed by superconducting quantum interference device (SQUID) magnetometry measurements. The authors also carried out SQUID measurements of HEO/MAO samples with film thicknesses from ~ 50 nm to over $1 \mu\text{m}$ and quantified the relationship between anisotropy energy density and film thickness. These results show that the thinner is the HEO film, the greater is the anisotropy energy density ($\sim 3 \times 10^6 \text{ erg cm}^{-3}$), indicating that it is more affected by crystalline magnetic anisotropy. It is speculated that this magnetic anisotropy behavior comes from the epitaxial strain imposed by the substrate.

The team then carried out XAS measurements at $T = 300$ K as shown in **Fig. 2(a)** in order to check the valency and local geometry of the transition-metal ions. **Figure 2(b)** shows a schematic illustrating 3d orbital splitting when transition-metal elements occupy octahedral and tetrahedral sites. By comparing with standard reference samples, we concluded that Cr ions exhibit octahedral coordination with a valency of Cr^{3+} . On the other hand, Mn was found to adopt multiple valence states with Mn^{2+} in tetrahedral coordination and with Mn^{3+} and Mn^{4+} in octahedral coordination. The Mn^{2+} ion exhibits a half-filled 3d orbital, implying that it has a spherically symmetric

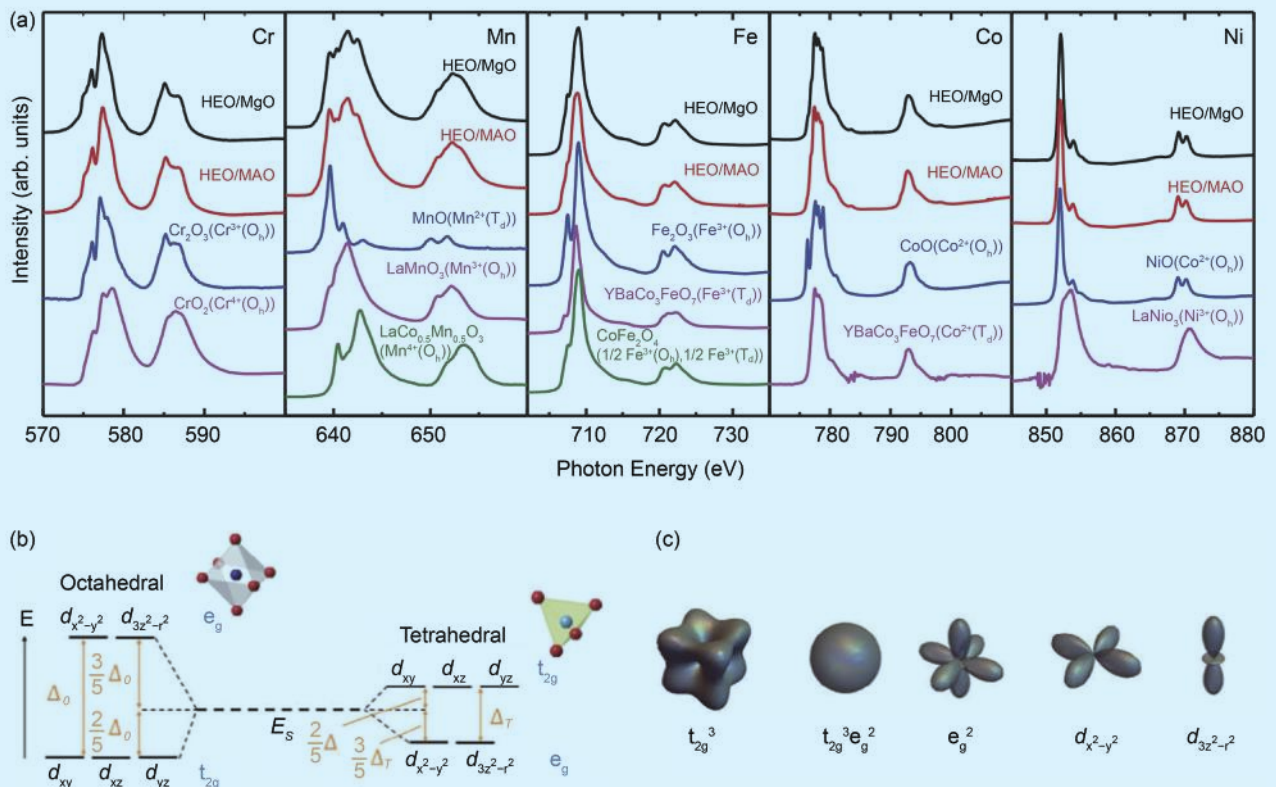


Fig. 2: XAS measurements. (a) XAS spectra of HEO/MgO and HEO/MAO structures. (b) 3D orbital splitting of a transition-metal ion at the octahedral and tetrahedral sites into e_g orbitals ($d_{x^2-y^2}$, $d_{3z^2-r^2}$) and t_{2g} orbitals (d_{xy} , d_{xz} , d_{yz}). (c) The electron clouds of different occupied orbitals. [Reproduced from Ref. 1]

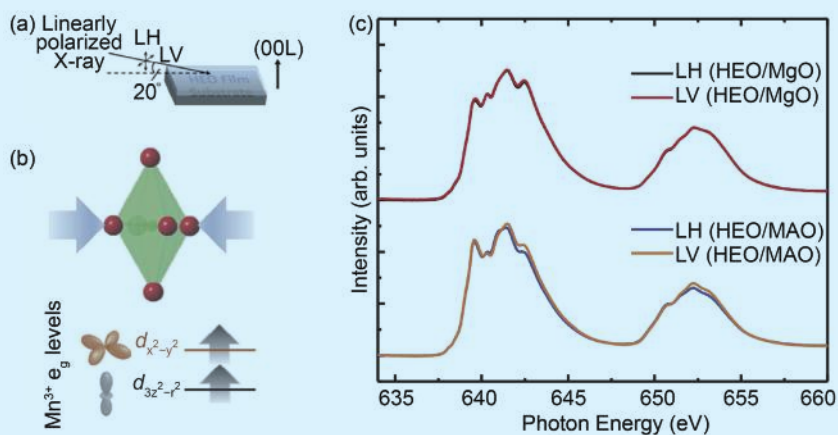


Fig. 3: XLD measurements. (a) Schematic of an XLD measurement. (b) A representation of MnO_6 under compressive strain and the subsequent effect on e_g splitting. (c) Mn $L_{2,3}$ XLD spectrum of the HEO/MgO and HEO/MAO structures. [Reproduced from Ref. 1]

electron cloud shape of $t_{2g}^3e_g^2$, as shown in **Fig. 2(c)**. For Mn^{4+} octahedral coordination, three electrons occupy the d_{xy} , d_{xz} , and d_{yz} orbitals, forming an electron cloud with cubic symmetry in t_{2g}^3 , similar to that of Cr^{3+} . Similarly, it was found that Fe atoms show Fe^{3+} valency and adopt a half-filled 3d orbital for octahedral and tetrahedral sites, both corresponding to a spherically symmetric electron cloud shape $t_{2g}^3e_g^2$, while Co atoms show Co^{2+} valency in tetrahedral coordination with a fully occupied e_g but half-filled t_{2g} shell. Therefore, it exhibits an electron cloud with cubic symmetry in $t_{2g}^3e_g^4$. Lastly, Ni atoms exhibit an octahedral Ni^{2+} configuration with a fully occupied t_{2g} shell and half-filled e_g shell, resulting in a $t_{2g}^6e_g^2$ configuration and an electron cloud with cubic symmetry.

Finally, the team employed XLD to probe the asymmetric orbital occupation of Cr, Mn, Fe, Co, and Ni to verify the correlation between strain states and magnetic behavior. In the XLD measurements, linearly polarized X-rays were used with a grazing angle of 20° as depicted in **Fig. 3(a)**. The out-of-plane polarization (LH) absorption of Cr, Fe, Co, and Ni L edge spectra was very similar to the in-plane polarization (LV) absorption of the HEO/MAO structure. It was concluded that because of their symmetrical electron cloud shapes, these four elements should not contribute to orbital anisotropy. On the other hand, **Fig. 3(c)** indicates a discernible difference in the XLD spectrum of the Mn L edge absorption between the HEO/MgO (tensile) and HEO/MAO (compressive) structures. The LH absorption of the Mn L edge spectrum is weaker than that of LV in the HEO/MAO, suggesting a larger number of in-plane empty states in the e_g band. This indicates a higher occupancy of out-of-plane ($3z^2-r^2$) orbitals. This occupancy is primarily influenced by the strain state, which in turn determines the magnetic behavior of the HEO thin film. Since only the Mn^{3+} cation contains four electrons in the 3d orbitals,

the crystalline magnetic anisotropy in HEO/MAO is primarily attributed to the higher electron occupancy of ($3z^2-r^2$) orbitals. **Figure 3(b)** depicts a schematic diagram illustrating the splitting of 3d orbitals in octahedral Mn under in-plane compressive strain. Thus, the authors could show that the difference in magnetic anisotropy originates from distortion in Mn^{3+} coordination, which is attributed to the strain applied from the substrate.

In conclusion, this study revealed that Mn^{3+} is the primary driver of crystalline magnetic anisotropy in the spinel HEO $(\text{Fe, Co, Ni, Cr, Mn})_3\text{O}_4$ due to strain-modified orbital occupation.¹ This study shows that advanced synchrotron techniques can be fruitfully used to investigate the role of individual elements in influencing the electronic and magnetic properties of HEOs and to provide valuable insights for understanding the fundamental physics of HEOs. (Reported by Ashish Chainani)

This report features the work of Ying-Hao Chu, Chang-Yang Kuo, Yi-Cheng Chen and their collaborators published in Adv. Funct. Mater. 34, 2312856 (2024).

TPS 45A Submicron Soft X-ray Spectroscopy TLS 17A1 X-ray Powder Diffraction

- XRD, XAS, XMCD, XLD
- Materials Science, Condensed-matter Physics

Reference

1. W.-E. Ke, J.-W. Chen, C.-E. Liu, Y.-C. Ku, C.-F. Chang, P. Shafer, S.-J. Lin, M.-W. Chu, Y.-C. Chen, J.-W. Yeh, C.-Y. Kuo, Y.-H. Chu, Adv. Funct. Mater. **34**, 2312856 (2024).

Visualization of the Distribution of Mixing Enthalpy and Entropy

Mixing enthalpy and entropy can be calculated by using the X-ray nanoprobe to understand the thermodynamic stability, solubility, and miscibility of the materials.

It is well known that high-entropy alloys (HEAs) can be studied by enthalpy and entropy, but have you ever thought about using them to analyze luminescent materials? E-Wen Huang (National Yang Ming Chiao Tung University) and his collaborators present a new approach to mapping thermodynamic parameters through the use of the spatially resolved synchrotron X-ray methods (TPS 23A and TPS 21A). It demonstrates how the distribution of mixing enthalpy and entropy correlates with the local optical properties of the activator Eu ions in the BaAl_2O_4 host lattice. The study clearly demonstrates the use of synchrotron radiation for the combinatorial approach, including X-ray nano-diffraction, X-ray fluorescence (XRF), X-ray absorption spectroscopy (XAS), and X-ray excited optical luminescence (XEOL), to map the valence states and spatial distributions of Eu^{2+} and Eu^{3+} ions. To understand the mechanisms by which the substitution of Eu ions in the host lattice affects the crystal structure and related properties, they quantify mixing enthalpy and entropy to explain the interactions between Eu ions and host elements, particularly Al and Ba. XRF maps (Fig. 1) not only provide the elemental distributions, such as those for Ba, Al, and Eu, but they also visualize the valence state distributions of Eu^{2+} and Eu^{3+} ions.

This study provides critical insights into the thermodynamic and structural stability of Eu ions, offering guidance for designing and optimizing phosphor materials with enhanced performance and stability (Fig. 2). The results reveal that Eu^{3+} ions aggregate within a specific region, while Eu^{2+} ions occupy the surrounding areas, highlighting distinct distribution patterns. Thermodynamic calculations show that Eu^{3+} ions exhibit favorable interactions with Al, which are characterized by a negative mixing enthalpy ($\Delta H < 0$) and a positive mixing entropy ($\Delta S > 0$), indicating high solubility and increased disorder. Conversely, the mixing behavior of Eu^{3+} ions with Ba is less favorable, as indicated by positive ΔH and ΔS values, which suggest repulsive interactions and greater disorder within

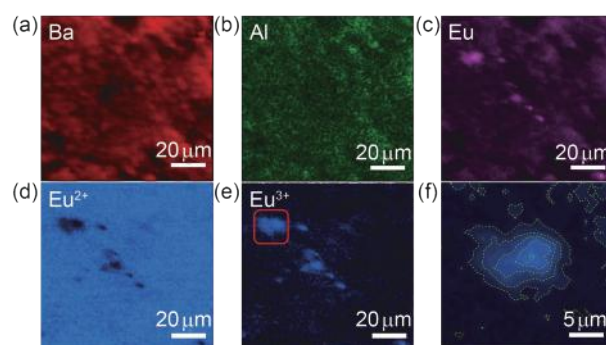


Fig. 1: XRF maps of (a) Ba, (b) Al, (c) Eu, (d) Eu^{2+} , and (e) Eu^{3+} in the Eu-doped BaAl_2O_4 . (f) The enlarged map of the red square area marked in (e). [Reproduced from Ref. 1]

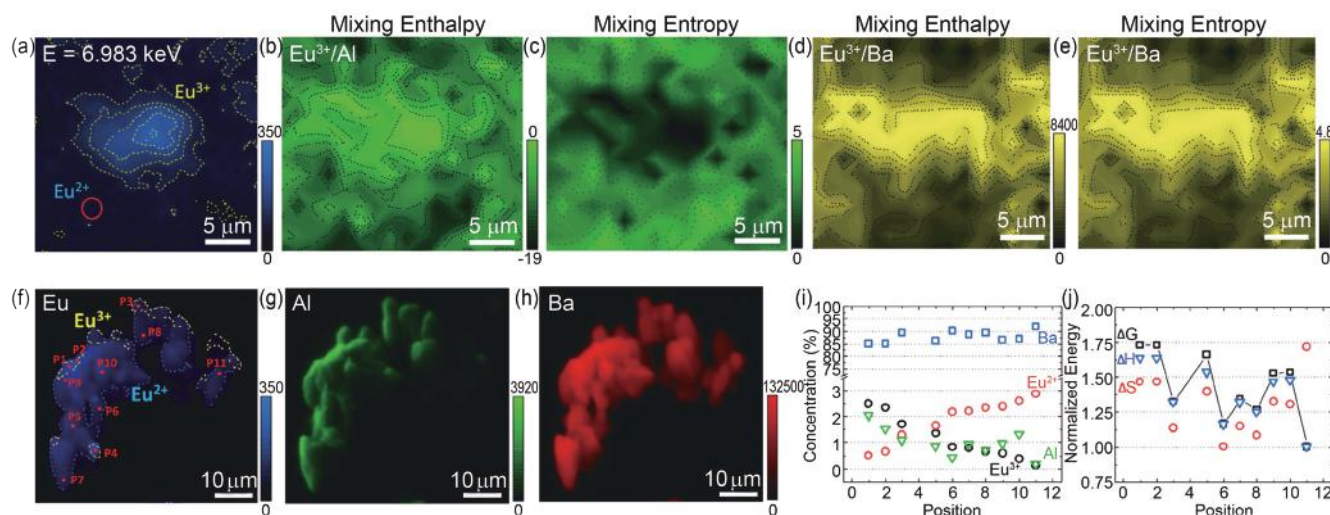


Fig. 2: (a) Distribution of Eu^{2+} and Eu^{3+} ions at X-ray energy of 6.983 keV. (b) Mixing enthalpy and (c) mixing entropy of Eu^{3+} ions and Al elements, respectively. (d) Mixing enthalpy and (e) mixing entropy of Eu^{3+} ions and Ba elements, respectively. (f) Distribution of Eu with 11 selected points for X-ray Laue diffraction. (g) Distribution of Al. (h) Distribution of Ba. (i) Concentration variation of constituent elements at different positions. (j) Gibbs free energy of mixing, mixing enthalpy, and mixing entropy at 11 different positions. [Reproduced from Ref. 1]

the system. The study further confirms that the crystal structure remains stable despite variations in composition, with no significant impact from Eu doping. Furthermore, Eu^{2+} ions display a stronger affinity for Al than Ba, leading to the observed aggregation of Eu^{3+} ions. These findings provide valuable insights into the thermodynamic behavior and structural stability of Eu ions, offering important guidance for the design and optimization of advanced phosphor materials.

The study investigates the effects of X-ray irradiation on BaAl_2O_4 doped with Eu^{2+} and Eu^{3+} ions, focusing on emission intensity and color changes (Fig. 3). In the XEOL spectrum, the peak at 500 nm corresponds to the Eu^{2+} $4f5d \rightarrow 4f$ transition, and the peaks at 588, 612, 650, and 698 nm are attributable to Eu^{3+} ${}^5\text{D}_0 \rightarrow {}^7\text{F}_i$ transitions. Under X-ray exposure, a significant decrease in Eu^{2+} emission intensity and a minor decrease in Eu^{3+} emission, which are likely due to thermal accumulation that increases non-radiative coupling, are observed. The ratio of Eu^{3+} to Eu^{2+} gradually increases with irradiation time, while X-ray absorption spectra indicate no significant changes in the electronic structure. The CIE chromaticity coordinates shift from (0.032, 0.524) to (0.043, 0.520), indicating a transition from green to white. This change is attributed to an increased presence of electrons in the conduction band, which is facilitated by X-ray-induced electron–electron scattering and phonon-assisted processes. The findings highlight the potential for X-ray dosimetry applications and tunable emission color strategies through defect state engineering in wide-bandgap semiconductors.

In summary, the behavior of Eu ions demonstrates that their aggregation is influenced by their different valence states and thermodynamic factors, providing insights into how rare-earth elements distribute and dissolve. Understanding how Eu ions interact with Al and Ba is important for designing and improving phosphor materials. The study further shows that even when doping is not uniform, the crystal structure maintains its stability, which helps improve the reliability of the doped materials. The assessment of thermodynamic parameters provides key data for understanding how rare-earth materials interact, supporting the development of efficient luminescent materials. This result demonstrates that the photoluminescence of the Eu-doped BaAl_2O_4 phosphor can be effectively modulated through the precise control of X-ray irradiation levels.

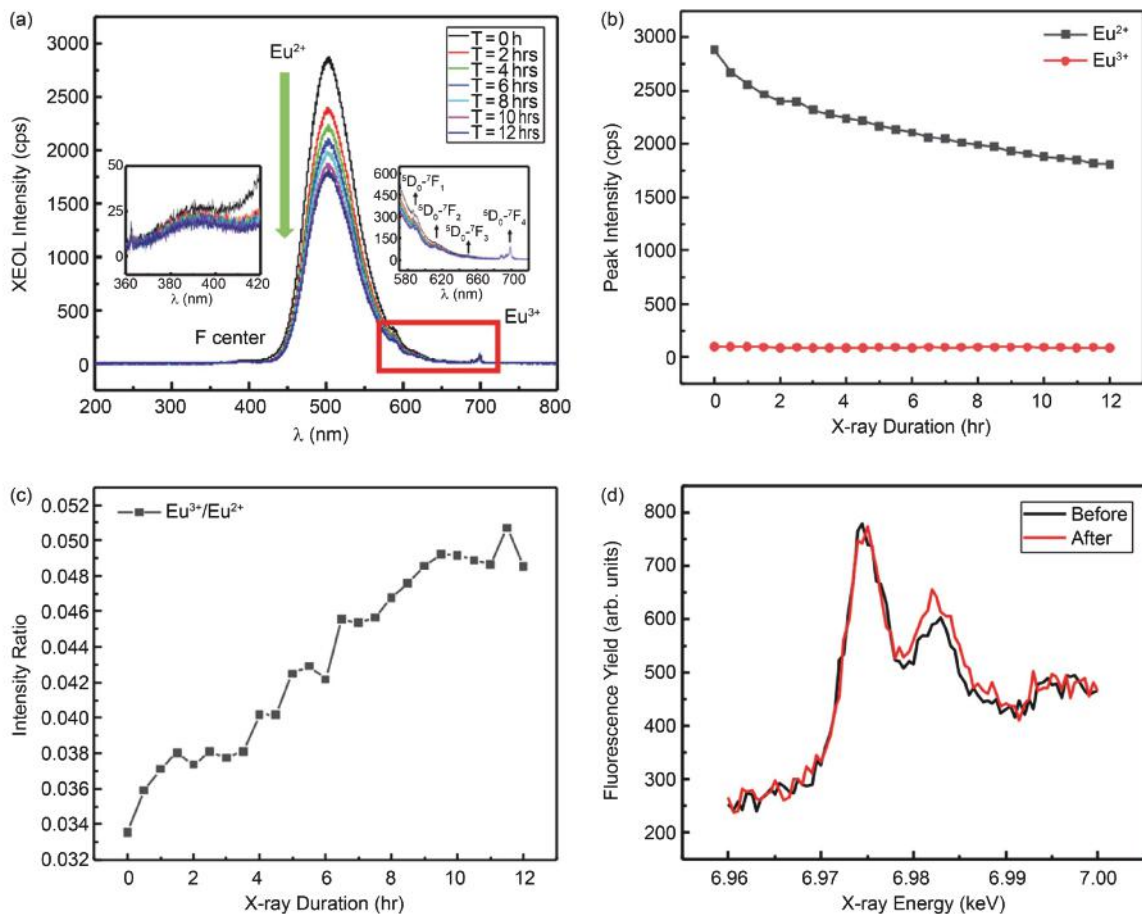


Fig. 3: (a) The XEOL spectrum of $\text{Eu}^{2+}/\text{Eu}^{3+}$ co-activated ions in BaAl_2O_4 phosphor; (b) the intensity variation of Eu^{2+} and Eu^{3+} emission peaks; (c) the intensity ratio of Eu^{2+} and Eu^{3+} emission peaks under X-ray irradiation from 0 to 12 h. (d) The variation of fluorescence yield characterized by X-ray absorption near edge structure before and after X-ray irradiation. [Reproduced from Ref. 1]

Overall, this study offers a theoretical foundation for designing and using rare-earth phosphor materials, focusing on thermodynamics and structural stability and supporting the ongoing development of high-performance white-light LED technologies. (Reported by Bi-Hsuan Lin)

This report features the work of E-Wen Huang and his collaborators published in *Appl. Phys. Lett.* **124**, 094105 (2024).

TPS 21A X-ray Nanodiffraction

TPS 23A X-ray Nanoprobe

- XRF, XEOL, XAS
- Materials Science, Structure, Emission Properties, Applications of High-Entropy Materials

Reference

1. Y.-H. Wu, T.-N. Lam, S.-W. Ke, W.-J. Lee, C.-Y. Lee, B.-Y. Chen, G.-C. Yin, W.-Z. Hsieh, C.-Y. Chiang, M.-T. Tang, B.-H. Lin, E.-W. Huang, *Appl. Phys. Lett.* **124**, 094105 (2024).

Exploring Quantum Materials by Using Synchrotron X-rays

Superconducting qubits composed of aluminum (Al) and aluminum oxide (Al₂O₃) play a pivotal role in advancing quantum information science.

The United Nations proclaimed 2025 as the International Year of Quantum Science and Technology (IYQ). Quantum technology will have a significant impact on human life. Synchrotron facility plays a vital role in the development of science and technology. This article will introduce how synchrotron X-rays can be used for quantum technology. Minghui Hong (National Taiwan University) and his collaborators investigate the structural and superconducting properties of nanometer-thick aluminum (Al) films grown on sapphire substrates using molecular beam epitaxy (MBE), with an emphasis on the use of an *in situ* deposited aluminum oxide (Al₂O₃) capping layer to maintain the pristine condition of the ultrathin films. This paper aims to describe the key challenges in developing materials for superconducting quantum circuits, where high crystallinity and minimized dielectric losses are essential for enhancing the coherence times. By carefully controlling the growth parameters and implementing the Al₂O₃ capping layer, they achieved high-quality Al films with remarkable crystallinity, abrupt interfaces with adjacent layers, and superconducting properties, which demonstrate potential for applications in quantum information technologies.

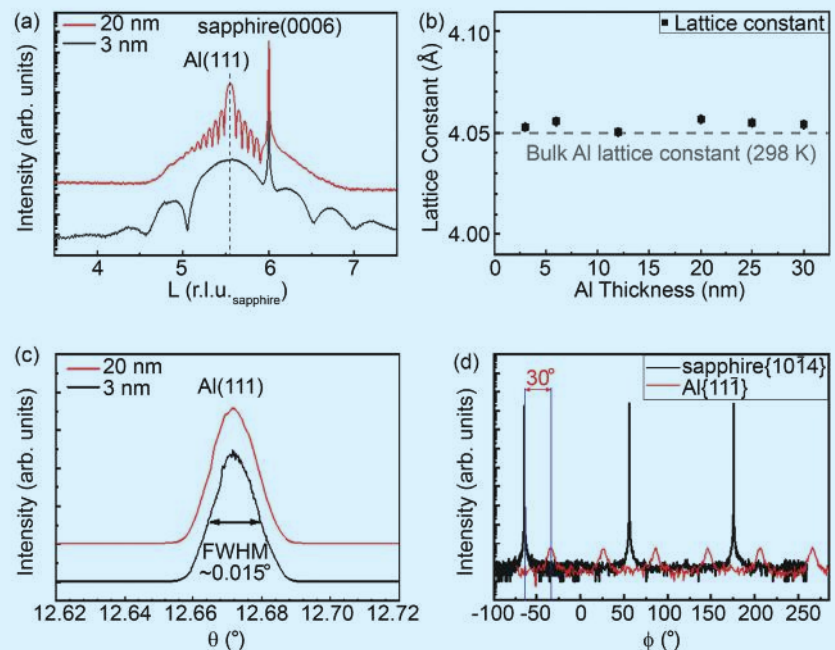


Fig. 1: (a) Radial scans along the surface normal of 3 and 20 nm Al film. (b) Lattice constants derived from inter-planar spacing along surface normal as a function of film thickness. (c) θ -rocking curves of 3 and 20 nm-thick Al films. (d) Azimuthal ϕ -scans across off-normal Al{111} and sapphire{1014} reflections. [Reproduced from Ref. 1]

Figure 1 demonstrates the high structural quality, crystallographic orientation, and stability of lattice properties in the nanometer-scale Al films. The radial scans along the surface normal, θ -rocking curves, and azimuthal ϕ scans exhibit the structure, crystallographic quality, and the epitaxial alignment of the nanometer-thick Al films. X-ray diffraction (XRD) using synchrotron X-ray (TLS 17B1 and TLS 13A1) can provide the capability for measuring the nanometer-thick Al films.

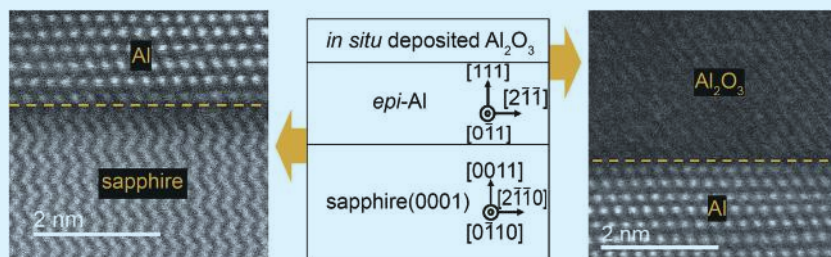


Fig. 2: High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) images of cross-sectional views of the MBE-grown *epi*-Al/sapphire heterostructure (left) and the *in situ* deposited Al₂O₃/*epi*-Al heterostructure (right). A schematic representation of the heterostructure (center) shows the orientations of the *epi*-Al film and the sapphire substrate. [Reproduced from Ref. 1]

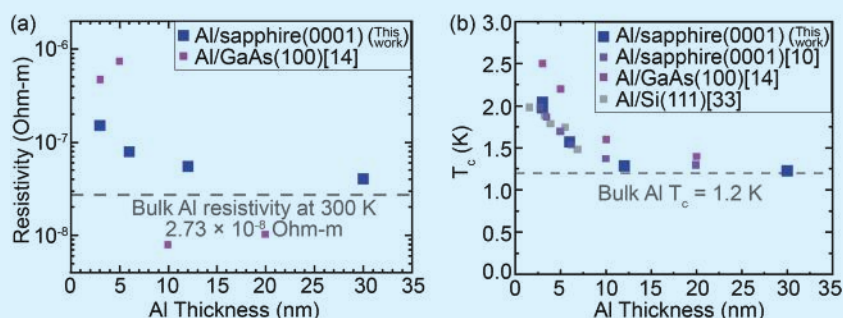


Fig. 3: (a) Resistivity vs. Al film thickness and (b) superconducting critical temperature vs. thickness of Al films. [Reproduced from Ref. 1]

Figure 2 exhibits the excellent crystal growth capability of MBE and visually confirms the success of the *in situ* Al₂O₃ capping layer technique in maintaining the pristine, well-ordered structure of ultrathin Al films. These results are prerequisites for the development of reliable, low-dielectric-loss superconducting devices in quantum computing, demonstrating that the perfected growth process can produce the high-quality, stable interfaces required for effective superconducting quantum circuits.

To understand the potential of these ultrathin Al films in superconducting quantum circuits, we illustrate in **Fig. 3** the electrical properties of the nanometer-scale Al films, particularly their resistivity and superconducting critical temperature (T_c), as a function of film thickness. This study successfully demonstrated superconductivity in these nanometer-thick Al films, with T_c varying with the film thickness. T_c ranged from 1.2 K for thicker films up to approximately 2 K for the thinnest films. This variation in T_c with thickness is likely due to quantum confinement effects or modifications in phonon modes, which alter the superconducting properties as the dimensions of the film decrease. These results suggest that these ultrathin Al films could be valuable for applications requiring superconducting materials at scaled dimensions as they maintain superconductivity even at nanometer-scale thicknesses.

In summary, this study provided valuable insights into the growth and superconducting properties of ultrathin epitaxial Al films for quantum computing applications. By employing precise MBE growth techniques and implementation of an *in situ* Al₂O₃ capping layer, Hong and his collaborators successfully demonstrated the potential of these ultrathin films to meet the serious demands of superconducting qubits, such as high crystallinity, abrupt interfaces, and stable superconducting properties at low temperatures. This paper highlights a new direction for fabricating Josephson junctions and microwave resonators, the key components in superconducting qubits for quantum computing. (Reported by Bi-Hsuan Lin)

This report features the work of Minghwei Hong and his collaborators published in J. Appl. Phys. 136, 074401 (2024).

TLS 13A1 X-ray Scattering

TLS 17B1 X-ray Scattering

- XRD, XRR
- Materials Science, Surface, Interface, Superconducting Quantum Circuits

Reference

1. Y. H. G. Lin, C. K. Cheng, L. B. Young, L. S. Chiang, W. S. Chen, K. H. Lai, S. P. Chiu, C. T. Wu, C. T. Liang, J. J. Lin, C. H. Hsu, Y. H. Lin, J. Kwo, M. Hong, *J. Appl. Phys.* **136**, 074401 (2024).

Chemical Science

The chemical science section of this year's NSRRC Activity Report highlights groundbreaking research leveraging synchrotron-based techniques. Studies span diverse fields, including organic photovoltaics, near-infrared (NIR) optoelectronics, photoresponsive materials, catalytic ammonia synthesis, and astrochemistry. These investigations underscore the critical role of synchrotron radiation in elucidating atomic and molecular phenomena, driving scientific and technological advancements.

A pivotal study on nonfullerene acceptors for organic photovoltaics explores the structure–property–performance relationships of a novel acceptor, CB16. By removing the central thiadiazole unit, researchers achieved an 18.32% power conversion efficiency, surpassing the Y6-based counterparts. Small/wide-angle X-ray scattering provided insights into molecular packing and interactions, setting new benchmarks for simplified acceptor designs to enhance the solar cell efficiency.

In NIR optoelectronics, two studies push organic light-emitting diode (OLED) emission wavelengths beyond 900 nm. One exploits an interfacial energy transfer mechanism to achieve hyperfluorescence at 925 nm and 1022 nm using Pt(II) complex and fluorescent dye bilayers, with implications for telecommunications and bioimaging. The other study investigates chromium-doped materials, revealing that luminescence quenching in $\text{GaInO}_3:\text{Cr}^{3+}$ arises from hole-type thermal quenching rather than electron transfer—a key insight for improving NIR-emitting materials for sensors and LEDs.

A breakthrough in light-responsive materials features MXeneGel, a composite hydrogel integrating MXene nanosheets and azobenzene-based supramolecular complexes. Exhibiting reversible phase transitions under UV and visible light without compromising conductivity, this innovation paves the way for reconfigurable soft electronics. Synchrotron techniques were instrumental in probing the gel's structural transformations.

Catalysis remains a focal point, with a study on ammonia synthesis using carbon-supported ruthenium catalysts. Researchers optimized mesoporous carbon supports to enhance catalytic performance and stability, aligning with renewable-energy initiatives. Synchrotron characterizations of Ru nanoparticle dispersion and electronic promoter interactions revealed the mechanisms mitigating hydrogen poisoning, offering scalable solutions for efficient ammonia production.

Astrochemical research explores molecular ices and doped graphene. One study examines the behavior of 1-propanol ice under interstellar conditions, showing its persistence beyond the melting point without crystallization—key for understanding cosmic dust mantles. Another investigates ethanolamine ice, providing insights into its formation pathways and spectroscopic properties, enriching knowledge of prebiotic chemistry in star-forming regions. A final study on N-doped graphene correlates its photoluminescence properties with astrophysical observations of the Red Rectangle Nebula. Synchrotron radiation played a crucial role in characterizing bonding environments and electronic structures.

Collectively, these studies demonstrate the transformative impact of synchrotron-based research. From renewable energy advancements to unraveling cosmic mysteries, NSRRC continues to foster collaborations that push the boundaries of chemical sciences, contributing to sustainable and innovative solutions. (By Yu-Jong Wu)

Insights into Molecular Packing

Controlling π - π stacking in non-fullerene acceptors could be key to enhancing organic photovoltaic performance.

Organic photovoltaics (OPVs) have emerged as a promising renewable energy technology due to their lightweight, flexible, and scalable nature, making them ideal for next-generation solar energy solutions. A key factor driving the advancement of OPVs is the development of non-fullerene acceptors (NFAs), which have surpassed fullerene-based acceptors in optical absorption, tunability, and efficiency.¹ Among these, Y6-based NFAs stand out as benchmarks, enabling power conversion efficiencies (PCEs) exceeding 18% through their unique A-DA² D-A-type molecular architecture.² This structure, featuring a central thiadiazole (Tz) unit and a C-shaped ortho-benzodipyrrole skeleton, supports strong absorption and efficient charge transport. Despite these successes, challenges such as the synthetic complexity of Y6 and its tendency toward aggregation hinder further improvements and limit scalability.

To address these challenges, teams led by Yen-Ju Cheng (National Yang Ming Chiao Tung University) and U-Ser Jeng (NSRRC) are exploring innovative molecular designs aimed at maintaining high performance while minimizing structural complexity and aggregation. This research focused on three carefully designed NFAs—CB16, Y6-16, and SB16—to better understand the relationships between molecular structure, packing behavior, and device performance. As shown in Fig. 1, CB16 simplifies the Y6 architecture by removing the central Tz unit while preserving the C-shaped ortho-benzodipyrrole skeleton.

This modification is intended to reduce self-aggregation and improve donor-acceptor interactions. Y6-16, a derivative of Y6 with side chains identical to those of CB16, retains the Tz unit and serves as a benchmark for comparison. Additionally, SB16 features an S-shaped para-benzodipyrrole skeleton, offering a direct comparison in molecular geometry. These three NFAs were strategically selected to elucidate how structural modifications influence molecular packing, charge transport, and overall device performance.

Grazing-incidence wide-angle X-ray scattering (GIWAXS) and simultaneous small- and wide-angle X-ray scattering (SWAXS) provided detailed insights into the molecular packing and phase behavior of the NFAs in both neat films and when blended with the donor polymer PM6. Figure 2 highlights the GIWAXS patterns and corresponding 1D scattering profiles, revealing the key differences in molecular packing among the three NFAs. CB16 exhibits vertically oriented π - π stacking with abundant small nanodomains, facilitating the formation of bicontinuous networks essential for efficient charge transport. This packing arrangement reflects the benefits of removing the Tz unit, which results in reduced aggregation while maintaining robust donor-acceptor interactions. By contrast, SB16 demonstrates large, phase-separated domains with poor π - π stacking due to its S-shaped geometry, resulting in suboptimal performance. Y6-16, while similar to CB16 in packing features, shows

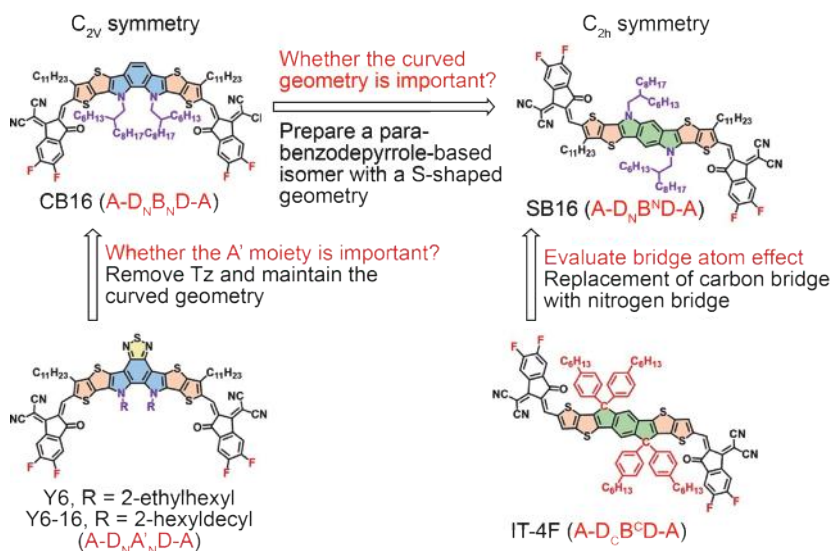


Fig. 1: Chemical structures of Y6, Y6-16, and IT-4F that inspired the design of CB16 and SB16 NFAs. [Reproduced from Ref. 3]

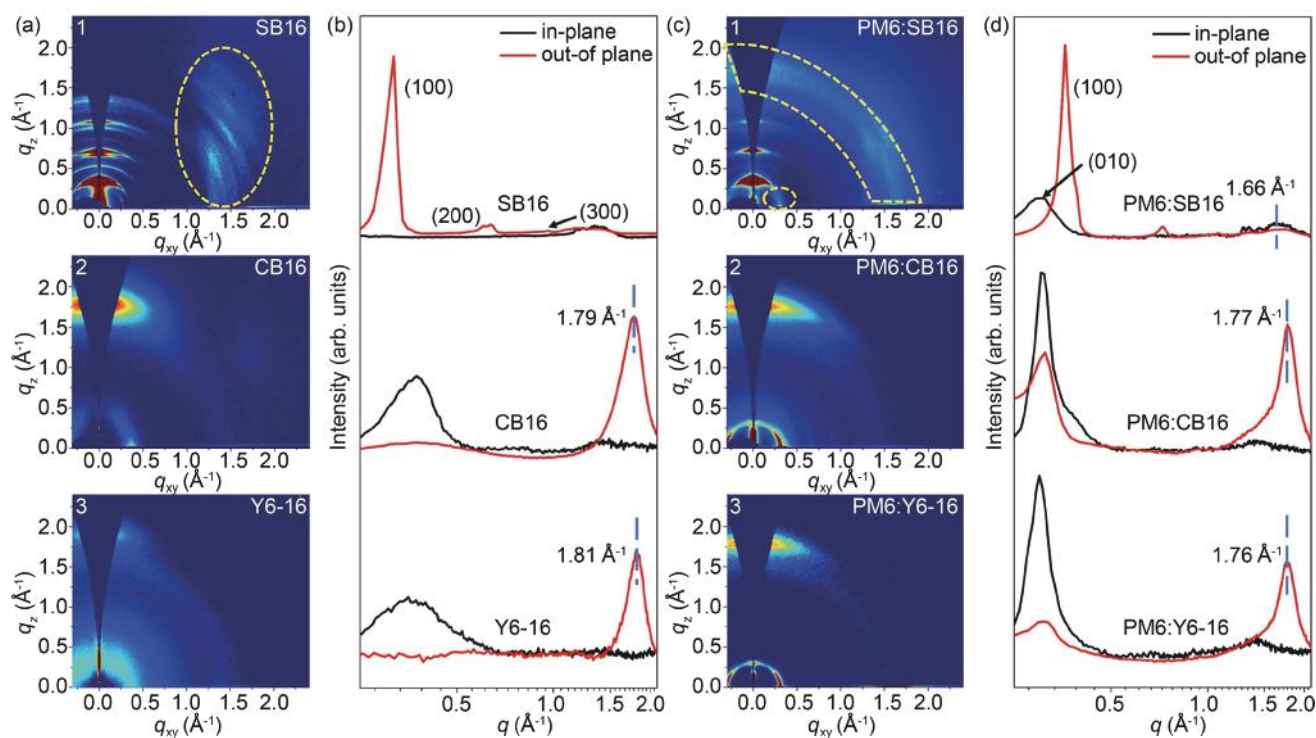


Fig. 2 2D GIWAXS patterns of the SB16 (a-1), CB16 (a-2), and Y6-16 (a-3) and their blended films PM6:SB16 (c-1), PM6:CB16 (c-2), and PM6:Y6-16 (c-3) and their corresponding 1D scattering profiles along the in-plane and out-of-plane directions for the neat films (b) and blended films (d), respectively. [Reproduced from Ref. 3]

slightly reduced donor–acceptor interactions due to the presence of the Tz unit. In device studies, CB16, when blended with PM6, achieved a remarkable PCE of 18.32% in binary OPV devices, surpassing both those of Y6-16 and SB16. The enhanced performance of CB16 is attributed to its optimized molecular packing, reduced aggregation, and efficient charge transport properties. By removing the Tz unit, CB16 not only simplifies molecular design but also balances structural simplicity with high performance, making it a promising candidate for scalable OPV applications. The contrast in performance between CB16 and SB16 underscores the critical role of molecular geometry, with the C-shaped architecture of CB16 and Y6-16 offering significant advantages over the S-shaped design.

In summary, this study provides insights into the structure–property–performance relationships of NFAs. The C-shaped A-DNBND-A skeleton in CB16 plays a crucial role in promoting efficient π – π stacking, reducing aggregation, and enhancing donor–acceptor interactions. The removal of the Tz unit simplifies synthesis while improving phase separation and charge transport, demonstrating the potential for designing high-performance NFAs with reduced complexity. Furthermore, advanced synchrotron-based characterization techniques such as GIWAXS and SWAXS at **TLS 23A1** proved invaluable for revealing the molecular packing behaviors and guiding rational

molecular design. By leveraging rational sample design and state-of-the-art characterization methods, this study not only advances the understanding of NFAs but also paves the way for future innovations in OPVs. The findings emphasize the importance of integrating molecular design, structural analysis, and device optimization to overcome limitations in existing NFAs. (Reported by Hao Ming Chen, National Taiwan University)

This report features the work of Yen-Ju Cheng and U-Ser Jeng published in J. Am. Chem. Soc. 146, 833 (2024).

TLS 23A1 Small/Wide Angle X-ray Scattering

- GIWAXS, GISAXS
- Materials Science, Thin-film Chemistry

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Advancing the Frontiers of Near-Infrared Optoelectronics

Innovative approaches advance NIR emission technologies by improving their quantum efficiency, energy transfer, and thermal stability.

The field of near-infrared (NIR) optoelectronics has emerged as a critical area of innovation driven by the increasing demand for high-performance materials and devices across diverse applications, from bioimaging and sensing to advanced communication systems. Researchers have actively striven to overcome fundamental challenges such as low quantum efficiencies, energy loss, and thermal instability. The development of novel materials and device architectures has emerged as a cornerstone of progress in this domain. This highlight presents two pioneering studies that exemplify the synergy of materials sciences, photophysics, and engineering in advancing NIR technologies.

Traditional organic light-emitting diodes (OLEDs) in the NIR range have faced challenges related to low external quantum efficiencies (EQEs). This issue is caused by the emission energy gap law, which results in significant nonradiative losses as emission wavelengths extend into the infrared spectrum. This limitation often leads to inefficiencies that hinder the practical implementation of NIR OLEDs in fields such as bioimaging, data communication, and advanced sensing. To overcome these barriers, a collaborative research group led by Yun Chi (City University of Hong Kong, China) and Pi-Tai Chou (National Taiwan University) has introduced an innovative bilayer device architecture incorporating Pt(II) complexes and fluorescent dyes such as BTP-eC9.¹ The components of the architecture are engineered to form a synergistic donor–acceptor system. By leveraging interfacial energy-transfer mechanisms, this architecture achieves hyperfluorescence with peak emissions at 925 nm and EQEs of 2.24%, setting a new benchmark in the field. Additionally, the integration of a transfer printing method preserves the integrity of delicate molecular assemblies, ensuring efficient energy transfer and device stability. A grazing-incidence wide-angle X-ray scattering (GIWAXS) analysis performed at **TLS 13A1** revealed that the Pt(II) complexes in the OLED architecture exhibited highly ordered edge-on π – π stacking, which is crucial for efficient energy transfer. The BTP-eC9 fluorescent dye displayed lamellar-type face-on π – π stacking, which is also important for its function as an energy acceptor and emitter, facilitating the interfacial energy transfer mechanisms. Thus, the Pt(II) complexes act as highly efficient energy donors, transferring triplet-state energy to the singlet-state acceptors (BTP-eC9) *via* a Förster resonance energy transfer (FRET) mechanism, as demonstrated in **Fig. 1**. This process bypasses nonradiative loss channels, enhancing fluorescence intensity. A bilayer architecture with precise control over material interfaces minimizes back energy transfer and maintains structural order. The implementation of transfer printing addresses the challenge of assembling self-organized layers while maintaining their molecular arrangement, demonstrating the potential for large-scale manufacturing. This research not

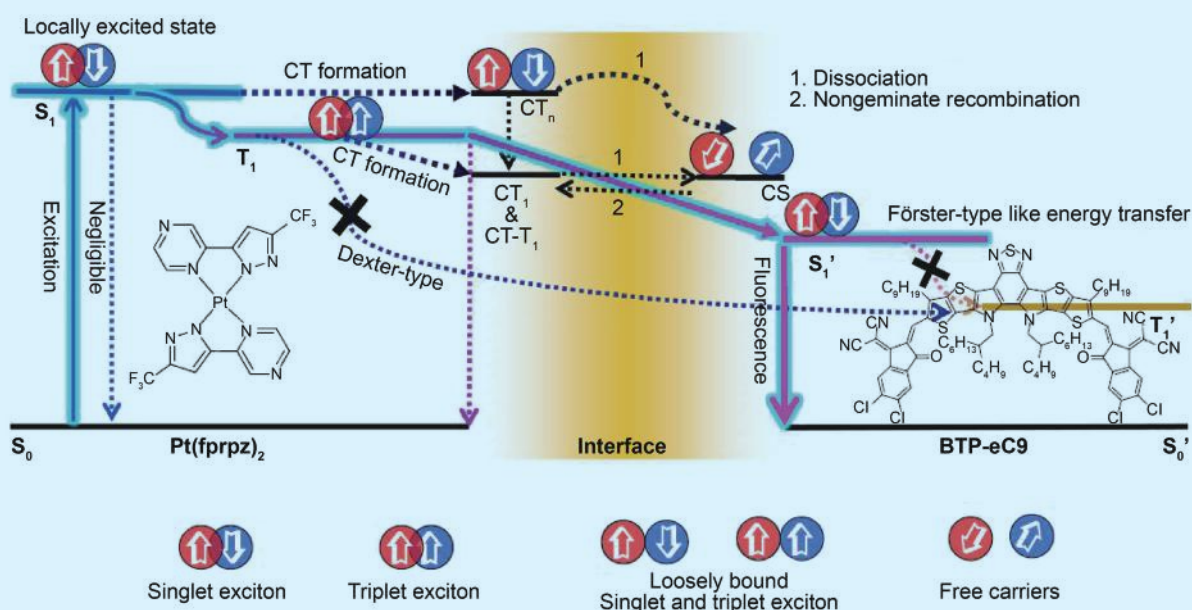


Fig. 1: An overview of the interfacial energy transfer dynamics that underpin NIR OLED functionality: the solid sky-blue pathway illustrates the interfacial energy-transfer process, which facilitates FRET. In this context, the S₀ and S₁ states represent the ground and excited states, respectively, of the singlet manifold, and the T₁ state denotes the triplet state. [Reproduced from Ref. 1]

only demonstrates a leap in device performance but also establishes a robust framework for tackling the inherent limitations imposed by the emission energy gap law.

The second significant research contribution comes from Sebastian Mahlik of the University of Gdansk, Poland, focusing on inorganic materials, specifically, Ga–In oxides doped with Cr^{3+} ions.² This study investigates the dual-purpose functionality of these materials as NIR phosphors for light-emitting diodes (LEDs) and ultraviolet photodetectors. The research examines the intricate interplay between radiative and nonradiative processes, emphasizing luminescence-quenching mechanisms. Cr^{3+} -activated materials have attracted significant attention for their ability to emit broadband NIR light, positioning them as ideal candidates for phosphor-converted LEDs. However, traditional interpretations of luminescence quenching often fail to explain the unique thermal behaviors observed in these systems. This study presents a novel perspective by attributing luminescence quenching to hole-based thermal ionization rather than electron transfer to the conduction band—a paradigm shift in understanding transition-metal-doped luminescent materials. High-resolution synchrotron X-ray diffraction (XRD) data, obtained at **TPS 19A**, revealed that Ga–In oxide samples predominantly exhibit a monoclinic $\beta\text{-Ga}_2\text{O}_3$ structure. Increasing indium (In^{3+}) doping levels led to the coexistence of two phases: the monoclinic $\beta\text{-Ga}_2\text{O}_3$ phase and the cubic In_2O_3 phase. This phase separation underscores the limited solubility of In^{3+} in the Ga_2O_3 host lattice and the structural changes induced by In^{3+} substitution. Greater indium concentrations introduced more disorder into the crystal lattice, as evidenced by the broadening of peaks in diffraction patterns and the appearance of mixed phases at higher doping levels. This disorder affected the local crystal field around Cr^{3+} dopants, significantly influencing their optical and photoelectric properties.

The study presents a groundbreaking hole-based thermal quenching mechanism, which challenges the conventional focus on electron transfer. This mechanism accounts for the material's luminescence behavior at elevated temperatures. The $\text{Ga}_{1.98-x}\text{In}_x\text{O}_3:0.02\text{Cr}^{3+}$ system exhibits tunable NIR emission and efficient photocurrent generation, rendering it suitable for both light emission and detection applications. By combining high-resolution synchrotron XRD with photoluminescence analysis, the research links In-induced structural modifications to enhanced optical performance. The substitution of In^{3+} ions alters the crystal field environment, optimizing energy level alignments for NIR emission and expanding the material's functionality in optoelectronic applications.

Together, these studies provide a comprehensive exploration of the challenges and solutions shaping the future of NIR optoelectronics. The meticulous research on OLEDs emphasizes the importance of molecular precision and device architecture, whereas the insights into Cr^{3+} -activated materials highlight the interplay between structure, energy dynamics, and multifunctionality. By connecting organic and inorganic systems, this compilation highlights the shared principles that underpin success in the field: innovation in material design, rigorous characterization, and a vision for translational impact. (Reported by Yu-Jong Wu)

This report features the work of Yun Chi, Pi-Tai Chou and their collaborators published in Nat. Commun. 15, 4664 (2024), and the work of Sebastian Mahlik and his collaborators published in JACS 146, 22807 (2024).

TPS 19A High-resolution Powder X-ray Diffraction

TLS 13A1 X-ray Scattering

- GIWAXS, XRD
- Materials Science, Inorganic Chemistry, Solid-state Chemistry, Photoluminescence

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Light-Activated Gel: Paving the Way for Smarter Electronics

A novel light-responsive gel combining MXene nanosheets with supramolecular complexes was investigated, showcasing reversible phase transitions and enhanced conductivity.

MXenes are a rapidly emerging class of two-dimensional (2D) materials that have garnered significant attention in materials science due to their unique properties and broad applications. First discovered in 2011, MXenes are synthesized by selectively etching out elements from layered transition metal carbides, nitrides, or carbonitrides. MXenes exhibit a unique combination of metallic conductivity, excellent mechanical flexibility, high surface area, and tunable surface chemistry. These attributes make them highly promising for various applications, including energy storage, electromagnetic interference, water purification, and catalysis. The research team led by Jiun-Tai Chen from National Yang Ming Chiao Tung University explored conductive composite gels with multifunctional capabilities. By incorporating inorganic dopants into an organic matrix, the gel's conductivity can be significantly improved while introducing new properties such

as thermal responsiveness, light responsiveness, and self-healing abilities.

The research team studied a light-responsive MXene-based composite gel, termed MXenegel, which integrates azobenzene-containing supramolecular complexes with MXene nanosheets (Fig. 1).¹ This innovative material exhibits reversible photo-modulated phase behavior, transitioning between liquid and solid states under UV and visible light, respectively, while maintaining its electrical conductivity, making it suitable for traditional solid-state electronics. The motivation behind this research stems from the need for intelligent and eco-friendly electronic components that can adapt to environmental changes and promote sustainability. Traditional solid-state materials often lack compatibility with dynamic substrates, such as human skin, and do not respond to environmental stimuli. The MXenegel addresses these challenges

by providing a new charge transport pathway that can sense environmental changes and be reprogrammed or recycled.

Multiple instruments were used in this work, providing spectroscopical and electronic evidence to support the unique behavior of MXenegel. The MXenegel was shown to undergo a gel-to-sol transition upon UV light irradiation, which is attributed to the disassembly of the AzoC6@2 α CD inclusions intercalated between the MXene layers. This phase transition is reversible, as the sol-state MXenegel can be converted back to a gel-like structure under visible light irradiation or by keeping it in a dark room at room temperature. Additionally, the X-ray diffraction spectra performed at TLS 23A1 of the NSRRC played an important role in this research. The small-angle X-ray scattering (SAXS) spectra revealed the microstructural details of MXenegel, confirming the successful formation of the AzoC6@2 α CD inclusion complexes and their intercalation between MXene layers (Fig. 2).

In summary, this work shows a light-modulated MXenegel with reversible phase transition based on photoresponsive host-guest chemistry. Detailed NMR, 2D-ROESY, XPS, and X-ray scattering analyses obtained from synchrotron facilities revealed the microstructure of the MXenegel. The potential applications of MXenegel in electronic circuitry are diverse and promising. The material could serve as a light-responsive wire in electronic circuits, enabling its integration into solid-state electronics

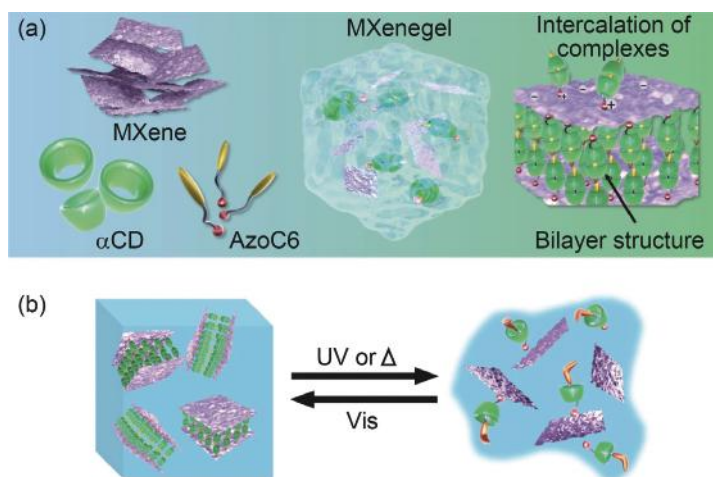


Fig. 1: Schematic illustration of the light-responsive MXenegel. (a) α CD and AzoC6 form a bilayer structure of AzoC6@2 α CD supramolecular complexes in MXenes. The positive head ends of the complexes are electrostatically attached to the negatively charged MXene surfaces. (b) Light-responsive sol-gel transition behavior of the MXenegel. [Reproduced from Ref. 1]

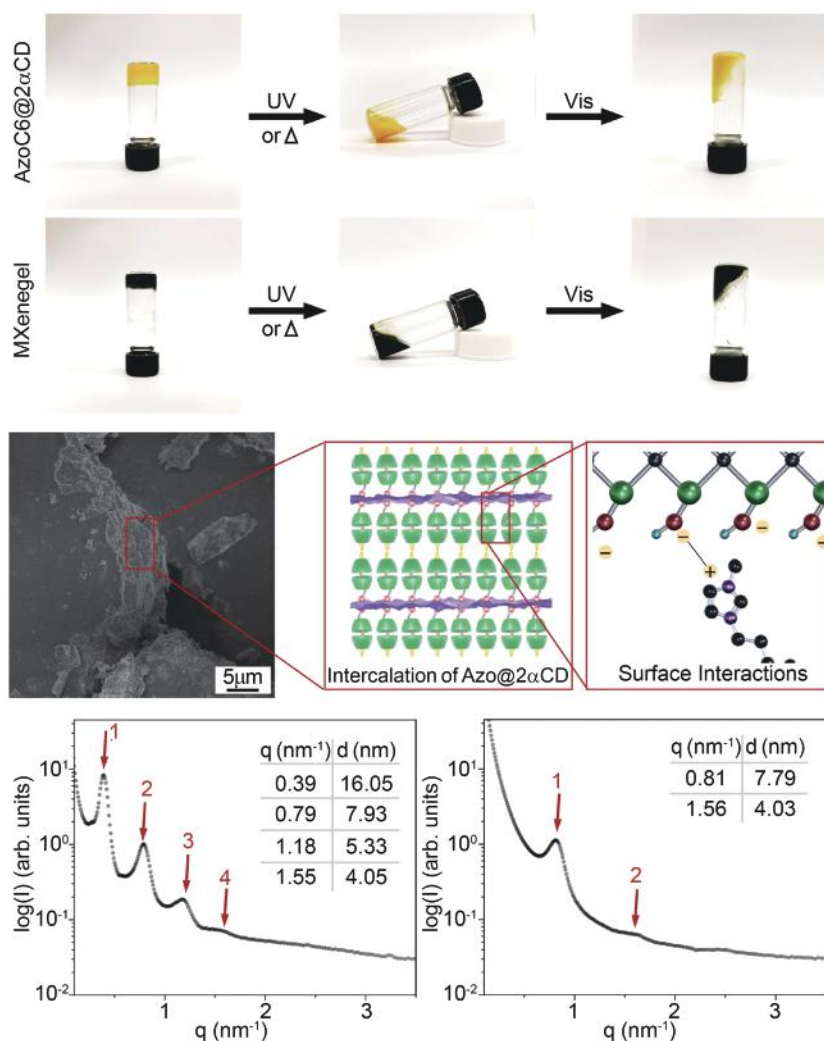


Fig. 2 Light-responsive phase behavior of AzoC6@2 α CD and 30 wt% MXene/gel under different light irradiations. SEM images of the vacuum-dried MXene/gel, in which a cross-sectional view of the MXene/gel layers is displayed. The maxima are indicated in the SAXS spectra of AzoC6@2 α CD hydrogel and MXene/gel. [Reproduced from Ref. 1]

as conductive wires or switches. This material demonstrates a reversible photo-modulated phase behavior, allowing it to function as a photo-controllable switch, which can be utilized to control devices like LEDs. Additionally, MXene/gel can be used as a writable and reconfigurable conductive ink, making it suitable for brush printing on various substrates, thus expanding its application in flexible and eco-friendly electronics. (Reported by Yu-Liang Lin, National Yang Ming Chiao Tung University)

This report features the work of Jiun-Tai Chen and his collaborators published in Nat. Commun. 15, 916 (2024).

TLS 23A1 Small/Wide Angle X-ray Scattering

- Supramolecular Complex Scattering
- Materials Science, Chemistry, Surface, Thin-film Technology, Chemical Kinetics

Reference

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Versatile Roles of Cesium

Cs-promoted Ru catalysts play a key role in enhancing efficient ammonia synthesis.

Ammonia synthesis is a cornerstone of the global chemical industry as it is essential for fertilizer production and is increasingly recognized as a potential hydrogen carrier for renewable energy applications.¹ However, the conventional Haber–Bosch (HB) process requires high temperatures and pressures, leading to substantial carbon emissions and energy consumption.² In the pursuit of more sustainable and energy-efficient catalytic systems, research teams led by Shih-Yuan Chen (National Institute of Advanced Industrial Science and Technology, Japan), Hsin-Yi Tiffany Chen (National Tsing Hua University), Ho-Hsiu Chou (National Tsing Hua University), and Chia-Min Yang (National Tsing Hua University) investigate the impact of carbon support graphitization on the activity and stability of cesium (Cs)-promoted ruthenium (Ru) catalysts for ammonia synthesis. By systematically tuning the graphitization degree of mesoporous carbon plates (MCPs), this study provides fundamental insights into the relationship between carbon support structure, Ru dispersion, and catalytic performance.

The research introduces a series of Ru catalysts supported on MCPs with varying degrees of graphitization, synthesized through controlled carbonization. MCP-1100, which possesses the highest degree of graphitization, emerges as the optimal support, offering superior thermal stability, enhanced electron conductivity, and minimized carbon methanation. **Figure 1(a)** illustrates that higher carbonization temperatures lead to increased graphitic ordering, further validated by high-angle annular dark-field scanning transmission electron microscopy images. These images reveal well-dispersed Ru nanoparticles on MCP-1100 (**Fig. 1(b)**). By contrast, catalysts supported on lower-graphitization-degree MCPs exhibit Ru particle agglomeration, which negatively impacts catalytic efficiency. The catalytic performance evaluation, presented in **Fig. 1(c)**, demonstrates that the Cs-promoted Ru/MCP-1100 catalyst achieves an ammonia synthesis rate of $43 \text{ mmol NH}_3 \cdot \text{g}^{-1} \cdot \text{h}^{-1}$ at 410°C under ambient pressure, significantly outperforming conventional Fe-based catalysts under the similar conditions. These results indicate a strong correlation between the degree of graphitization and ammonia synthesis rates, with higher graphitization promoting better Ru dispersion and electronic conductivity. The critical role of cesium promotion is further supported by density functional theory (DFT) calculations (**Fig. 1(d)**), which show that CsOH weakens hydrogen adsorption on Ru, facilitating ammonia formation by accelerating hydrogen desorption. This cooperative effect between Ru and Cs enhances catalytic efficiency by promoting the crucial dissociative adsorption of N_2 while preventing catalyst deactivation.

A major highlight of this study is the application of *in situ* X-ray absorption spectroscopy (XAS) at **TLS 01C1** for the Ru K-edge and **TLS 17C1** for the Cs L_3 -edge, along with near-ambient-pressure X-ray photoelectron spectroscopy (NAP-XPS) at **TLS 24A1**. These techniques provide real-time insights into the electronic and structural evolution of Ru and Cs species

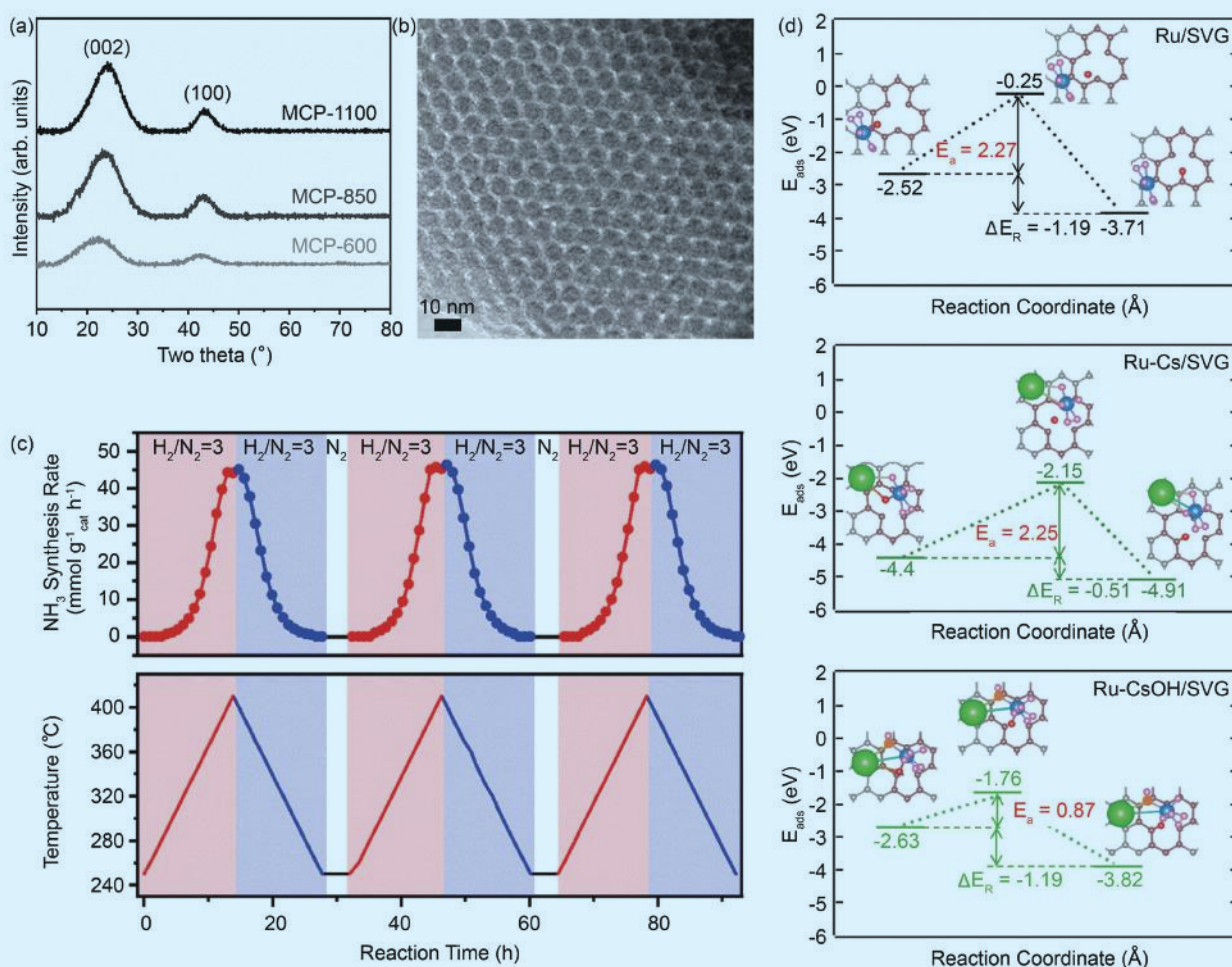


Fig. 1: (a) Wide-angle Powder X-ray Diffraction profiles of MCP-600, MCP-850, and MCP-1100. (b) TEM image of MCP-1100. (c) Temperature dependence of ammonia synthesis rate over 2.5Cs-10Ru/MCP-1100 under intermittent operating conditions. (d) Reaction coordinate diagram of hydrogen spillover from a Ru atom to single-vacancy graphene (SVG) in Ru/SVG, Ru-Cs/SVG, and Ru-CsOH/SVG models with hydrogen saturated at a Ru/H ratio of 1:6. E_a refers to the activation energy for hydrogen spillover from non-spillover models to spillover models through transition states (structures shown on the left, right, and middle in each image, respectively). ΔE_R refers to the reaction energy between non-spillover and spillover models. Blue, Ru; pink, H; red, spilled-over H; green, Cs; orange, O; gray, C atom away from the C point defect; brown, C atom close to the C point defect. [Reproduced from Ref. 3]

under various reaction conditions. **Figure 2** presents *in situ* X-ray absorption near-edge structure (XANES) and NAP-XPS spectra, which reveal a dynamic transformation of cesium species from CsOH to metallic Cs⁰ during the reaction. This transformation, facilitated by hydrogen spillover from Ru sites, enhances the electron-donating ability of Ru, reducing hydrogen poisoning and increasing N₂ activation. The XAS and NAP-XPS results confirm that Cs⁰ acts as an electronic promoter, effectively modifying the electronic structure of Ru to boost NH₃ synthesis rates.

In summary, this study establishes that Ru nanoparticle size, Cs/Ru ratio, and the degree of graphitization critically influence NH₃ synthesis rates and resistance to carbon methanation. By leveraging *in situ* XAS, NAP-XPS, kinetic analyses, and theoretical modeling, this work provides a real-time mechanistic understanding of how Cr species dynamically enhance Ru's catalytic performance. The findings establish a new design paradigm for next-generation ammonia synthesis catalysts, offering a scalable and energy-efficient alternative to conventional HB catalysts while mitigating carbon emissions. These insights contribute to the ongoing transition toward sustainable and low-carbon chemical manufacturing. (Reported by Hao Ming Chen, National Taiwan University)

This report features the work of Chia-Min Yang, Ho-Hsiu Chou, Hsin-Yi Tiffany Chen and Shih-Yuan Chen published in *Appl. Catal. B-Environ.* **346**, 123725 (2024).

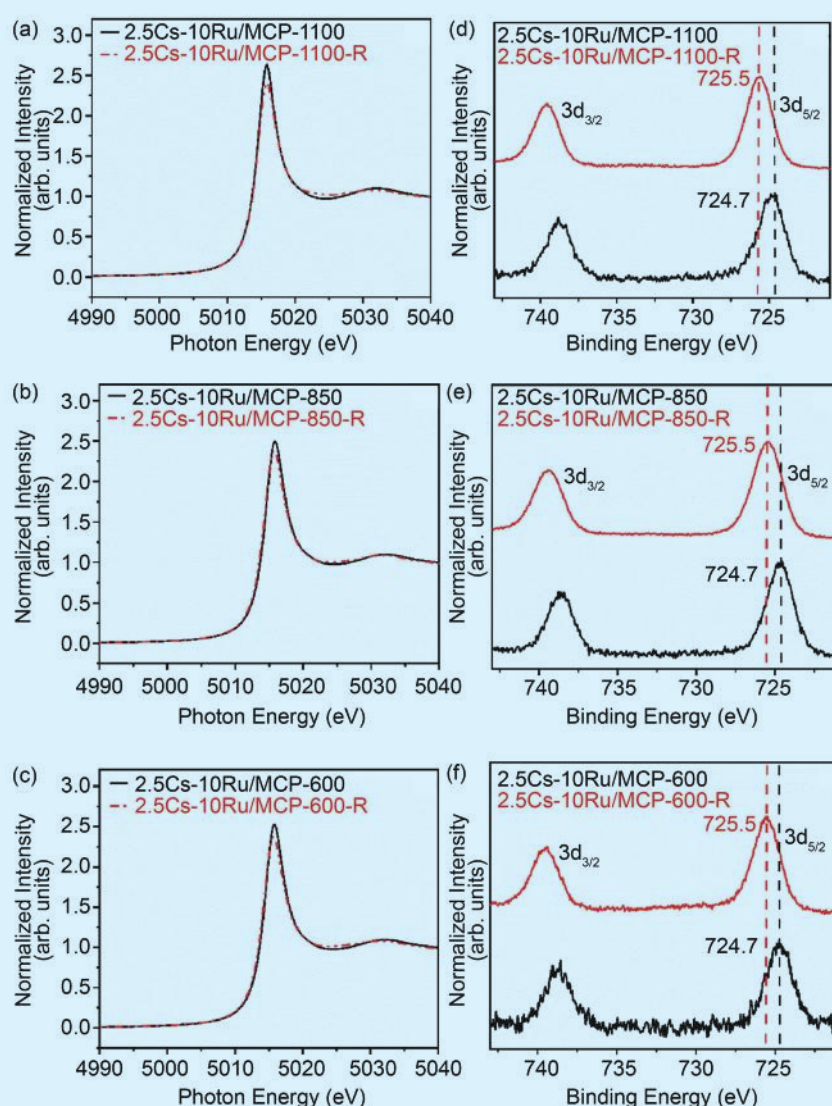


Fig. 2: Results of *in situ* XAS and NAP-XPS measurements. (a–c) XANES spectra at the Cs L₃-edge, and (d–f) NAP-XPS profiles of the Cs 3d region. [Reproduced from Ref. 3]

TLS 01C1 EXAFS

TLS 17C1 EXAFS

TLS 24A1 XPS, UPS, XAS, APXPS

- *In situ* XAS, NAP-XPS
- Materials Science, Ammonia Synthesis

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Illuminating the Cosmic Laboratory with Vacuum Ultraviolet Light

Exploring the origin and evolution of interstellar and circumstellar molecules enhances our understanding of the Universe.

In the vast expanse of the cosmos, scientists are continuously uncovering secrets that bridge the gap between stars and the building blocks of life. Among these pursuits, three recent studies used the vacuum ultraviolet (VUV) beamlines of the Taiwan Light Source (TLS) to illuminate intriguing connections between interstellar chemistry, materials science, and the potential origins of life. These stories unfold like chapters in a grand cosmic narrative, each offering a glimpse into the intricate dance of molecules and light in the universe.

The first story begins with ethanolamine, a simple molecule with profound implications. Known as a precursor to amino acids, ethanolamine has the potential to unveil how the building blocks of life might have formed in the harsh environments of space. A joint research group led by Bhalamurugan Sivaraman from Physical Research Laboratory of India, delved into its mysteries by recreating astrochemical conditions in the laboratory. Using advanced spectroscopy, they probed the spectral fingerprints of this molecule, capturing the behavior of ethanolamine ice as it is warmed from frigid interstellar temperatures.¹ Infrared and VUV spectroscopy provided critical insights, revealing how the molecule interacts with ultraviolet photons and sublimates as it transitions to higher temperatures. The end station connected to **TLS 03A1** employed ultrahigh vacuum chambers to mimic interstellar conditions, with ethanolamine deposited on cryogenically cooled substrates. These setups allowed for precise control of temperature and environment, ensuring that the behavior of the molecule could be observed without interference. Coupled with computational models, this work pieced together plausible pathways for the molecule's formation on cosmic dust grains, providing a roadmap for future discoveries of prebiotic chemistry in the cosmos.

Next, their focus shifted to 1-propanol, a fatty alcohol that holds the potential to shed light on the origins of proto-cell membranes. The tale of 1-propanol is one of resilience, as experiments revealed its unusual stability in the icy realms of the interstellar medium. Despite warming beyond its melting point, this molecule defied expectations, remaining amorphous and adhering to simulated dust grain surfaces.² Mid-infrared spectroscopy was again employed to trace the molecular vibrations of 1-propanol across a range of temperatures, while VUV spectroscopy captured its absorption characteristics in the 115–220 nm range, as shown in **Fig. 1**. Using a cryostat system with

LiF windows and controlled heating rates, researchers meticulously documented the phase transitions and sublimation behavior of 1-propanol. The VUV spectra of 1-propanol were recorded well beyond its melting point of 147 K, demonstrating that the solid-state sample remained amorphous throughout the warming process, from 10 K to 175 K, until sublimation. This represents the first observation of a molecule persisting on a cold substrate beyond its melting point in a UHV chamber. In contrast, 2-propanol, a positional isomer of 1-propanol, exhibits entirely different behavior: it crystallizes at approximately 120 K and sublimates at 150–155 K, prior to reaching its melting point. Mid-infrared spectroscopy was again employed to trace the molecular vibrations of 1-propanol across a range of temperatures, while vacuum ultraviolet spectroscopy captured its absorption characteristics in the 115–220 nm range. Complementing these experiments, molecular dynamics simulations unravelled the microscopic interactions between the alcohol molecules, offering a new perspective on the complexity of icy mantles in space. This surprising discovery challenges conventional ideas about the phase behavior of interstellar ices and expands our understanding of the molecular diversity in space.

Meanwhile, a different kind of light emerged from the depths of the Red Rectangle Nebula—a mysterious blue luminescence (BL). This glow had puzzled scientists for decades, with its origins tied to the enigmatic interplay of polycyclic aromatic hydrocarbons. BL is characterized by

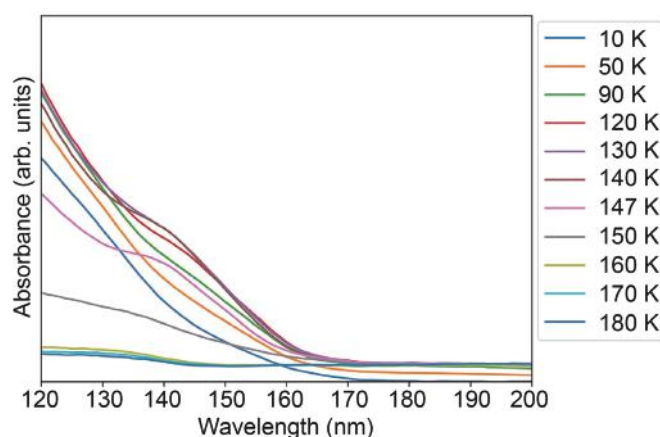


Fig. 1: VUV spectra of 1-propanol ice recorded after deposition at 10 K. The deposited ice was then warmed to higher temperatures and spectra recorded at specific temperatures until sublimation. [Reproduced from Ref. 2]

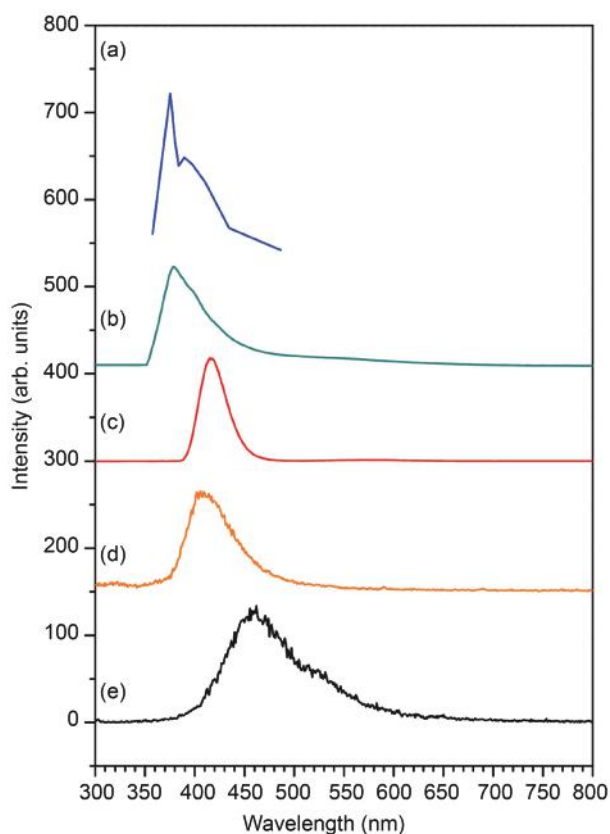


Fig. 2: (a) Blue luminescence recorded in the Red Rectangle Nebula,⁴ and PL spectra measured at 10 K upon excitation with 121.6 nm for (b) photochemically N₂-covered graphene, (c) photochemically O₂-covered graphene, (d) commercial N-doped graphene flakes, and (e) commercial graphene oxide flakes. [Reproduced from Ref. 3]

an asymmetrical spectral band peaking around 375–378 nm in the ultraviolet and visible regions. Its discovery in astrophysical environments such as the Red Rectangle Nebula links it to aromatic compounds and quantum effects in interstellar dust. In a breakthrough study,³ Y.-J. Wu (NSRRC) and his collaborators turned to graphene, a material as versatile as it is remarkable. By doping graphene with nitrogen atoms and exposing it to VUV light, they recreated conditions that echoed the astrophysical environments where BL thrives. The experimental setup involved single-layer graphene films, covered by a few layers of N₂ or O₂ solids and mounted on MgF₂ substrates, which were then cooled to 10 K and exposed to intense VUV light at TLS 21A2. Photoluminescence spectroscopy revealed a peak at 378 nm, closely matching the BL spectra recorded in the Red Rectangle Nebula, as shown in Fig. 2. Further analysis using Raman spectroscopy and X-ray photoelectron spectroscopy identified the structural defects and pyrrolic-N and pyridinic-N groups in the graphene lattice responsible for the luminescence. These defects disrupt the carbon network's symmetry, introducing localized electronic states that emit the characteristic blue light when excited. This study established N-doped graphene as a potential carrier of BL observed in astrophysical environments.

Together, these studies weave a rich tapestry of discovery, connecting laboratory experimentation with the mysteries of the universe. They remind us that the smallest molecules, whether in icy grains or luminous nebulae, hold the power to unlock profound insights into our cosmic origins. As scientists continue to explore these molecular frontiers, the stories of ethanolamine, 1-propanol, and N-doped graphene serve as beacons, guiding us toward a deeper understanding of the molecular universe and our place within it. (Reported by Yu-Jong Wu)

*This report features the work of Bhalamurugan Sivaraman and his collaborators published in *Astrophys. J.* **975**, 181 (2024) and *MNRAS* **530**, 1027 (2024), and the work of Yu-Jong Wu and his collaborators published in *Astrophys. J.* **977**, 230 (2024).*

TLS 03A1 High-flux VUV Beamline

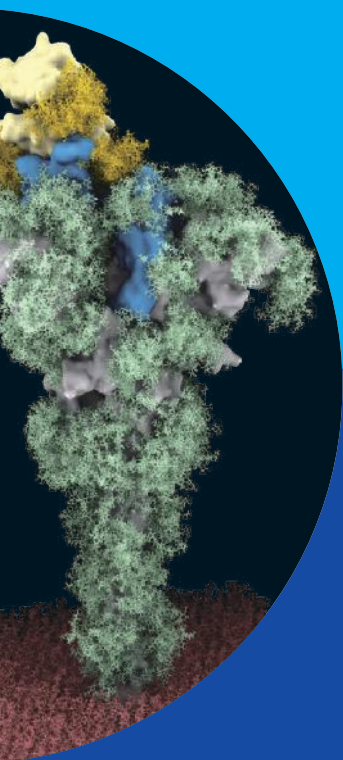
TLS 21A2 VUV Photochemistry

- Photoabsorption, Photoluminescence
- Astrochemistry, Molecular Science

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Soft Matter



Soft-matter science covers a diverse range of materials, including polymers, colloids, liquid crystals, biomacromolecules, and self-assembled systems. These materials exhibit hierarchical structures and dynamic behaviors that require advanced characterization techniques to unravel their complex properties. Synchrotron radiation provides powerful tools for probing soft matter at multiple length scales, offering high-resolution insights into their structure, interactions, and functional properties.

Several beamlines are dedicated to soft-matter research at the Taiwan Photon Source (TPS) and Taiwan Light Source (TLS). **TPS 13A** biological small-angle X-ray scattering (BioSAXS) enables structural analysis of biomacromolecules in solution. **TPS 44A** quick-scanning X-ray absorption spectroscopy provides element-specific insights into local electronic and atomic structures. **TLS 23A1** small/wide-angle X-ray scattering facilitates the characterization of hierarchical architectures in polymers, biomaterials, and nanostructured systems.

This section presents four recent studies that demonstrate the versatility of synchrotron techniques in soft-matter research. The first study investigated glycoprotein structures by using **TPS 13A** BioSAXS combined with computational modeling to characterize the conformational properties of highly glycosylated proteins. The findings offer insights into glycan-mediated interactions and their role in biological functions. The second study explored centrosome regulation during cell division, focusing on the phase separation of Cep57 and its role in microtubule nucleation. **TPS 13A** BioSAXS helped elucidate the molecular mechanisms governing centrosomal organization and its impact on cellular processes.

The third study examined the aqueous-phase crystallization of metal–organic frameworks, with **TPS 13A** *in situ* SWAXS providing real-time tracking of nucleation and growth mechanisms. The results revealed how biomolecules influence framework formation, shedding light on biomimetic strategies for sustainable material synthesis. The fourth study focused on the stabilization of single platinum atoms within silicate nanochannels for enhanced catalytic efficiency. **TPS 44A** X-ray absorption spectroscopy was used to confirm atomic coordination environments, while **TLS 23A1** and **TPS 13A** were utilized to aid analysis of the hierarchical structure and dispersion of Pt atoms.

These studies exemplify how synchrotron-based techniques enable a deeper understanding of soft-matter systems, providing essential structural and functional insights. The ability to probe nanoscale architectures with high precision continues to drive advances in biomaterials, catalysis, and fundamental science, paving the way for future discoveries and technological applications. (by Orion Shih)

Advancing Glycoprotein Insights: Bridging Glycobiology and Structural Biology

Protein glycosylation is integral in biology; however, quantitative annotation of the structure-function relationship of glycosylation is challenging. This article reports the findings of an interdisciplinary and international team formed to tackle this challenge.

The surfaces of cells and viruses are often coated with a thick layer of sugar (also known as glycans), presenting a challenge for identifying specific proteins or biomolecules within this complex environment. In nature, however, efficient biomolecular interactions take place within this forest of glycans. Most extracellular proteins are post-translationally modified by glycans with varying sizes and complexity. The masking effect of these glycans allows bacteria and viruses to evade recognition and attack by the host immune system.

Structural biology focuses on providing pivotal structural information at an atomic resolution to help to understand the mechanisms governing biological functions. Such an underlying principle is also used by the immune system, where antibodies recognize the structural characteristics of foreign substances, such as antigens, to achieve immune protection. Nevertheless, structural biology encounters two technical challenges when examining the intricate structures of protein glycans. First, glycan molecules have a complex chemical composition, and their biosynthesis lacks a one-to-one template. Second, glycans are highly dynamic, making it challenging to accurately define three-dimensional spatial distributions. Therefore, the intrinsic microscopic compositional and conformational heterogeneity presents a fundamental challenge to the accurate modeling of glycan structures.

The research team led by Shang-Te Danny Hsu (Academia Sinica) tried to solve this problem from the structural understanding of protein glycosylation started in 2018 through a collaboration with Hui-Wen Chang at the National Taiwan University and Kay-Hooi Khoo at Academia Sinica. They used cryo-electron microscopy (cryo-EM) to determine the near-atomic structure of the spike protein of a type I feline infectious peritonitis virus (FIPV).¹ They identified an exceptionally well-defined cryo-EM map enabling the modeling of one of the longest N-glycan structures in the literature. The atomic model of the fully glycosylated FIPV spike protein was built manually using the information derived from mass spectrometry (MS) analysis. Nonetheless, the data analysis and model building are laborious and not very quantitative.

To address these issues, they collaborated with Khoo to develop a robust workflow to quantitatively characterize the N-glycosylation profiles of the spike proteins of SARS-CoV-2 variants.² They also determined the cryo-EM structures of the same spike proteins, with an emphasis on recovering structural information of glycans. They further collaborated with Cyril Hanus at the Inserm, France, and Mateusz Sikora at the Max Planck Institute of Biophysics, Germany, to develop the computational tool called GlycoSHIELD³ to integrate the experimental observables with computational modeling to automate the process of model building of glycoproteins (Fig. 1). Such a procedure can be accomplished using a personal computer in minutes rather than using a high-performance computing center for full-blown all-atom molecular dynamics simulations of fully glycosylated spike proteins solvated in an explicit solvent model. Additionally, GlycoSHIELD offers predictive powers for identifying potential receptor binding sites and antigenic targets for therapeutic purposes based on the common characteristic that the less-shielded protein surfaces are generally more likely to be used for host receptor binding and targeted by neutralizing antibodies.

Small-angle X-ray scattering (SAXS) is exceptionally versatile in describing protein structures and dynamics in solution. The research team has benefited tremendously from the support of the biological SAXS TPS 13A beamline research team at the NSRRC. In the context of GlycoSHIELD, they used SAXS to characterize two highly glycosylated proteins, namely, the extracellular domains (ECDs) of N-cadherin pertinent to cell–cell adhesion and

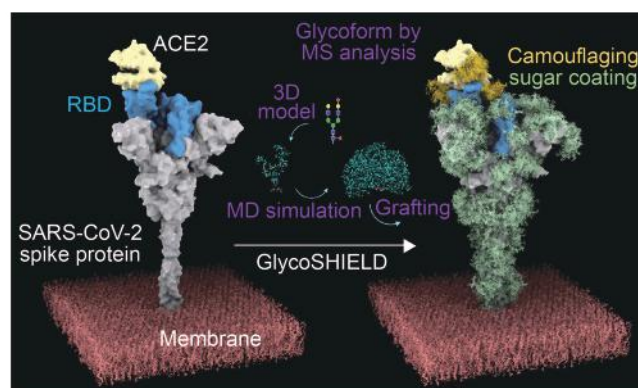


Fig. 1: Schematic overview of the principle of GlycoSHIELD.

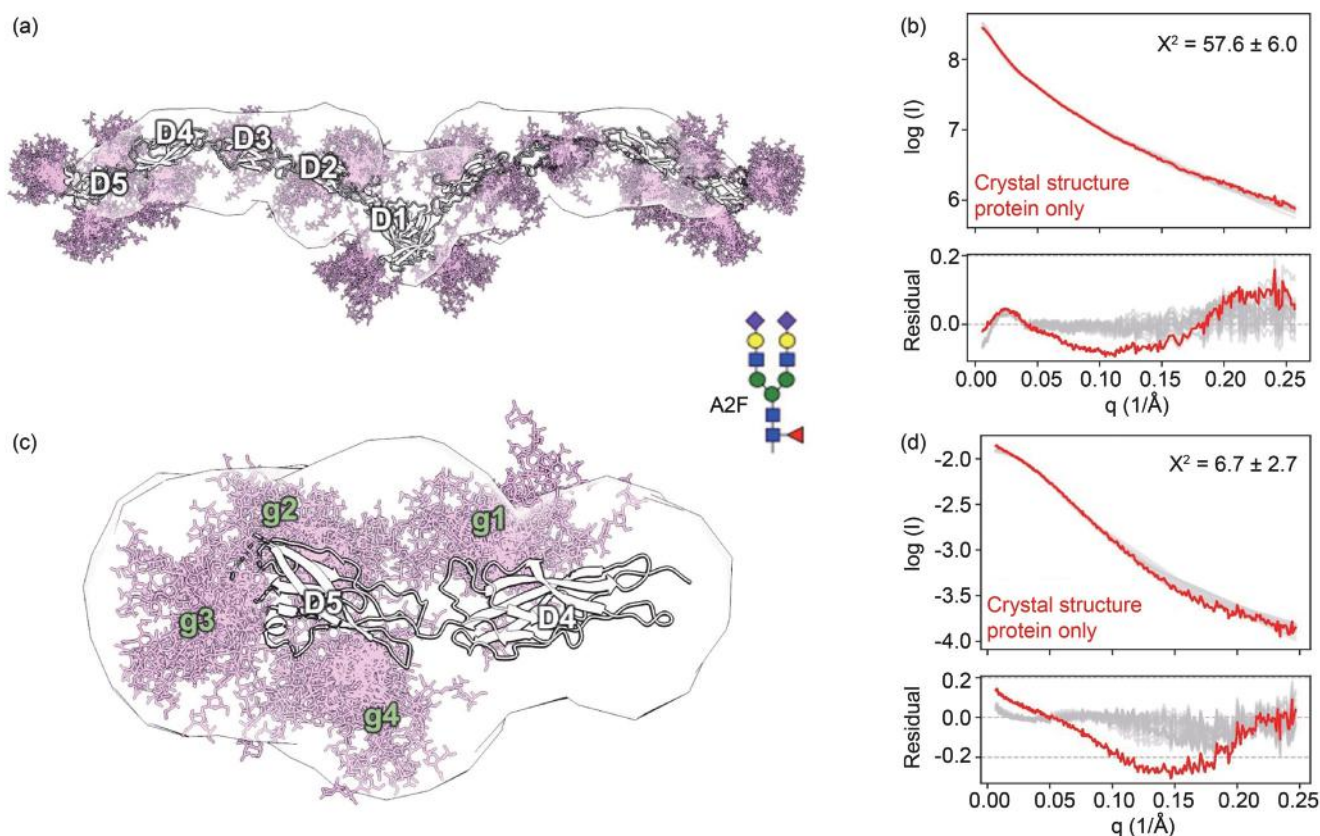


Fig. 2: SAXS analysis of N-cadherin variants. (a) The molecular envelope of N-cadherin ECD 1-5 (D1-D5) in a seagull-like homodimeric assembly. The crystal structure, indicated by a white outline, is modified with a biantennary complex type (A2F) N-glycan in its putative N-glycosylation sites, which are indicated by pink sticks. (b) The experimental SAXS profile is superimposed with the back-calculated SAXS profiles based on the crystal structure (red) and the additional N-glycan structures in 20 different conformations (gray). The residuals are shown below the SAXS profiles. Similar structural representations of the D4 and D5 of N-cadherin are shown in (c), and the corresponding SAXS data analyses are shown in (d). [Reproduced from Ref. 3]

the ECD of the receptor-like protein tyrosine phosphatase (PTPRA), which is crucial for transmitting extracellular signals to trigger intracellular chemical reactions.

N-cadherin is a calcium-binding glycoprotein comprising five folded ECDs (**Fig. 2(a)**). Molecular dynamics simulations have shown that glycosylation is important for maintaining the extension of N-cadherin. However, most of the glycans are removed to solve the crystal structure of N-cadherin, which is an intrinsic issue in protein crystallography. The team used SAXS to demonstrate that the fully glycosylated N-cadherin variants indeed adopt an extended seagull-like dimer structure, which is consistent with the crystallographic finding (**Fig. 2(a)**). By modeling different glycan ensembles onto the putative N-glycosylation sites, they could further improve the agreement between the experimental and back-calculated SAXS profiles based on the atomic ensemble structures. This illustrates how the combination of GlycoSHIELD modeling and experimental SAXS inputs can generate a more realistic glycoprotein conformational ensemble (**Figs. 2(a)–2(d)**).³

Structural analysis of highly glycosylated intrinsically disordered proteins represents an even more significant

challenge to structural biology. In collaboration with Khoo and his colleagues, they comprehensively profiled the glycosylation of the PTPRA-ECD, which comprises only 120 amino acids with four N-glycosylation sites and over 30 O-glycosylation sites. Using the SAXS data derived from the PTPRA-ECD fused to a dimeric Fc scaffold and GlycoSHIELD, the team generated an ensemble structure with the N- and O-glycans decorating PTPRA-ECD to account for the spatial occupancy by the N- and O-glycans (**Fig. 3**). They further collaborated with Takayuki Uchihashi at Nagoya University in Japan to directly visualize the opening and closing states of the PTPRA-ECD through high-speed molecular force microscopy to confirm the bottlebrush-like molecular envelope of PTPRA-ECD inferred by SAXS. This interdisciplinary exercise underscores the importance of an integrated structural-biology approach to characterize such a highly complex and dynamic system.⁴

The current modeling algorithm of GlycoSHIELD is limited to the steric effect of glycan ensemble structures on the protein of interest. An improved algorithm is required to account for the non-covalent interactions. Furthermore, the compositional heterogeneity of the individual glycosylation sites presents a combinatorial problem of a large number

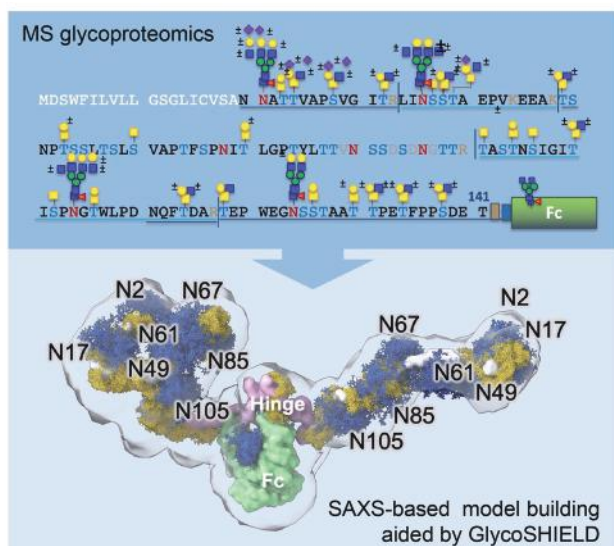


Fig. 3: Integrated structural analysis of the densely N- and O-glycosylated and intrinsically disordered PTPRA-ECD fused to Fc. The representative N- and O-glycoforms derived from MS analysis are shown on the top, and the atomic model of the fully glycosylated protein structure is shown below. The N- and O-glycans are shown in blue and yellow, respectively. The molecular envelope of the Fc-fused PTPRA-ECD is shown on a transparent surface. [Reproduced from Ref. 4]

of overall glycosylation patterns that one should consider when describing the physicochemical properties of the protein of interest at an atomic level. The optimization of GlycoSHIELD's molecular models and experimental observations is necessary to achieve a balance between

the computational performance and the completeness of experimental observations while ensuring that the molecular models of glycoproteins comply with the basic laws of physics and chemistry, an area that will require further explorations. (Reported by Shang-Te Danny Hsu, Academia Sinica)

This report features the work of Shang-Te Danny Hsu and his collaborators published in Cell 187, 1296 (2024), and the work of Kay-Hooi Khoo and his collaborators published in JACS Au 3, 1864 (2023).

TPS 13A Biological Small-angle X-ray Scattering

- Biological Small/Wide Angle X-ray Scattering
- Biological Macromolecules, Protein Solution, Life Science

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The Key to Accurate Cell Division: Centrosome Regulation

Cep57 regulates centrosome maturation and microtubule nucleation via phase separation, ensuring accurate cell division.

Liquid–liquid phase separation (LLPS) is a process driven by multivalent interactions among biomolecules, such as proteins and nucleic acids, forming dense, dynamic structures known as biomolecular condensates. Recent evidence supports LLPS as a fundamental organizing principle for membrane-less organelles. The centrosome, consisting of a pair of centrioles surrounded by pericentriolar material (PCM), plays a crucial role in regulating cell division. As cells prepare for mitosis, the centrosome undergoes a maturation process characterized by the expansion of the PCM and an increase in its microtubule nucleation capacity by recruiting centrosomal scaffolding proteins and microtubule nucleation factors. This facilitates the assembly of the mitotic spindle and ensures the precise segregation of chromosomes during mitosis.

Human Cep57 is a coiled-coil scaffold protein located in the inner layer of the PCM and controls the process of centriole duplication and centrosome maturation for faithful cell division. Cep57 truncation mutations are genetically linked to mosaic-variegated aneuploidy syndrome, which features centrosome amplification and aberrant spindle formation. However, the molecular mechanisms by which Cep57 regulates PCM organization and microtubule assembly remain unknown. Phase separation has been implicated in some key centrosomal scaffolds, such as SPD-5 (a human CDK5RAP2 functional homolog) and the Cep63/Cep152 complex. During interphase, Cep57 forms a complex with the middle layer scaffold Cep63 and Cep152, serving as regulators for centrosome maturation. However, it is unclear whether Cep57 undergoes phase separation and how these centrosomal scaffolding components coordinate to organize the centrosome.

To address these questions, the research team led by Hui-Chun Cheng at National Tsing Hua University employed various analytical techniques and successfully demonstrated that Cep57 assembles into micron-sized biomolecular condensates through LLPS driven by multivalent interactions. Blocking multivalent interactions on Cep57 induced centrosome amplification in the cells. Furthermore, Cep57 condensates were found to facilitate microtubule nucleation by concentrating α/β -tubulin in the condensates. Finally, a novel molecular interplay was observed between the centrosomal scaffold, which is the negative regulation of Cep63 on LLPS, and the microtubule nucleation activity of Cep57. The following report briefly presents their results.

Cheng's team observed that overexpression of Cep57 forms spherical puncta in cells and some of them colocalized with microtubules (**Fig. 1(a)**). To study the molecular mechanism of the puncta formation, they purified Cep57 and found that Cep57 is highly soluble in high salt conditions (1 M NaCl) but self-assembles into micron-sized spherical droplets with liquid-like properties when salt was reduced to 200 mM (**Fig. 1(c)**).

As full-length Cep57 is prone to degradation, a Cep57S construct was created, which has similar properties to Cep57 but allows for higher protein quality to perform biochemical and biophysical characterization (**Fig. 1(b)**). To identify the regions responsible for LLPS, the N-terminal and C-terminal fragments of Cep57 were expressed and purified. It was found that both terminals are necessary for condensate formation. As it was observed that the phase separation of Cep57 is salt-dependent, electrostatic interactions were speculated to play a role in phase separation. Mutagenesis analysis revealed a highly conserved K/R motif located in the C-terminal direction from the NTD, which also contributes to the phase separation of Cep57S. The combined data showed that the NTD, CTD, and K/R motifs contribute to the phase separation of Cep57S.

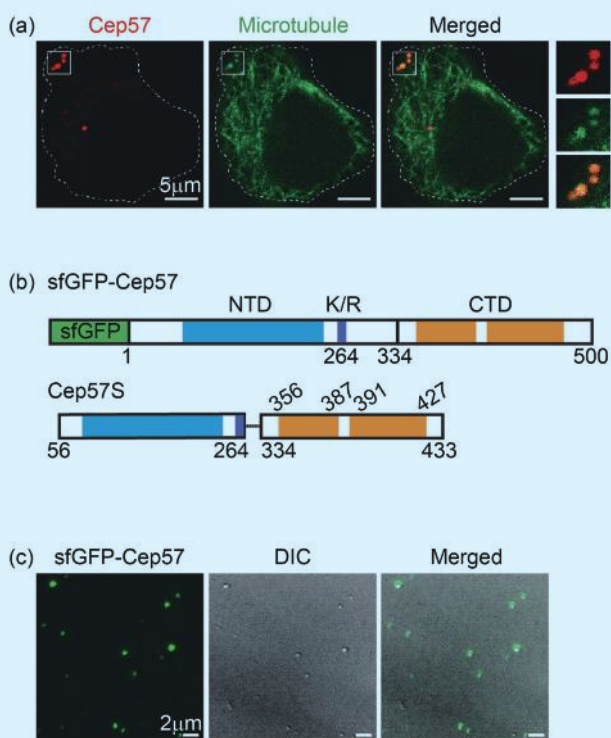


Fig. 1: Human Cep57 undergoes liquid-liquid phase separation under physiological conditions. (a) Confocal images of HeLa cells co-expressing Cep57-mCherry (red) and Neon-MAP4m as the microtubule marker (green) for 16 h. (b) Domain organization of sfGFP-Cep57 and Cep57S. Coiled-coil N-terminal domain (NTD), blue; coiled-coil C-terminal domain (CTD), orange; poly-K/R motif (K/R), dark blue. Cep57S is composed of residues 56 to 264 fused to 334 to 433. (c) TIRF images of sfGFP-Cep57 droplets. The sfGFP-Cep57 (25 nM) were visualized 4 h after condensation in low salt (200 mM NaCl). [Reproduced from Ref. 1].

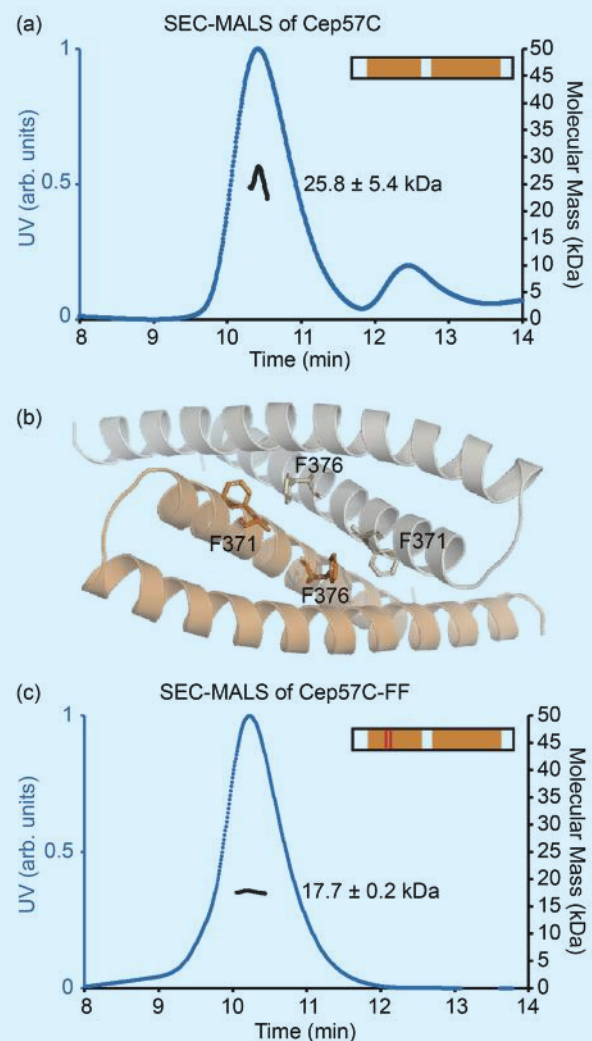


Fig. 2: CTD dimerization facilitates phase separation of Cep57. (a) SEC-MALS profile of Cep57C. The calculated molecular mass for Cep57C is 25.8 kDa. (b) The crystal structure of Cep57C is shown as a dimer. The side chains of F371 and F376 are in stick presentation. (c) SEC-MALS profile of Cep57C-FF. [Reproduced from Ref. 1]

From the SEC-MALS analysis and crystal structure of Cep57C, Cheng's team showed that Cep57C, which adopts a helix-turn-helix fold, forms a symmetric dimer with residues F371 and F376 at the dimer interface (Figs. 2(a) and 2(b)). When these two residues were mutated to alanines in the Cep57S-FF mutant, the dimer formation was weakened (Fig. 2(c)), and the phase separation ability decreased. Therefore, Cep57C dimerization is essential to promote Cep57 condensation.

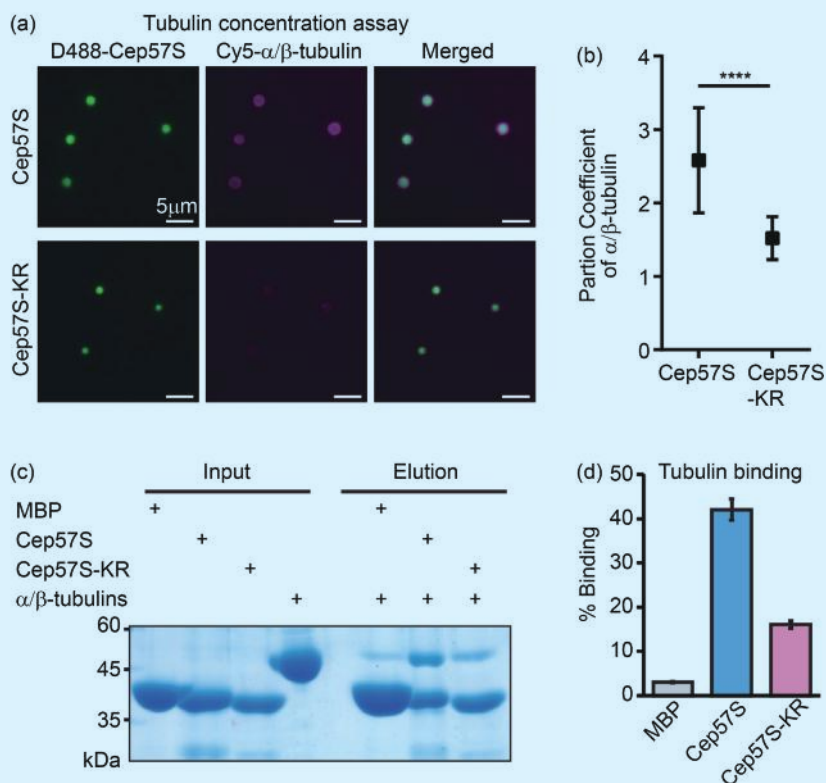


Fig. 3: The Cep57 scaffold concentrates α/β -tubulin for microtubule assembly. (a) D488-Cep57S or D488-Cep57S-KR condensates with Cy5-tubulin dimers in 66 μM nocodazole. Images were taken 20 min after the addition of tubulin. (b) Quantification of the α/β -tubulin partition coefficient to Cep57S ($n = 69$) or Cep57S-KR ($n = 76$) condensates in experiments from panel (a) pooled from three repeats. [****] $p < 0.0001$. Data represent mean \pm SD. (c) His-tag pull-down assay of tubulin with His-Cep57S and His-Cep57S-KR. His-MBP was used as the negative control. Quantification of binding of α/β -tubulin in experiments. The percentage of binding was defined as the intensity of tubulin in the eluent divided by the intensity of the loaded sample ($N = 3$). (d) Percentage of cells with centrosomal microtubule asters in the microtubule regrowth experiments. HeLa cells were sequentially transfected from left to right with negative control siRNA + empty vector, Cep57 siRNA + empty vector, Cep57 siRNA + Cep57, and Cep57 siRNA + Cep57-KR. Each data point was derived from observations of 100 to 200 cells. [*] $p < 0.05$; [**] $p < 0.01$. $N = 3$. [Reproduced from Ref. 1]

Cep57S condensates were also found to concentrate α/β -tubulin by about 2.6-fold to promote microtubule polymerization inside the Cep57S condensates (Figs. 3(a) and 3(c)). The *in vitro* pull-down assay revealed that the K/R motif is the tubulin binding site. A siRNA rescue experiment with the Cep57S-K/R mutant demonstrated that Cep57 plays a role in centrosomal microtubule organization in a K/R motif-dependent manner (Figs. 3(c) and 3(d)).

The research shows that Cep63 prevents the LLPS and microtubule nucleation functions of Cep57S. When Cep57S formed co-condensates with Cep63, the ability to concentrate α/β -tubulin was decreased (Fig. 4(a)). Additionally, both the condensate size and partition coefficient of Cep57S decreased as the concentration of Cep63N, the Cep57 binding domain, increased (Figs. 4(b) and 4(c)). SAXS measurements taken at the TPS 13A beamline showed that the fractal network of Cep57S was diminished by the addition of Cep63N, further suggesting that Cep63 dissolves Cep57S condensates by disrupting the network structure (Fig. 4(d)). Collectively, these results

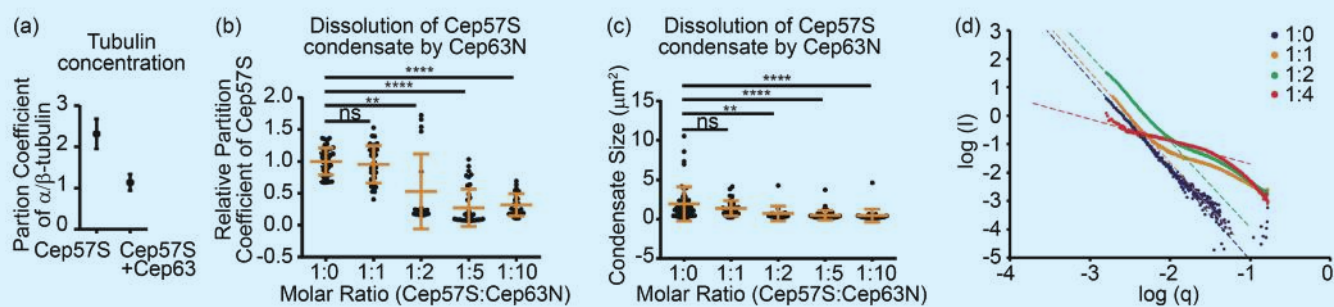


Fig. 4: Cep63 inhibits Cep57S LLPS and function. (a) The partition coefficient of α/β -tubulin in Cep57S or Cep57S-Cep63 condensates. $n = 51$ (Cep57) and $n = 102$ (Cep57S-Cep63) pooled from three independent experimental repeats. (b,c) Partition coefficient and size analysis of Cep57S condensates with increasing concentrations of MBP-Cep63N. The concentration of Cep57S was fixed at 2 μM . Data from two independent experiments were pooled for quantification; $n = 51$ (1:0), $n = 34$ (1:1), $n = 19$ (1:2), $n = 39$ (1:5), and $n = 28$ (1:10). (d) SAXS profile of Cep57S fixed at 10 μM in the presence of MBP-Cep63N at indicated ratios (Cep57S:MBP-Cep63N). Proteins were incubated at 22 $^{\circ}\text{C}$ for 12 hours before the SAXS experiments. The dotted line represents extrapolation of the linear regression of scattering data in the q range from 0.01 \AA^{-1} to 0.002 \AA^{-1} or 0.01 \AA^{-1} to 0.006 \AA^{-1} (for 1:1 only). Slopes for linear regression were -3.2 (1:0), -3.2 (1:1), -3.0 (1:2), and -0.8 (1:4). As MBP-Cep63N increased, the absolute value of the slope decreased, implying that the complexity of the structure decreased. [Reproduced from Ref. 1]

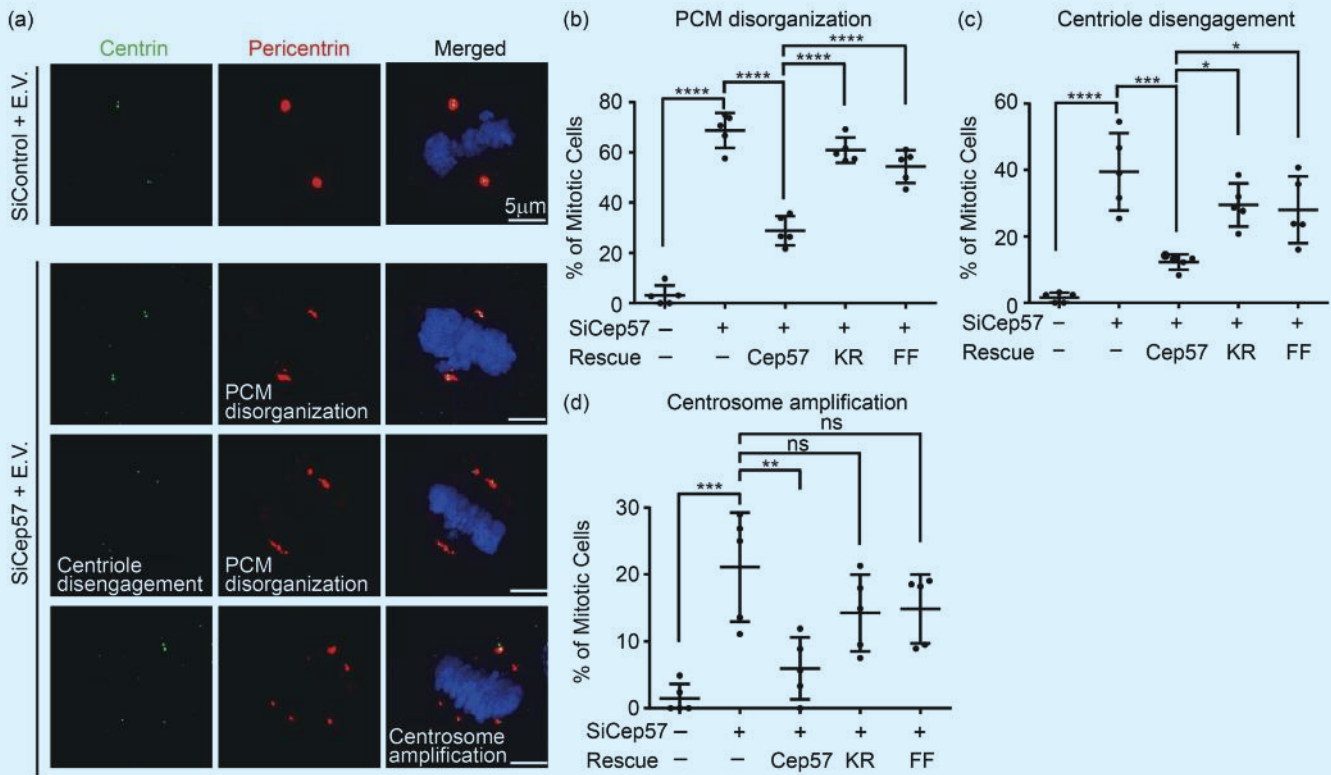


Fig. 5: Multivalent interactions of Cep57 regulate the structure and number of human centrosomes. (a) Fluorescence images of mitotic HeLa cells sequentially transfected with negative control siRNA and mCherry empty vector, or Cep57 siRNA and mCherry empty vector. Centrin, green; pericentrin, red; DNA, blue. (b–d) Percentage of mitotic cells with indicated phenotypes. HeLa cells were sequentially transfected from left to right with negative control siRNA + mCherry empty vector, Cep57 siRNA + mCherry empty vector, Cep57 siRNA + Cep57-mCherry, Cep57 siRNA + Cep57-KR-mCherry, and Cep57 siRNA + Cep57-FF-mCherry, followed by synchronization. Each data point was collected from 30 to 50 mitotic cells. $N = 5$. [*] $p < 0.05$, [**] $p < 0.01$, [***] $p < 0.001$, and [****] $p < 0.0001$. [Reproduced from Ref. 1]

suggest that modulating LLPS of Cep57 by Cep63 serves as a mechanism to regulate the structural expansion and functional activity of the PCM scaffolding proteins.

Finally, cell assays with Cep57-FF and Cep57-KR mutants were performed to examine the functional significance of Cep57 in centrosome duplication. Depletion of endogenous Cep57 by siRNA results in PCM disorganization, centriole disengagement, and centrosome amplification in the mitotic cells (Fig. 5), while introducing siRNA-resistant Cep57 construct rescues these phenotypes. In contrast, Cep57-FF or Cep57-KR mutants failed to rescue these phenotypes. Hence, Cep57 maintains the PCM integrity through multivalent interactions.

Collectively, this study provides crucial insights into the molecular mechanisms by which Cep57 contributes to centrosome organization through LLPS. It highlights the role of Cep57 condensates in promoting microtubule nucleation by concentrating α/β -tubulin and reveals a novel regulatory interaction where Cep63 negatively modulates Cep57's phase separation and microtubule nucleation activity. These findings enhance our understanding of the dynamic assembly of the centrosome, its regulation during cell division, and the potential implications of Cep57

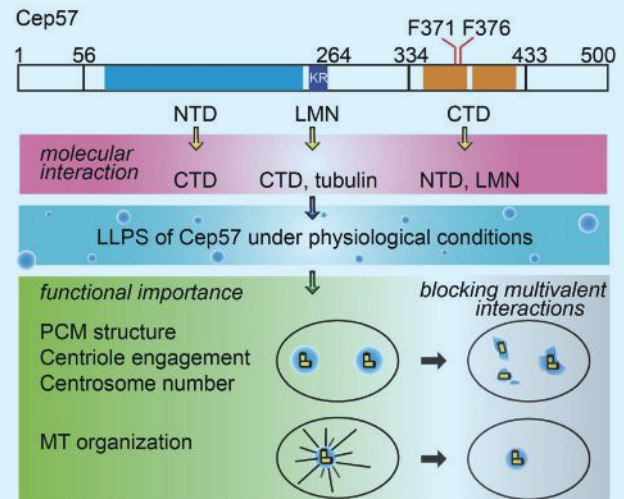


Fig. 6: A model for the assembly of human Cep57 in centrosome regulation. The domain organization of Cep57 is depicted with interactions that drive LLPS under physiological conditions. SiRNA rescue experiments reveal that the multivalent interactions of Cep57 are essential for PCM organization, and the polybasic LMN (LLPS and Microtubule Nucleation) motif facilitates microtubule aster formation. [Reproduced from Ref. 1]

mutations in centrosome-related pathologies, such as mosaic-variegated aneuploidy syndrome. (Reported by Hui-Chun Cheng, National Tsing Hua University)

This report features the work of Hui-Chun Cheng and her collaborators published in PNAS 121, e2305260121 (2024).

TPS 13A Biological Small-angle X-ray Scattering

- Biological Small/Wide Angle X-ray Scattering
- Biological Macromolecules, Structural Biology, Life Science

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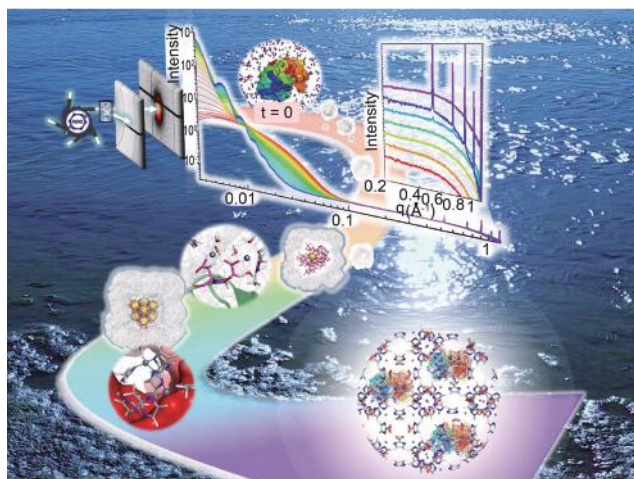
Blueprints in a Drop

This research aims to revolutionize the synthesis of metal–organic frameworks (MOFs) in water by unlocking the molecular codes of biomineralization, paving the way for sustainable innovation.

Metal–organic frameworks (MOFs) have emerged as superior materials for sustainable and transformative applications. Composed of metal ions and organic linkers, MOFs possess exceptional porosity and tunability, making them ideal for diverse uses such as energy conversion, catalysis, water harvesting, and biosensing. However, traditional MOF synthesis methods often rely on organic solvents, raising environmental concerns and hindering scalability and bio applications. Water-based methods for producing zirconium-based and zeolitic MOFs offer significant environmental and economic benefits while enabling the incorporation of biomolecules such as DNA and proteins. Fa-Kun Shei (National Central University) and his team focus on improving protein encapsulation efficiency within MOFs, addressing a critical challenge for expanding their practical applications.^{1,2}

Water's dual role in facilitating proton transfer while suppressing undesired reactions adds complexity to MOF nucleation and crystallization. To address this, Hsiao-Ching Yang's team (Fu Jen Catholic University) has developed a ground-breaking approach to elucidate water's critical role in reaction pathways. Through all-atom molecular dynamics simulations and quantum dynamics calculations, they investigated proton transfer mechanisms and structural evolution during MOF formation, uncovering essential molecular-level insights into factors influencing protein encapsulation. To validate their theoretical findings, Yang collaborated with U-Ser Jeng (NSRRC) and his team at the **TPS 13A** beamline, successfully bridging computation structures with experimental data.³

Accordingly, the interdisciplinary to transdisciplinary teamwork led by Shei, Jeng, and Yang launched a collaborative effort to explore the biomimetic nucleation and crystallization journey using ZIF-8, a zeolitic imidazolate framework, as a model system.³ Through combining advanced techniques such as *in situ* small-/wide-angle X-ray scattering (SWAXS), multiscale simulations, and quantum calculations, the team meticulously tracked the overall process of nucleation and crystallization of ZIF-8 in aqueous solutions in real-time. This complementary approach of all-atom molecular dynamics simulations and experimental observation offered a comprehensive understanding of the molecular blueprint underlying MOF formation, decoding the ZIF-8 nucleation and crystallization process. Their research showed that the process unfolds in three distinct stages:



The Protein@ZIF-8 Crystallization Journey

Stage I: Acidity Flip Proton Transfer Initial Assembly to Amorphous Nuclei. Water in the zinc–water complex is replaced by imidazole ligands, initiating an “acidity flip” that accelerates proton transfer. This shift drives the structural organization of zinc ions into amorphous cluster nuclei; imidazole ligands replace water molecules within zinc–water coordination complexes, forming the first blueprint of the crystalline structure and lead to the assembly of amorphous nuclei—the initial building blocks of the MOF structure. No ordered precursors or diffraction peaks were observed, marking the early aggregation of secondary building units (SBUs) into amorphous nuclei.

Stage II: Transition to Crystalline Nuclei and Ordered Structures. The amorphous nuclei undergo a critical transformation, evolving into ordered nuclei. This stage involves amorphous nuclei being consumed to form ordered mesoscale structures, overcoming energy barriers of approximately 21 kcal/mol in this rate-determining step. The emergence

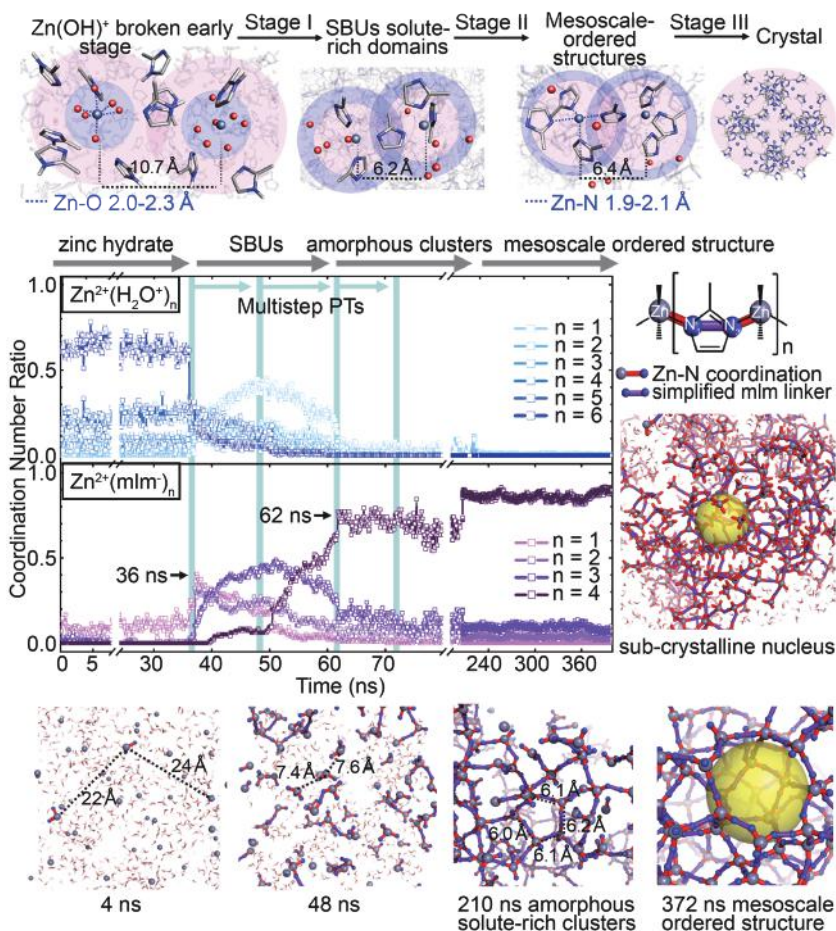


Fig. 1: MD analysis of ZIF-8 reveals a multistep Proton Transfer (PT) mechanism and structural evolution showing key Zn-O (water), Zn-N, and HmIm (N1(H), N3) interactions enabling precise protonation control with SBUs and amorphous clusters forming nucleation sites for crystalline growth in solution. [Reproduced from Ref. 3]

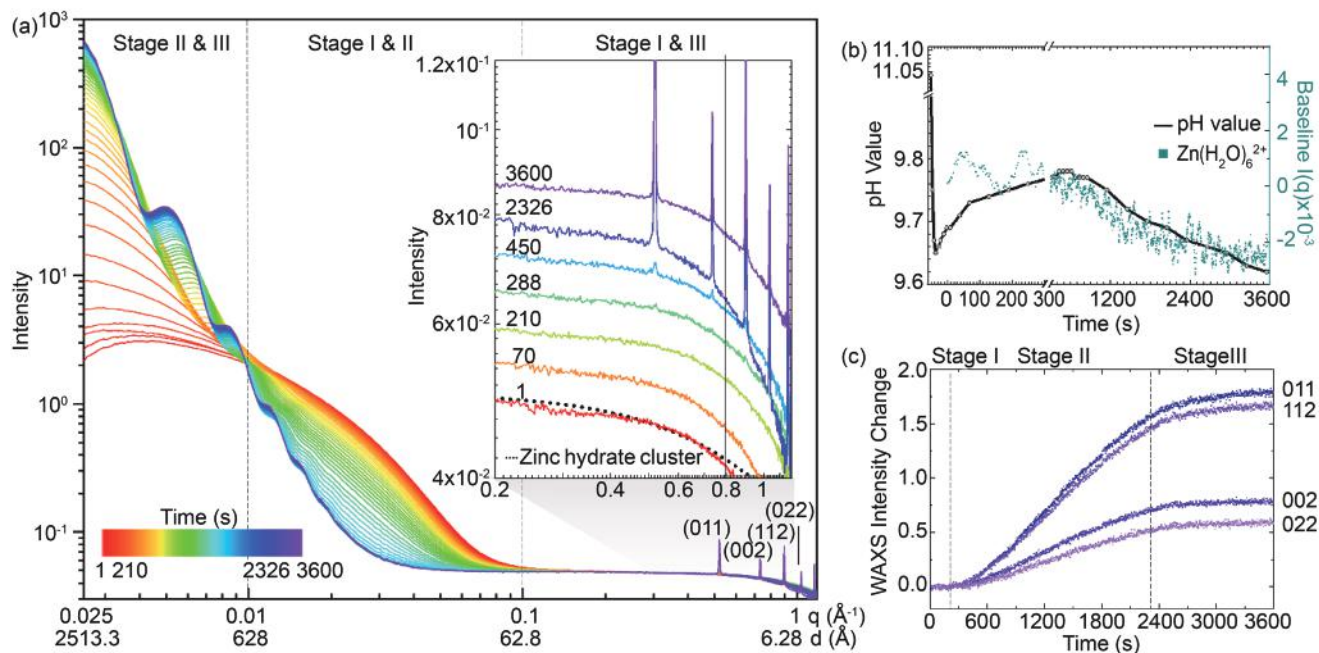


Fig. 2: *In situ* and *operando* SWAXS of ZIF-8 synthesis in aqueous solution for a HmIm/Zn²⁺ molar ratio of 1:57 at 10 °C. (a) The q -range for scattering extends from 0.0025 to 1.2 Å⁻¹, with data acquired every second for a total duration of 3600 seconds. The inset shows an enlarged WAXS region. (b) The scattering profile baseline exhibits a clear dependence on the reaction solution's pH value. (c) The WAXS peaks corresponding to Miller indices (011), (002), (112), and (022) of ZIF-8, which all display correlated time-dependent intensity changes and can be normalized to one reference. [Reproduced from Ref. 3]

of diffraction peaks at specific q -values corresponds to the growth of crystalline nuclei, characterized by the growth of diffraction peaks at $q = 0.52, 0.74, 0.90,$ and 1.04 \AA^{-1} , corresponding to the Miller indices (011), (002), (112), and (022), respectively.

Stage III: Formation of Stable Nanoparticles toward Crystals. Stable crystal nanoparticles emerge when molecular interactions between the crystals and the

surrounding solution reach temperature-dependent thermal equilibrium. This occurs when the structural dimensions stabilize, corresponding to the completion of the second stage. This appears to involve a temperature-dependent thermal equilibrium of the molecular interaction dynamics at the crystal–solution interface.

Biomolecular Integration in MOF Encapsulation

The results showed an inverse relationship between

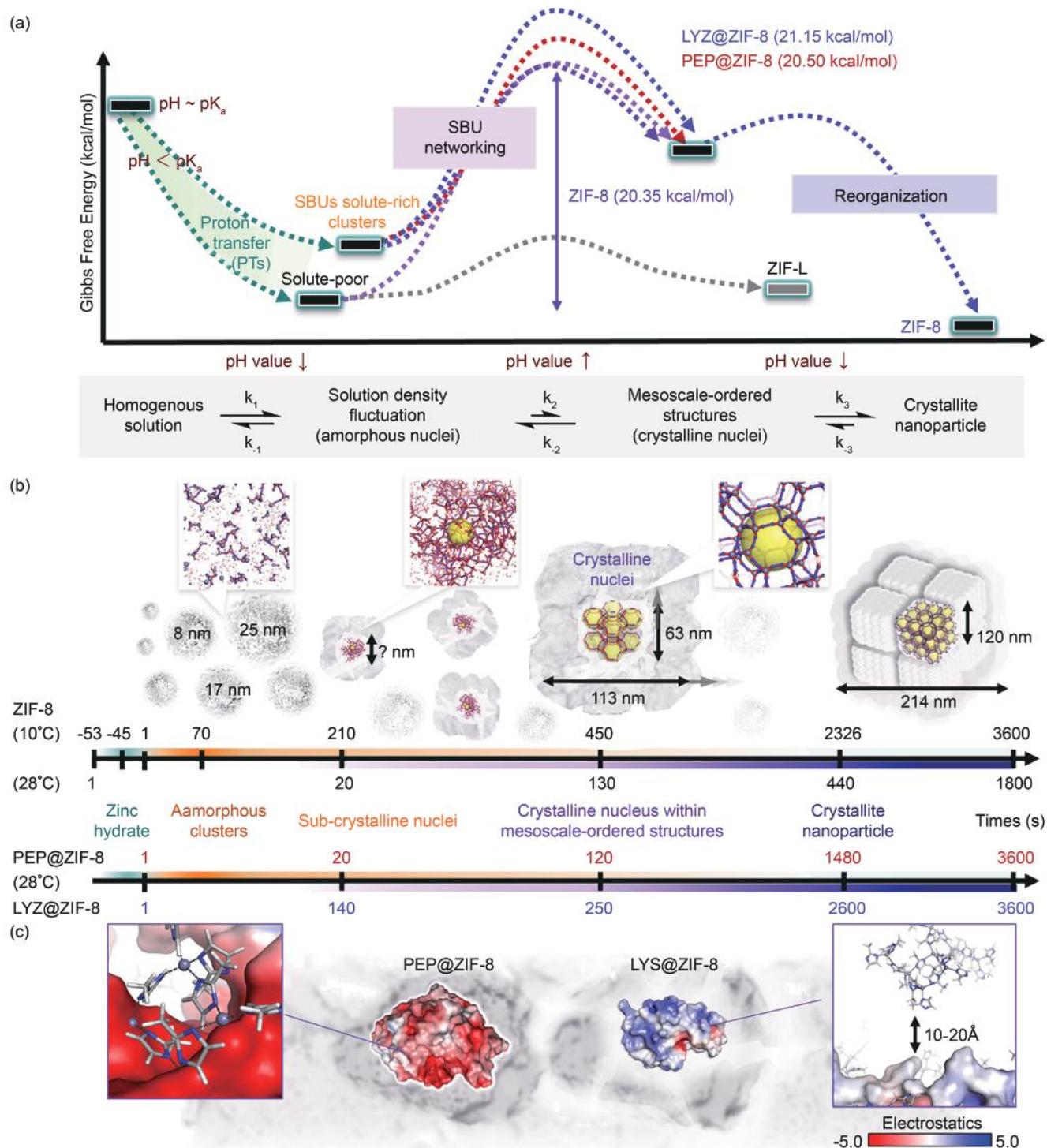


Fig. 3: *In situ* SWAXS reveals ZIF-8 and protein@ZIF-8 synthesis mechanisms. (a) Gibbs free energy landscapes for ZIF-8 systems. (b) Time-resolved nucleation and growth under various conditions. (c) Protein effects: PEP (negative, red) binds strongly, while LYZ (positive, blue) shows poor interaction due to electrostatic repulsion. [Reproduced from Ref. 3]

temperature and particle size. Experimental data showed that as the temperature increased from 10 to 28 °C, the microcrystal size (D 011) decreased from 139 to 80 nm, while the nanoparticle size decreased from 337 to 97 nm. This phenomenon is mainly attributed to the increased nucleation rate at higher temperatures. With the formation of more crystalline nuclei, the available reactants are distributed among more growing particles, resulting in smaller final particle sizes. Further innovation emerged from studying the effects of biomolecules on MOF formation. By incorporating proteins such as pepsin and lysozyme, researchers demonstrated that amino acid proton transfer ability and concentration directly influence the morphology and encapsulation efficiency of biomolecule–MOF composites. This study identified how proteins act as nucleation sites within amorphous MOF structures. These insights not only advance our understanding of biomineralization but also hint at designing proteins as nucleation sites for amorphous MOF growth.

In summary, this transdisciplinary research offers profound insights into the molecular mechanisms of biomimetic mineralization, where biological principles inspire synthetic material design. The findings pave the way for designing tailored MOF systems for applications. By bridging computational modeling simulations and experimental validation, this study provides a comprehensive “blueprint in a drop”—a molecular-level precision for understanding and optimizing MOF formation. By mastering these

microscopic processes, the team unlocks more sustainable and innovative applications in fields ranging from energy storage to pharmaceuticals. The journey from a single aqueous drop to a complex crystalline network underscores the profound potential of small beginnings in shaping the advanced materials of the future. (Reported by Hsiao-Ching Yang, Fu Jen Catholic University)

This report features the work of Hsiao-Ching Yang and her collaborators published in ACS Nano 18, 25170 (2024).

TPS 13A Biological Small-angle X-ray Scattering

- *In situ* SWAXS
- Physical Chemistry, Chemical Physics, Computation Chemistry, Materials Science, Aqueous Chemistry

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Unlocking Hydrogen Power with Single-Atom Pt Catalysts

3D hierarchically organized metal single atoms have attracted considerable attention for their high efficiency in various catalytic reactions.

Single-atom catalysts have garnered significant attention because of their exceptional activity and efficiency across a broad spectrum of catalytic reactions.¹ However, developing stable single-atom catalysts and cocatalysts that maintain high performance presents significant challenges, particularly in achieving uniform dispersion, stabilization, and a sufficiently high density of single-atom sites. Currently, the majority of reported single-atom catalysts have predominantly been achieved using two-dimensional substrates lacking steric infrastructures. The dispersion of these single-atom catalysts relies heavily on their adsorption or coordination with substrates, such as carbon blacks or graphene surfaces. These weak interactions struggle to counteract the aggregation of single-atom catalysts into nanoparticles during catalytic reactions, leading to suboptimal performance.

Recent studies have suggested the potential use of metal–organic frameworks (MOFs) to create three-dimensional (3D) hierarchical structures, thus allowing the deployment of catalysts/cocatalysts inside MOF cages through diffusion or onsite reduction. This approach aims to mitigate the aggregation effect of single atoms, thereby sustaining their performance. However, achieving a uniform deposition of single catalysts/cocatalysts throughout the deep inner MOF cages presents a significant challenge.

U-Ser Jeng and his team at the NSRRC recently developed a method that involves embedding single platinum (Pt) atoms within silicate nanochannels for a paired single-atom cocatalyst and catalyst to achieve efficient and stable photocatalytic centers of a high number density in 3D substrates. Using phosphotungstic acids (PTAs) as templates, they achieved a high loading of single Pt atoms (3.0 wt%). Advanced techniques, including X-ray absorption spectroscopy (TPS 44A)² and electron microscopy, revealed that Pt atoms are stabilized *via* four-oxygen coordination within PTA, effectively reducing the energy-driving aggregation (Fig. 1). This system's design relies on controlling the ratio of Pt atoms to PTA. A critical ratio of 3.7 ensures nearly pure single-atom dispersion. At higher ratios, Pt clustering becomes evident. The research team used a three-stage solution synthesis process of template formation, Pt adsorption, and reduction at the air-liquid interface to create this unique structure. *In situ* grazing-incidence small-angle scattering (GISAXS) measurements (TLS 23A1 and TPS 13A)^{3,4} and thermogravimetric analyses demonstrated that increasing PTA content enhanced single-atom dispersion, while excessive PTA led to structural deterioration. The resulting Pt-PTA pairs within silicate nanochannels exhibit the outstanding hydrogen evolution reaction (HER) efficiency, achieving a hydrogen production rate of 300 mmol/h/g Pt, which is double that of previous systems. The PTA's empty tungsten d shell facilitates photoexcited electron transfer to Pt, enabling efficient hydrogen reduction. The nanochannels also prevent Pt clustering and sustain long-term performance. This breakthrough establishes a record-high Pt efficiency for HER among polyoxometalate (POM)-based systems. The 3D-ordered structure stabilizes single Pt atoms and creates a synergistic network for enhanced charge transfer. This innovative strategy highlights the potential for developing robust, high-performance single-atom catalysts for energy applications.

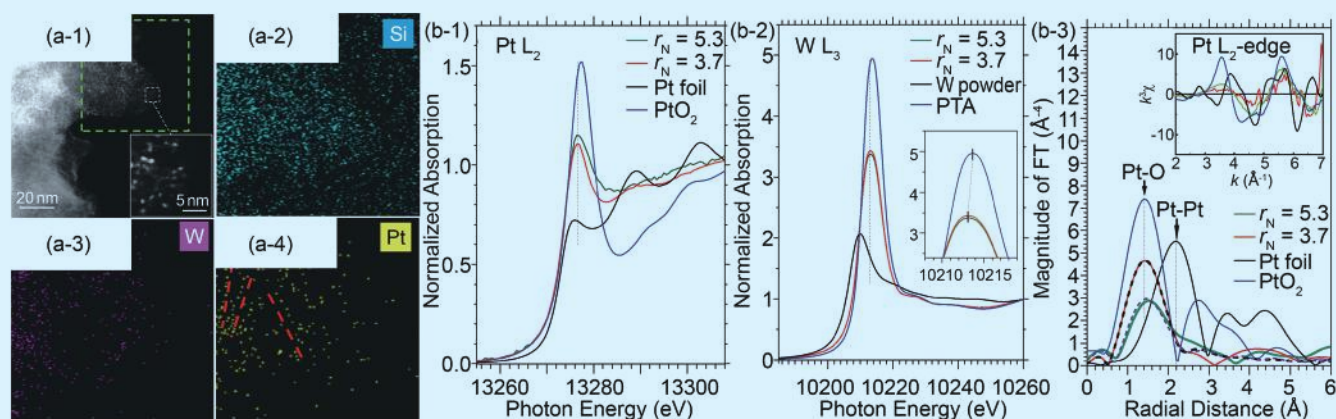


Fig. 1: (a-1) High-angle annular dark field-scanning transmission electron microscopy (HAADF-STEM) image of a typical zone on Pt-PTA within the silicate nanochannels of a low Pt atom/PTA number ratio, r_N , of 5.3; the inset (white square) zooms in on the local details, with the dispersed bright spots contributed by the scattering from heavier elements of Pt and W in the sample. Corresponding energy-dispersive X-ray spectroscopy (EDS) maps of (a-2) Si, (a-3) W, and (a-4) Pt, taken from the sample marked by the green dotted frame in (a-1). (b-1-3) X-ray absorption spectra measured at the (b-1) Pt L₂-edge and (b-2) W L₃-edge for the two samples of Pt-PTA within silicate nanochannels, with $r_N = 5.3$ and 3.7. Also shown are the spectra of Pt foil (measured in transmission mode), PtO₂, and tungsten and PTA powders for comparison. (b-3) Fourier transform of the samples and the references (with no phase corrections). The inset shows the corresponding (same colors) Pt L₂-edge k^3 -weight extended X-ray absorption fine structure (EXAFS) spectra. The data with $r_N = 3.7$ (or 5.3) are respectively fitted with the dotted (or dashed) curve by using a mean first-shell coordination number $N_c = 3.88$ (or 2.6) and a Pt-O bond length $R = 1.93$ Å (or 1.96 Å). [Reproduced from Ref. 5]

From the HER and X-ray absorption spectroscopy (XAS) results, Pt single atoms in Pt₁-PTA pair coordination within the arrayed silicate nanochannels outperform Pt nanoparticles with PTA in the same environment by fourfold efficiency (Fig. 2(a), see next page). Such one-to-one Pt₁-PTA pair coordination has many advantages in the photoelectric conversion of HER *via* metal-to-metal charge-transfer excitation. In this process, the WO₃-based PTA (having an empty d shell with tungsten W⁶⁺ in 5d⁰ configuration) provides photoexcited electrons, with the states of W⁵⁺ in 5d¹ configuration to the coordinated Pt₁ for hydrogen reduction. The PTA-4H site provides Pt₁ a stable absorption and four coordinated oxygen atoms for an oxidation state of ca. +2 (as revealed from the XAS and discrete Fourier transform calculation results). The median oxidation state of Pt₁ presumably is favorable for serving as a co-catalyst to accept photoelectrons from PTA and subsequently transfer the electrons to nearby protons for HER. The hydrogen adsorption energy ΔG of Pt (-0.1 eV) is closer to zero than that of PTA (-0.9 eV) in an acidic environment, further enhancing the HER kinetics *via* faster, easier hydrogen desorption after reduction. These advantages render the Pt₁ of the Pt₁-PTA pair an efficient co-catalyst in the photocatalytic HER mechanism. On top of the high HER efficiency within the Pt₁-PTA pair, the 3D network of a high number density of Pt₁-PTA pairs organized *via* the arrayed silicate nanochannels can further synergistically convert near neighbors' photoelectrons available within the network and those contributed by the PTA in the HER solution. Moreover, the nanochannel pore

structure (ca. 2 nm channel pore size) with densely intercalated PTA (ca. 1 nm size) suppresses the clustering of the channel Pt₁ during catalytic reactions for sustainable performance. With all these advantages, this new hierarchical structure achieves a record high Pt efficiency in HER among the POM-based photocatalytic systems. It may be considered an efficient electrode material in photoelectrochemical cells.

In summary, the team successfully developed a three-stage synthesis process at the air–liquid interface, enabling precise control over the deposition and dispersion of platinum atoms (Pt₁) within hexagonally packed silicate nanochannels. This approach achieved an efficient Pt₁–PTA configuration, demonstrating record-high performance in HER. The outstanding performance of this structure is attributed to the stable 4H-site coordination, enhanced electron transfer efficiency, high-density Pt₁–PTA pairs, and exceptional anti-clustering properties. Moreover, the integration of synchrotron radiation techniques, such as GISAXS and XAS, played a pivotal role in providing high-sensitivity *in situ* analysis, allowing a detailed understanding of the formation mechanism, structural characteristics, and reaction dynamics of Pt₁–PTA pairs within the nanochannels. These advanced characterization methods offered critical data for the experimental process and provided valuable insights for designing and optimizing 3D single-atom catalysts. This study highlights the potential of combining synchrotron radiation capabilities with advanced synthesis techniques to develop highly stable and efficient single-atom catalysts. It paves the way for innovative applications in photochemical and renewable energy conversion systems. (Reported by Je-Wei Chang)

This report features the work of U-Ser Jeng and his collaborators published in *ACS Nano* **18**, 1611 (2024).

TPS 13A Biological Small-angle X-ray Scattering

TPS 44A Quick-scanning X-ray Absorption Spectroscopy

TLS 23A1 Small/Wide Angle X-ray Scattering

- GISAXS, NEXAFS, WAXS
- Materials Science, Chemistry, Surface, Interface and Thin-film Chemistry, Condensed-matter Physics

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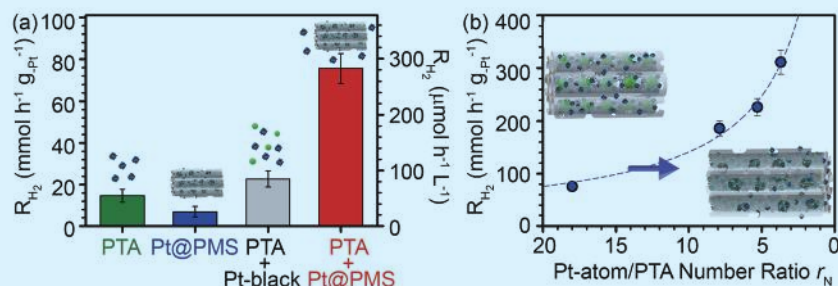
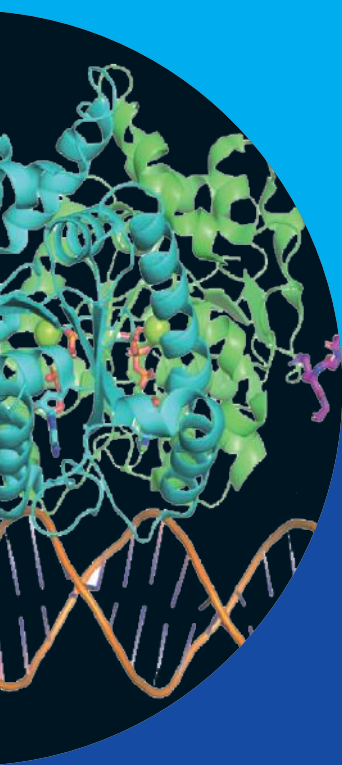


Fig. 2: (a) H₂ production rate measured for a 100 mL solution containing 5 mg of the nanocomposite of Pt–PTA within silicate nanochannels ($r_N=18$), 1.2 mM PTA, 0.2 M H₂SO₄, and isopropanol (20% vol.). Also shown are the H₂ production rates measured in similar solutions for the nanocomposite, solely PTA, and Pt black (of an equal Pt weight) suspension, in similar PTA solutions, as illustrated by the illustrations. (b) Increase in the H₂ production rate with the reduction of r_N of the nanocomposite with increasingly more Pt₁–PTA pairs; inset illustrations show the corresponding transition of Pt dispersion from nanoparticles (left) to Pt₁ on PTA (right) within the arrayed silicate nanochannels. [Reproduced from Ref. 5]

Life Science



Despite advancements in computational techniques, which were recognized by the 2024 Nobel Prize in Chemistry for groundbreaking work, and cryo-EM methods, synchrotron macromolecular crystallography remains an indispensable tool for revealing the intricate atomic structures of biomolecules. This high-resolution structural information is crucial for understanding the structure–function relationships, as well as for driving various scientific applications, from elucidating complex biological mechanisms to supporting drug development and analyzing bio-samples to unravel unsolved issues in biology.

The NSRRC in Taiwan maintains a robust infrastructure to support this field, featuring three specialized protein beamlines—**TLS 15A1**, **TPS 05A**, and **TPS 07A**—that are accessible to a broad community of users. Starting in 2025, the **TPS 05A** and **TPS 07A** beamlines will undergo an upgrade program, which will enable them to provide more diverse functionalities to meet the evolving needs of users better. Furthermore, the NSRRC offers a diverse array of specialized beamlines, including those for small-angle X-ray scattering (**TPS 13A**), soft X-ray tomography (**TPS 24A**), transmission X-ray microscopy (**TPS 31A**), quick-scanning X-ray absorption spectroscopy (**TPS 44A**), white X-ray (**TLS 01A1**), X-ray microscopy (**TLS 01B1**), and infrared microspectroscopy (**TLS 14A1**), enabling researchers to tackle a broad range of biological problems.

The NSRRC user community enjoyed a banner year in 2024, delivering an array of significant scientific breakthroughs. Consequently, we have selected four exemplary studies conducted at our facilities to feature in this activity report. The first study, conducted by Yuh-Ju Sun and Chwan-Deng Hsiao, examined the distinct molecular systems—ParABS in bacteria and Seg filaments in archaea—used by single-celled organisms to ensure accurate DNA partitioning during cell division, revealing the complex and elegant survival solutions even in the simplest life forms. The second study, conducted by Ming-Hon Hou, investigated using bidirectional bis-intercalating acridine compounds to target DNA junction sites as a potential anticancer strategy because these compounds can cross-link and stabilize DNA junctions, thereby disrupting cellular processes and inhibiting cancer cell proliferation. The third study was by Hui-Chun Cheng, who showed that H₂S modification of PKM2 through cysteine 326 sulfhydration reduces its activity, impairs cancer cell division, and leads to tumor suppression, suggesting blocking this H₂S-mediated PKM2 modification as a promising therapeutic approach for targeting cancer cell metabolism. Finally, the fourth study was by Shiou-Ru Tzeng, who revealed how the adaptor protein NlpI regulates the cell wall endopeptidase MepS to maintain bacterial cell wall integrity by modulating MepS activity and targeting it for degradation. (by Chun-Hsiang Huang)

Two Ancient Solutions to One Modern Problem: DNA Management in Single-Celled Organisms

Single-celled bacteria and archaea use different but equally clever molecular systems to partition their DNA during cell division; bacteria with their ParABS motor system and archaea with their Seg filament system. These microscopic mechanisms show that even the simplest life forms have complex and elegant survival solutions.

One of the most fundamental challenges in microscopic cellular life is ensuring accurate DNA partitioning during cell division. This process, much like dividing a vast library's contents between two new locations, requires intricate coordination and precise organization. To address this important scientific phenomenon, Yuh-Ju Sun (National Tsing Hua University) and Chwan-Deng Hsiao (Academia Sinica) collaborated by using various bioassays and protein crystallography techniques to reveal fascinating details regarding how single-celled organisms (bacteria and archaea) accomplish this crucial task. All crystallographic data were collected at **TLS 15A**, **TPS 05A**, and **TPS 07A** of the NSRRC.

Researchers have uncovered the sophisticated ParABS (*par* stands for partitioning) system in bacteria, consisting of three key components working in harmony. ParA acts as a molecular motor powered by ATP, ParB serves as a versatile DNA-binding protein, and the *parS* sequences function as specific DNA anchoring points.¹ In this study, the *Helicobacter pylori* ParB (*HpParB*) protein demonstrates remarkable adaptability, behaving like a skilled librarian who can both locate specific books and organize entire shelves. This versatility is controlled by CTP, which acts as a molecular switch. When CTP is absent, *HpParB* focuses on specific DNA sequences (e.g., *parS*), but in its presence, *HpParB* can slide freely along DNA (**Fig. 1(a)**).

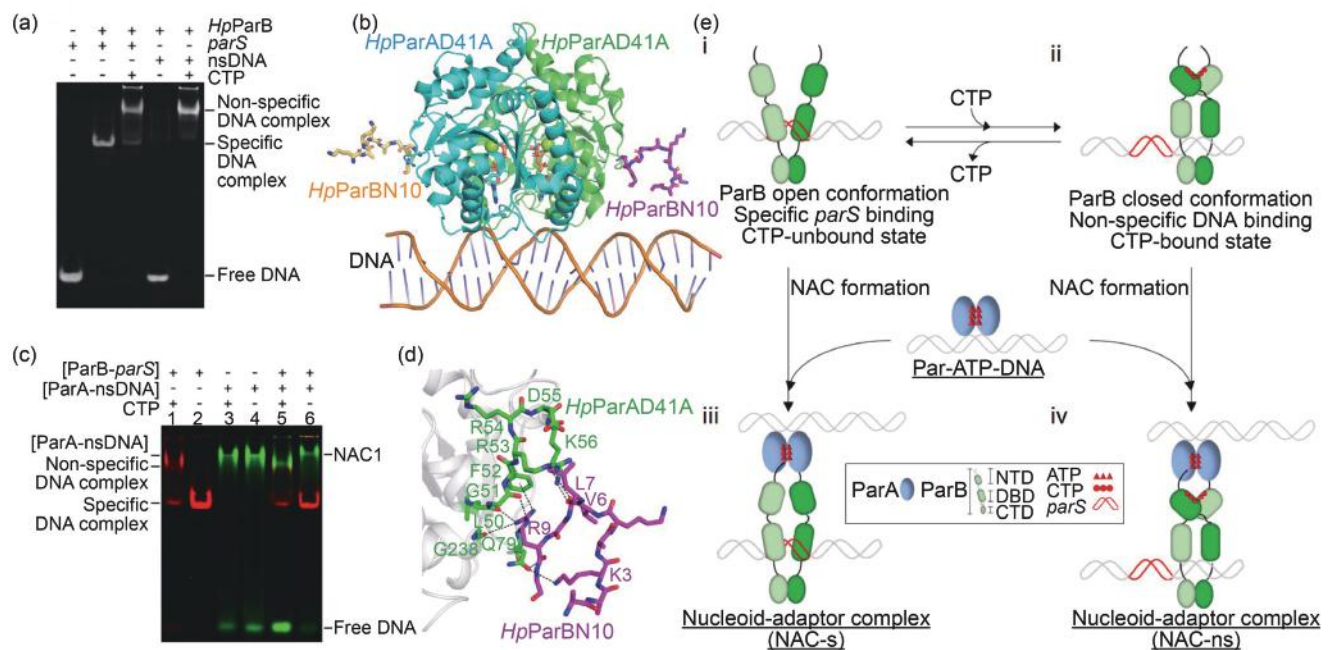


Fig. 1: (a) The electrophoretic mobility shift assay (EMSA) reveals *HpParB*'s pattern for binding to *parS* and non-specific DNA, both with and without CTP present. (b) The *HpParAD41A*-DNA-*HpParBN10* complex structure shows a cyan and green dimer, with orange and magenta *HpParBN10* peptides bound to each monomer, alongside a wheat-colored DNA molecule. (c) EMSA analysis was performed to examine binding interactions between *HpParB*-*parS* and *HpParA*-nsDNA complexes. Initially, *HpParB* and *HpParA* were separately incubated with Cy3-labeled *parS* and Cy5-labeled nsDNA, respectively, either with or without CTP. These preformed complexes were then combined and further incubated under CTP-present or CTP-absent conditions before EMSA detection. (d) In the *HpParAD41A*-DNA-*HpParBN10* complex, the magenta *HpParBN10* binding site interacts with the grey *HpParAD41A* ribbon structure, where green-colored residues form key contacts marked by dashed lines. (e) The ParABS system model shows how green-colored ParB, containing three domains—the N-terminal domain (NTD), the DNA-binding domain (DBD), and the C-terminal domain (CTD)—adopts CTP-regulated open and closed conformations for specific (i) and non-specific (ii) DNA binding. When these Par-DNA complexes interact with purple-blue ParA-ATP-DNA, they form either specific NAC-s (iii) or non-specific NAC-ns (iv) nucleoid-adaptor complexes. [Reproduced from Ref. 1]

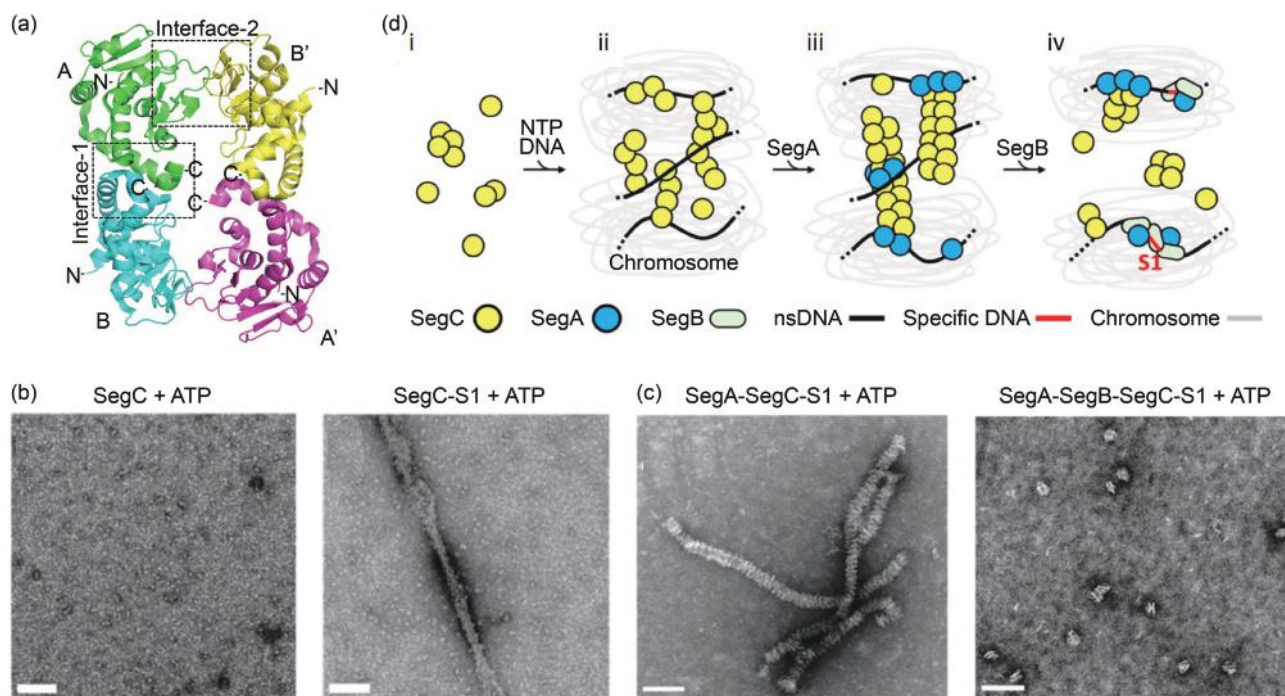


Fig. 2: (a) The SegC tetramer comprises four molecules (A, B, A', B') displayed in green, cyan, magenta, and yellow, respectively, with dimer interfaces marked by dotted squares as Interface-1 and Interface-2. (b) Negative-stain electron microscopy (EM) showing SegC structures with and without DNA and NTP. S1 (site 1) refers to a specific DNA sequence. (c) Negative-stain EM visualization of SegC, SegA, SegB, and DNA interactions. (d) A four-step model for SegC filament function: (i) Random distribution and multimerization of SegC in archaeal cells, (ii) NTP and DNA-dependent filament formation, (iii) SegA-mediated remodeling into higher-order filaments, and (iv) SegB-stimulated SegA ATPase activity leading to filament disassembly. [Reproduced from Ref. 2]

To improve our understanding of this system, researchers employed a clever strategy using a mutant form of *HpParA* (*HpParAD41A*) that displays a slower turnover rate, similar to using slow-motion photography to capture rapid movements. This approach revealed crucial details about how *HpParA* interacts with DNA and uses ATP for energy. *HpParAD41A* allowed scientists to observe the step-by-step process of how *HpParA* recognizes and binds to DNA, providing unprecedented insights into this fundamental mechanism (**Fig. 1(b)**).

One of the most significant discoveries in the bacterial system involves the formation of the nucleoid-adaptor complex (NAC; this term represents the composition of *HpParA*–*HpParB*–DNA). The research revealed that *HpParA* proteins form dimers when bound to CTP, creating a complex with DNA that can interact with *HpParB* through a specialized cation– π interaction. This interaction, particularly between *HpParB*'s Arg9 and *HpParA*'s Phe52, proves essential for the entire system's functionality (**Figs. 1(b) –1(d)**).

The bacterial study also unveiled a detailed molecular mechanism where CTP acts as a master regulator. When CTP is absent, *ParB* maintains an “open” configuration, specifically binding to *parS* sequences and preparing to interact with the *ParA*–ATP–DNA complex. The introduction of CTP triggers *ParB* to adopt a “closed” form,

enabling it to slide along DNA non-specifically and interact more efficiently with *ParA*. This CTP-dependent switching mechanism is crucial for promoting ATP hydrolysis by *ParA* and ensuring proper system function (**Fig. 1(e)**).

Meanwhile, in the ancient world of archaea, researchers have discovered a different but equally fascinating system involving three proteins: SegA, SegB, and the newly identified SegC. Detailed structural analysis revealed that SegC has a unique architectural design that allows it to form both dimers (pairs) and tetramers (groups of four). This molecular architecture, particularly the protein's C-terminal region, proves crucial for its functionality—when researchers removed this tail end, SegC lost its ability to bind DNA and form filaments (**Fig. 2(a)**). The SegC protein shows the remarkable abilities, binding to DNA without sequence specificity and forming thread-like structures (filaments) when it encounters DNA and energy molecules (**Fig. 2(b)**).

The coordination between these components involves SegC working with SegA to form larger filaments in the presence of ATP, while SegB can break these structures apart when needed (**Fig. 2(c)**). The archaeal system's unique feature lies in SegC's ability to break down various energy molecules (NTPs), though the exact role of this capability remains under investigation. The researchers proposed a step-wise process where SegC forms initial filaments, SegA helps

organize these structures, and SegB eventually breaks them down to complete the DNA organization process (Fig. 2(d)).

The implications of this research extend far beyond basic science. Understanding these fundamental processes could lead to new strategies for controlling bacterial growth, potentially contributing to antibiotic developments. Furthermore, since archaea are considered ancient relatives of complex organisms, these findings provide valuable insights into how DNA organization evolved over time. (Reported by Chun-Hsiang Huang)

This report features the work of Yuh-Ju Sun and Chwan-Deng Hsiao published in Nucleic Acids Res. 52, 7321 (2024) and Nucleic Acids Res. 52, 9966 (2024).

TPS 05A Protein Microcrystallography

TPS 07A Micro-focus Protein Crystallography

TLS 15A1 Biopharmaceuticals Protein Crystallography

- Protein Crystallography
- Biological Macromolecules, Protein Structures, Life Science

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Targeting DNA Junctions for Anticancer Drug Development

DNA helix–helix junctions form tetraplex base pairs at the junction interface, serving as “hotspots” for bidirectional bis-intercalating agents. This study investigates the structural basis for targeting DNA junctions with acridine bis-intercalators as a potential anticancer strategy.

Biological processes such as recombination or replication can generate DNA juxtaposed helix–helix structures and duplex crossovers.^{1,2} These structures require topoisomerases to decatenate the interlinked DNA crossover sites.³ Within the crossover structures, the base pairs of the duplexes can interact with each other, resulting in novel junctions. Targeting DNA junction sites with bis-intercalating compounds containing bidirectional linkers could inhibit topoisomerase activity, therefore representing an effective anticancer strategy.^{4,5} Bidirectional bis-intercalators have the unique ability to insert their chromophores simultaneously into the base pairs of two DNA duplexes. This non-covalent bridging ability of small molecules enables them to cross-link DNA junctions, thereby disrupting biological processes critical for cellular function. However,

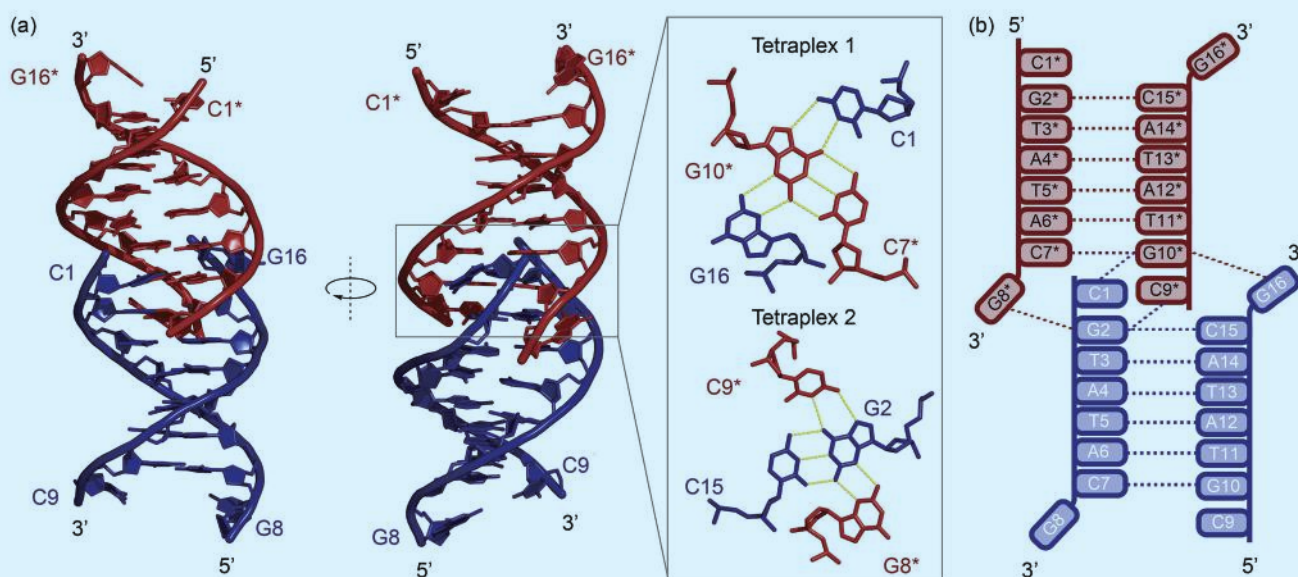


Fig. 1: Structural features of a d(CGTATACG)₂ DNA forming junction. (a) Crystal structure assembly of continuous duplexes forming an end-to-end helix–helix junction structure. One DNA duplex is shown in dark blue and the adjacent symmetry-related duplex is in dark red. Asterisks (*) represent residues in the adjacent duplex. Two layered tetraplex base pairings at the junction interface are shown in an enlarged view. (b) Schematic representation of the crystal structure of d(CGTATACG)₂, indicating the residues involved in junction formation. [Reproduced from Ref. 6]

the limited structural understanding of DNA junction formation and its interactions with small molecules has hindered the development of these targeted therapies.

The study by Ming-Hon Hou (National Chung Hsing University) and his team sheds light on the structural basis of DNA junction formation and provides valuable insights into targeting DNA junctions with bidirectional bis-intercalators for anticancer drug development.⁶ The elucidation of the complex crystal structure required the access to a high-resolution X-ray facility housed at the NSRRC beamline TLS 15A1. Hou's team solved the crystal structure of $d(\text{CGTATACG})_2$ DNA, which exhibited a unique duplex–duplex junction (Fig. 1). In the central region, the structure showed B-DNA-like right-handed features. Interestingly, the terminal CG base pairs contributed to forming a helix–helix junction with two tetraplex base pairs at the junction interface. Detailed analysis revealed that this structure closely resembles the duplex–duplex contacts in catenated DNA and that the tetraplex interface at the junction site serves as a “hotspot” capable of accommodating external ligands between the two neighboring duplexes.

Next, the team explored the possibility of targeting this junction structure with small-molecule compounds. Yih-Chern Horng (National Changhua University of Education) synthesized two alkyl-linked diaminoacridine compounds, DA4 and DA5 (Fig. 2(a)). Both DA4 and DA5 contain acridine chromophores connected by semi-flexible linkers that are four- and five-carbon long, respectively. These two acridine derivatives possess inter-duplex DNA intercalating properties. To investigate the binding mechanism of DA4 and DA5 with DNA, they determined the crystal structures of DA4 and DA5 with the $d(\text{CGTATACG})_2$ sequence in the $C222_1$ and $P2_1$ space groups, respectively, at a resolution of 1.58 Å. As expected, in

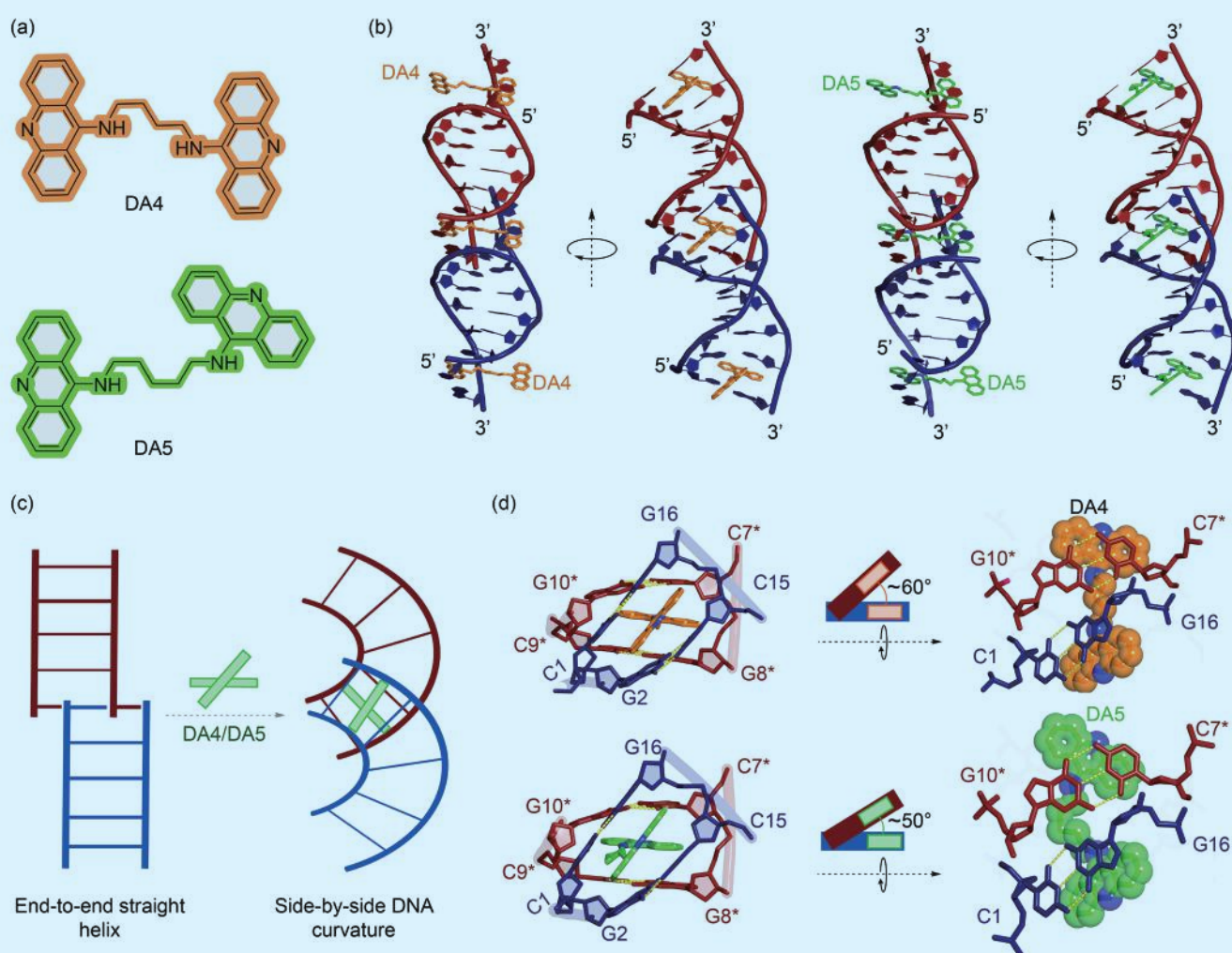


Fig. 2: (a) Chemical structures of the alkyl-linked diaminoacridine bis-intercalators DA4 (orange) and DA5 (green). (b) Crystal structure assembly of DA4–DNA and DA5–DNA complexes showing inter-duplex cross-linking of the DNA duplexes. (c) Schematic diagram showing topological changes in DNA upon intercalation of DA4 or DA5 at the terminal junction site. (d) Magnified view of the intercalation site shows the angled intercalation of DA4 and DA5 at the junction interface in steps C1pG2/C15pG16 in one duplex and C7*pG8*/C9*pG10* in the other adjacent duplex, viewed from the front and top. In DA4, the linker connecting the two acridine moieties is straight, whereas the linker in DA5 has a bent conformation. A single-atom difference in the linker of DA4 and DA5 led to distinct propeller geometries with approximately 60° and 50° between two ligands, respectively. [Reproduced from Ref. 6]

both crystal structures, the intercalation of DA4 or DA5 mediated DNA–DNA contacts and cross-linked adjacent duplexes (Fig. 2(b)). Compared to the unliganded native DNA junction structure, DA4 and DA5 induced significant changes in DNA topology, transforming it from an end-to-end straight helix to a side-by-side curved geometry (Fig. 2(c)). The cross-linking of DNA duplexes also caused a transition from the B-form to an A-form-like conformation, accompanied by bending and overwinding of the backbone. The different linker lengths and flexibilities of DA4 and DA5 resulted in distinct local structural and stabilizing effects on DNA. In the DA4–DNA complex, the four-carbon linker of DA4 adopted a straight geometry, while the DA5 linker bent toward one of the DNA backbones (Fig. 2(d)). This difference in linker flexibility caused the chromophores of DA4 and DA5 to stagger at different angles. In the DA4–DNA complex, the chromophores formed an angle of approximately 60° with the horizontal plane of the acridine ring, while in the DA5–DNA complex, the angle between the two acridine chromophores was approximately 50°. This flexibility of the DA5 linker allowed its chromophores to align more optimally, enabling continuous stacking interactions with DNA base pairs. Consequently, DA5 exhibited more stacking interactions with DNA than DA4. The bent linker and less tilted chromophore of DA5 brought its amino groups closer to the cytosine bases, facilitating a direct water-mediated interaction that likely resulted in a more stable complex with stronger binding. By contrast, the greater distance between the amino group of the DA4 linker and the keto group of cytosine in the DA4–DNA complex led to indirect and weaker water-mediated interactions. These findings suggest that DA5 induces stronger structural changes in DNA than DA4, potentially leading to stronger stabilizing effects. These results were further corroborated through biophysical experiments.

When tested in *in vitro* and *in vivo* models, the two acridine derivatives inhibited topoisomerase II activity, induced G2/M phase accumulation in the cell cycle, triggered apoptosis, and reduced cancer tumor growth, highlighting the anticancer potential of this mode of DNA binding. Notably, the results showed that DA5 exhibited more pronounced anticancer effects than DA4, likely due to its enhanced stability and stronger DNA-binding interactions at the junction. Through investigation of the structural basis for these results, Hou and his team demonstrated how small molecules can precisely target and stabilize DNA junction sites, inhibit topoisomerase activity, and impair cancer cell proliferation. These findings could guide the development of more effective derivatives in future, paving the way for targeting DNA–DNA duplex contacts through bis-intercalation. (Reported by Roshan Satange, National Chung Hsing University)

This report features the work of Ming-Hon Hou and his collaborators published in Nucleic Acids Res. 52, 9303 (2024).

TLS 15A1 Biopharmaceuticals Protein Crystallography

- X-ray Crystallography
- Biological Macromolecules, Cancer, DNA Junctions, Life Science

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Breaking Cancer's Energy Code: New Insights into Hydrogen Sulfide and Pyruvate Kinase M2 Regulation

Hydrogen sulfide modifies the pyruvate kinase M2 enzyme at cysteine 326, affecting cancer cell metabolism. Blocking this modification reduces PKM2 activity and impairs cancer cell division, leading to complete tumor suppression.

Cancer cells undergo significant metabolic reprogramming that is primarily characterized by the Warburg effect. In this process, they prefer aerobic glycolysis over oxidative phosphorylation. Although this process is less efficient for ATP production, it provides essential metabolic intermediates for rapid cancer cell proliferation under nutrient-limited conditions.

The glycolytic pathway is regulated by three key enzymes, with the pyruvate kinase M2 (PKM2) playing a crucial role. PKM2, which is predominantly expressed in cancer cells, differs from PKM1's catalytic properties. While PKM1 maintains high activity as a tetramer, PKM2's activity is typically low in cancer cells because of various post-

translational modifications, which allow redirection of glucose metabolism toward biomass synthesis.

Recent research has focused on hydrogen sulfide (H₂S), an endogenous gasotransmitter that shows concentration-dependent effects on cancer progression. H₂S primarily functions through protein sulfhydrylation, a post-translational modification where H₂S forms a persulfide (-SSH) bond on cysteine residues of target proteins.¹ At lower concentrations, H₂S promotes tumor growth through multiple mechanisms, including antiapoptotic effects, DNA repair, and angiogenesis. However, higher concentrations can inhibit cancer cell proliferation.

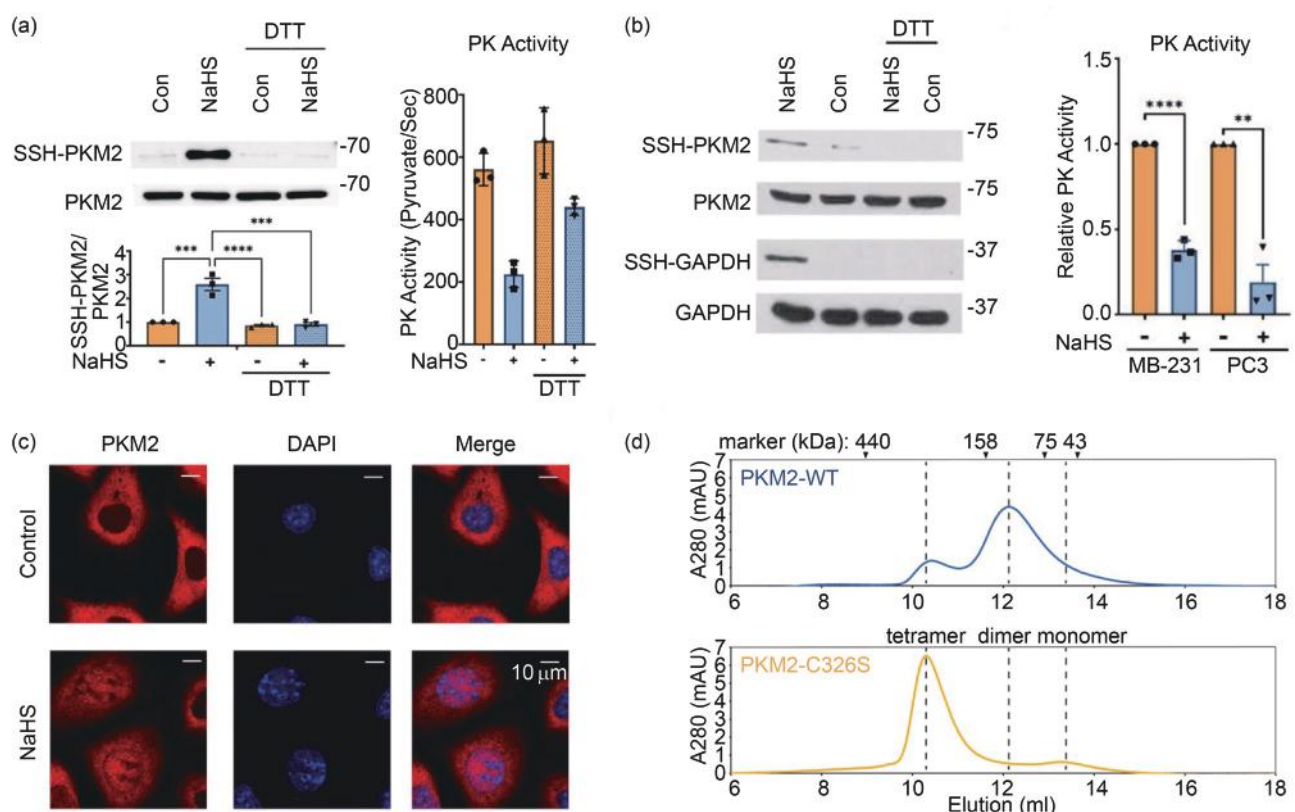


Fig. 1: (a) Results of two main analyses performed in the experimental study. The first part examined PKM2 sulfhydrylation in MDA-MB-231 cell lysates treated with NaHS and DTT by using a biotin switch assay followed by immunoblotting with an anti-PKM2 antibody. The second part investigated pyruvate kinase activity by measuring pyruvate production in recombinant PKM2 after treatment with NaHS and DTT under ice-cold conditions. (b) Results of the experiment investigating protein sulfhydrylation and enzymatic activity in two parts. The first part examined PC3 cell lysates treated with NaHS and DTT by using biotin switch assay and immunoblotting to detect sulfhydrylation of both PKM2 and GAPDH (used as a positive control). The second part focused on measuring pyruvate kinase activity in both MDA-MB-231 and PC3 cell lysates after NaHS treatment, specifically by quantifying pyruvate production. (c) Treatment of MDA-MB-231 cells with NaHS and visualization of PKM2 localization using immunocytochemistry with DAPI nuclear staining (scale: 10 μm). (d) Results of gel filtration analysis of PKM2 proteins (WT and C326S mutant) performed without FBP to examine their oligomeric states. [Reproduced from Ref. 2]

The interplay between H₂S and PKM2 represents a fascinating area of research, particularly given that PKM2 activity can be inhibited by L-cysteine, which serves as the primary source for H₂S production. This intricate connection, which is mediated through protein sulfhydrylation, reveals a critical regulatory mechanism in cancer cell metabolism. The emerging understanding of how H₂S-mediated protein sulfhydrylation influences cancer progression has opened promising new avenues for therapeutic interventions that specifically target cancer-specific metabolic pathways. To further explore this novel regulatory mechanism, Lu-Hai Wang (China Medical University), Hui-Chun Cheng (National Tsing Hua University), and Kai-Ti Lin (National Tsing Hua University) launched a collaborative effort to investigate the molecular mechanisms underlying H₂S-mediated PKM2 regulation in cancer cells.

The researchers first investigated the role of H₂S in PKM2 activity through protein sulfhydrylation. **Figures 1(a) and 1(b)** show that treatment with NaHS, a H₂S donor, induced PKM2 sulfhydrylation in both breast cancer MDA-MB-231 and prostate cancer PC3 cells; this modification reduced PKM2 enzyme activity, which could be reversed by dithiothreitol (DTT) treatment. Importantly, H₂S caused the dissociation of fructose 1,6-bisphosphate (FBP)-induced PKM2 tetramers into monomers or dimers. This led to enhanced PKM2 nuclear translocation (**Fig. 1(c)**) and increased expression of PKM2-responsive genes, such as cyclin D1 and glutaminase-1; the expression levels of these

genes show a positive correlation with tumor growth. When H₂S was depleted using the aminooxyacetic acid inhibitor or by knocking down H₂S-producing enzymes, such as cystathionine β-synthase and cystathionine γ-lyase, both PKM2 sulfhydrylation and nuclear translocation decreased.

Through mass spectrometry analysis, the researchers identified two sulfhydrylation sites on recombinant PKM2: cysteines 49 and 326. However, only cysteine 326 was found to be endogenously sulfhydrated in MDA-MB-231 cells. To further study the effects of sulfhydrylation at this site, they created a mutation replacing cysteine 326 with serine (PKM2^{C326S}). This mutation significantly reduced PKM2 sulfhydrylation and, notably, resulted in increased tetramer formation compared to wild-type PKM2 (**Fig. 1(d)**). Crystal structure analysis revealed that PKM2^{C326S} adopts a unique tetrameric conformation that is different from previously known conformations (**Fig. 2(a)**). The X-ray diffraction data were collected at **TPS 07A** of the NSRRC.

The functional consequences of blocking PKM2 sulfhydrylation at C326 were substantial. The PKM2^{C326S} mutation increased pyruvate kinase activity and led to significant metabolic changes in cells. Cells expressing PKM2^{C326S} showed increased oxygen consumption rates and enhanced mitochondrial oxidative phosphorylation, while extracellular acidification rates only slightly increased. These cells also showed reduced expression of most PKM2-responsive genes, decreased levels of glycolytic intermediates, and reduced nuclear translocation of PKM2.

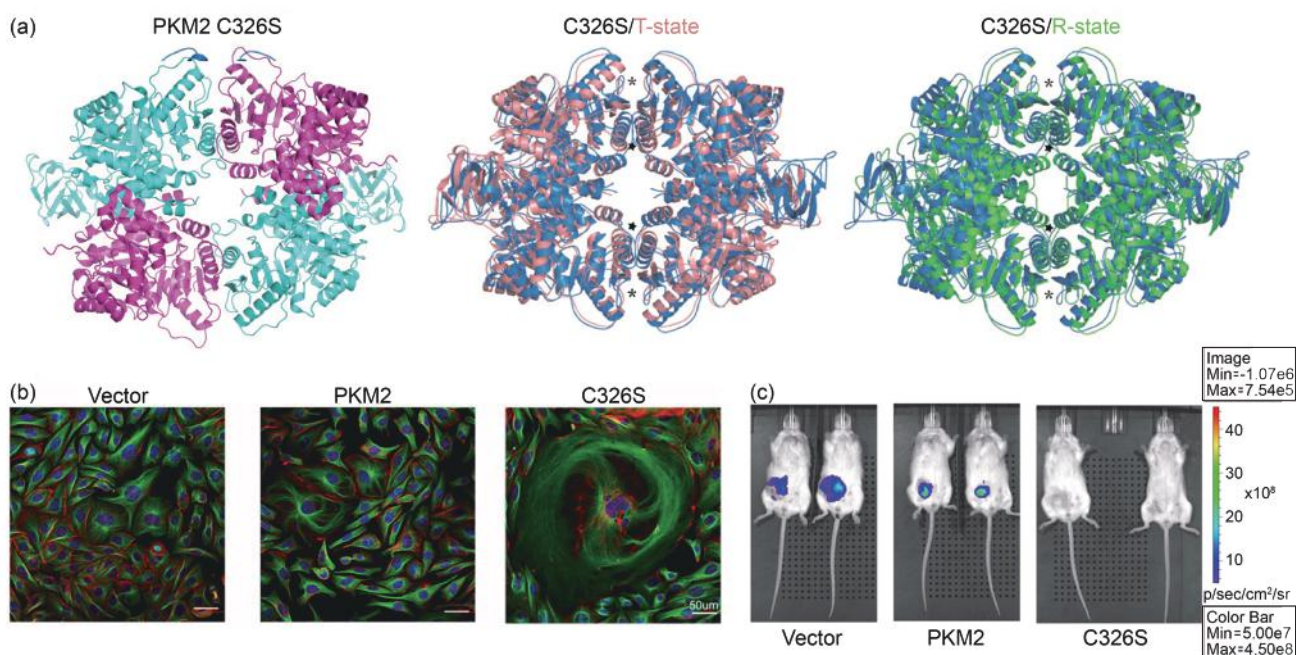


Fig. 2: (a) Structural analysis comparison of the crystal structure of PKM2 C326S mutant with both T-state (phenylalanine-bound) and R-state (FBP/serine-bound) conformations, highlighting key regions: the effector loop and $\alpha 14$ - $\alpha 15$ regions, which are indicated by asterisk and star, respectively. The overlaid structures are color-coded as C326S mutant in blue, T-state in pink, and R-state in green to allow for direct comparison of conformational differences between these states. (b) Immunostaining of MDA-MB-231 cells expressing vector, PKM2, or PKM2^{C326S}, showing actin (red), α -tubulin (green), and nuclei (DAPI, blue). Images are displayed at 50 μ m scale. (c) Bioluminescence imaging of tumor growth in mouse mammary fat pads at week 7 post-implantation of MDA-MB-231 cells expressing different PKM2 variants (vector, wild-type, or C326S). [Reproduced from Ref. 2]

The researchers observed significant negative effects on cancer cell division and proliferation in cells expressing PKM2^{C326S}. These cells showed an increased percentage of polyploidy cells and a higher frequency of giant multinucleated cells (Fig. 2(b)). Time-lapse microscopy revealed a 1.5-fold increase in cytokinesis failure in PKM2^{C326S}-expressing cells. Furthermore, PKM2^{C326S} failed to interact with the spindle checkpoint protein BUB3, and cells showed reduced proliferation rates. Similar effects were observed in cells with H₂S depletion, supporting the specific role of sulfhydration in these processes.

Perhaps most significantly, in a mouse xenograft model, tumor growth was completely suppressed in the PKM2^{C326S} group (Fig. 2(c)). While there were no significant differences in mouse body weight between groups, there was a dramatic reduction in tumor bioluminescence signals in the PKM2^{C326S} group, indicating strong anti-tumor effects.

These comprehensive results demonstrate that H₂S-mediated sulfhydration of PKM2 at C326 is a crucial mechanism regulating cancer cell metabolism. Blocking this modification through the PKM2^{C326S} mutation leads to the stabilization of PKM2 tetramers, enhanced oxidative phosphorylation, reduced nuclear translocation and transcriptional activity, impaired cell division, and

suppressed tumor growth. These findings suggest that targeting PKM2 sulfhydration could be a promising therapeutic approach for cancer treatment, particularly by rewiring glucose metabolism from aerobic glycolysis to oxidative phosphorylation. The study provides both mechanistic insights into cancer metabolism and potential therapeutic strategies for future drug development. (Reported by Chun-Hsiang Huang)

This report features the work of Hui-Chun Cheng and her collaborators published in Nat. Commun. 15, 7463 (2024).

TPS 07A Micro-focus Protein Crystallography

- Protein Crystallography
- Biological Macromolecules, Protein Structures, Life Science

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Structure of the Prc-NlpI-MepS Complex: Elucidating the Regulatory Mechanism of Bacterial Cell Walls

This study reveals how the adaptor protein NlpI regulates the activity and cellular levels of the cell wall endopeptidase MepS, which facilitates peptidoglycan remodeling and maintains cell wall integrity during bacterial growth and development.

Peptidoglycan (PG) is vital for protecting bacterial cells from osmotic pressure. It consists of linear glycan strands of alternating *N*-acetylglucosamine (NAG) and *N*-acetylmuramic acid (NAM), linked to short peptide chains. The main cross-linking of the short peptide chains is the 4–3 linkage between D-Ala and meso-diaminopimelic acid (DAP), forming a net-like structure that prevents osmotic rupture. During cell growth, the net-like structure must be cleaved to incorporate new PG strands, a process facilitated by several endopeptidases, including MepS, MepM, and MepH. In *Escherichia coli* (*E. coli*), these three endopeptidases are essential for cell wall expansion and their absence leads to abnormal cell shapes and lysis.

NlpI is an outer membrane-anchored lipoprotein found in Gram-negative bacteria (*e.g.*, *E. coli*) and plays multiple roles in cell division, cell wall metabolism, virulence, and host interactions. It interacts with various hydrolases and associates with the PG synthesis machinery, influencing the stability of cell envelope components. As an adaptor protein, NlpI can bind to three endopeptidases—MepS, MepM, and PBP4—and facilitates the formation of trimeric complexes (*e.g.*, MepS-NlpI-PBP4). In addition, NlpI helps localize these enzymes, connecting PG hydrolysis to expansion. Reconstitution experiments show that NlpI organizes PG multienzyme complexes, suggesting it aids in integrating hydrolases and synthases during PG expansion.

MepS is abundant during the log phase of cell growth but declines in the stationary phase. Its protein levels within the cell are regulated by the periplasmic PDZ-protease Prc (also called tail-specific protease), in complex with the adaptor NlpI. Without NlpI, Prc cannot effectively degrade MepS, highlighting NlpI's crucial role in MepS recruitment. Mutants lacking Prc or NlpI show increased MepS levels, leading to long filaments and growth defects in low-osmolarity conditions.

In 2017, the structure of the Prc-NlpI complex (PDB ID 5WQL) was determined by X-ray crystallography, revealing a symmetric NlpI homodimer attached to two bowl-shaped Prc proteins. NlpI, an adaptor protein with four tetratricopeptide repeats (TPRs), interacts with Prc through TPR2, forming an extensive electrostatic network. The unliganded PDZ domain of Prc has a misaligned conformation, which rearranges upon ligand binding, activating its proteolytic activity (**Fig. 1(a)**).¹ However, two important issues regarding the regulation mechanism of PG expansion have not been resolved, namely how NlpI regulates MepS to affect their activities and how NlpI modulates the protein levels of MepS in the presence of the Prc protease. To investigate the mechanisms underlying these two important issues, Shiou-Ru Tzeng (National Taiwan University) and her collaborators U-Ser Jeng (NSRRC) and Chun-Hsiang Huang (NSRRC) employed various experimental techniques, including nuclear magnetic resonance (NMR), biological small-angle X-ray scattering (BioSAXS), protein crystallography (PX), and other biochemical analysis methods. Specifically, X-ray diffraction and scattering data were acquired using the beamlines **TPS 05A**, **TPS 07A**, and **TPS 13A** at the NSRRC.

To elucidate how NlpI regulates MepS, NMR experiments were performed (**Fig. 1(b)**) and demonstrated that the mature full-length MepS (mMepS) can utilize its intrinsically disordered N-terminal region to interact with the adaptor protein NlpI. The crystal structure of the NlpI-mMepS complex reveals a heterohexameric assembly, where a homodimer of NlpI binds to four MepS molecules (**Fig. 1(c)**). This structural arrangement facilitates the colocalization and cooperative function of multiple MepS molecules, enhancing their avidity for PG binding and hydrolysis. Notably, upon binding to NlpI, the disordered N-terminal region of MepS undergoes a transition to an ordered state (**Fig. 1(d)**), promoting the dimerization of MepS. This structural insight provides a mechanistic understanding of how NlpI regulates the activity of MepS through modulating its oligomerization state and cooperative interactions with the PG substrate.

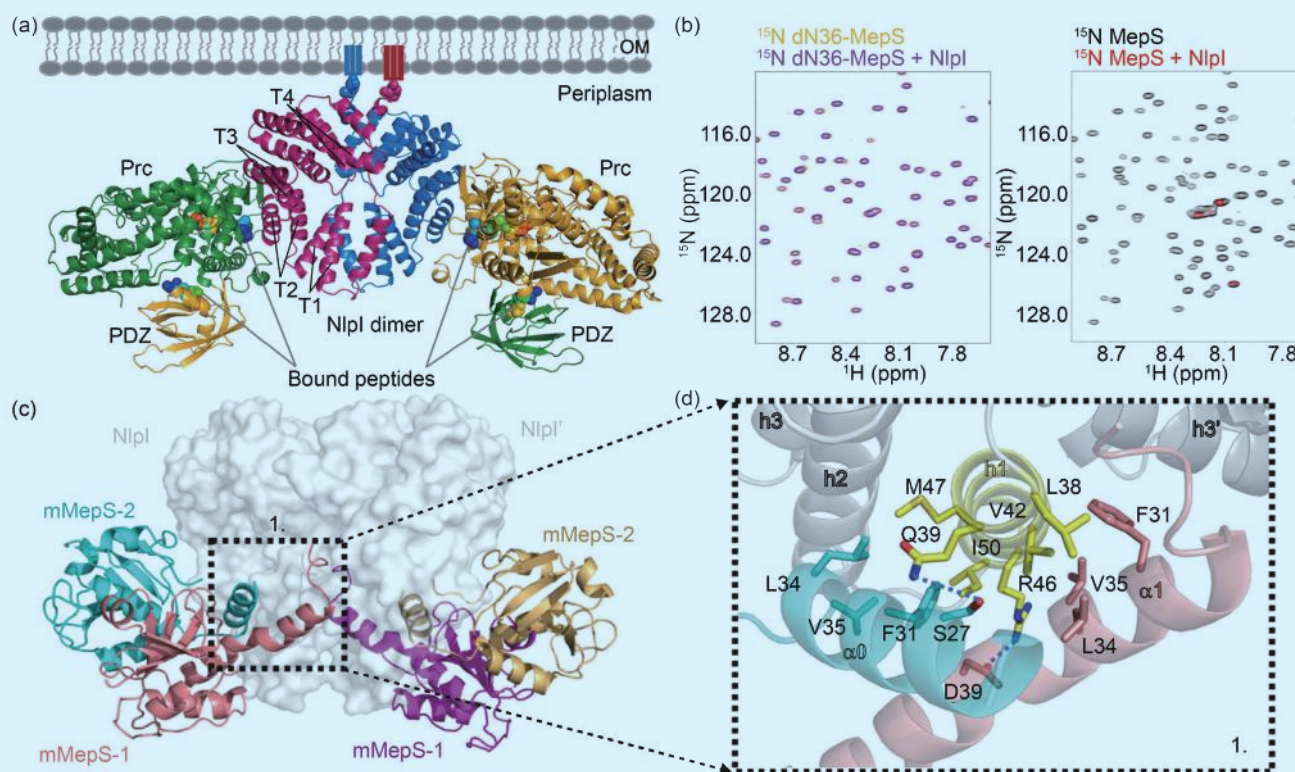


Fig. 1: (a) Overall structure of the NlpI-Prc complex shows a dimeric NlpI bound to two Prc proteins, represented in different colors. The PDZ domain of Prc and the four tetratricopeptide repeats (TPR1–4) of NlpI are labeled. The outer membrane (OM) and lipid anchors are depicted, with the first residues linked to the lipobox cysteine shown as spheres. Four co-crystallized substrate peptides are shown as rainbow-colored spheres. (b) NMR experiments were performed to study the interaction between NlpI and the mMepS proteins. ^1H - ^{15}N TROSY-HSQC spectra were acquired for the truncated mutant dN36-MepS, which is devoid of the N-terminal 36 residues, and mMepS in the absence and presence of unlabeled NlpI dimer. (c) Overall structure of the NlpI-mMepS complex reveals a dimeric NlpI (grey surface) bound to four mMepS proteins (illustrated in different colors). (d) The interactions between NlpI and mMepS involve specific hydrophobic contacts. [Reproduced from Ref. 1 and Ref. 2]

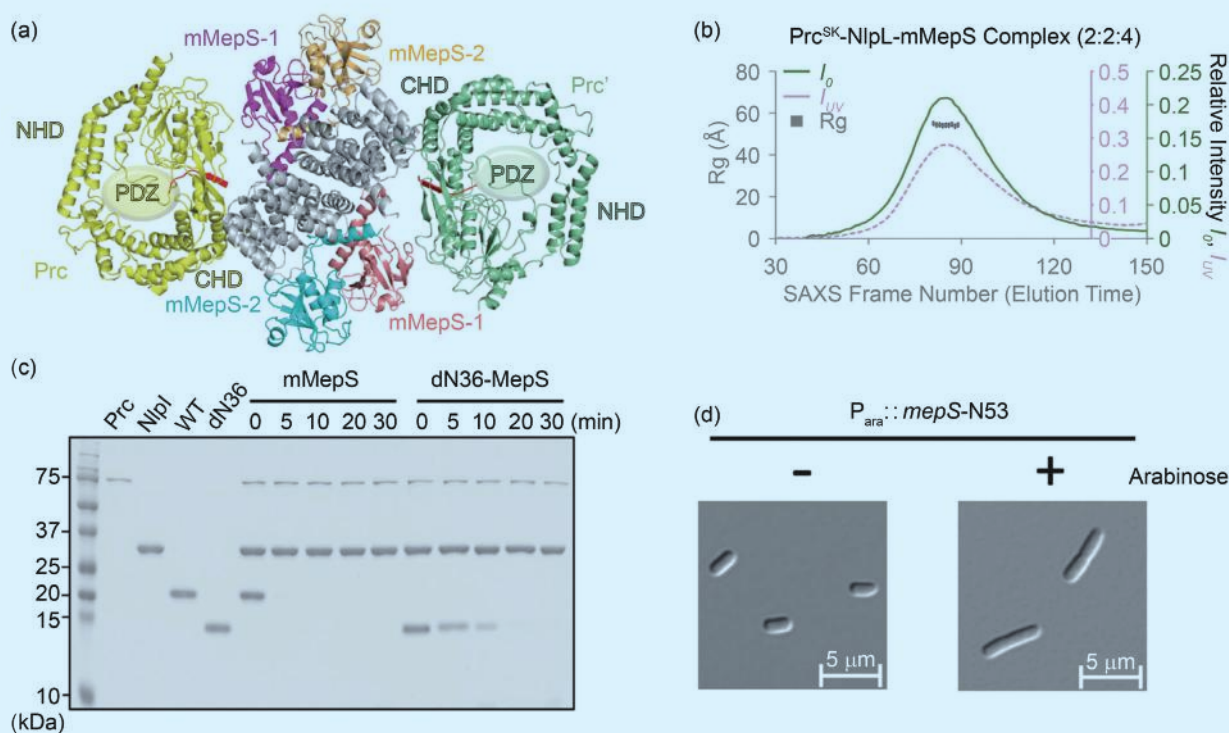


Fig. 2: (a) Overall structure of the Prc–NlpI–mMepS complex is shown, with NlpI in gray, Prc in yellow and pale green, and mMepS1 and mMepS2 in other colors. (b) A size-exclusion chromatogram of the Prc–NlpI–mMepS complex is presented, displaying the radius of gyration (R_g), zero-angle scattering intensity (I_0), and absorbance at 280 nm (I_{UV}). (c) *In vitro* degradation assays were conducted to examine the proteolytic activity of the Prc–NlpI system on mMepS and dN36-MepS. (d) The impact of overexpressing MepS-N53 on bacterial cell morphology was visualized using differential interference-contrast (DIC) microscopy. [Reproduced from Ref.1]

To understand how NlpI modulates the protein levels of MepS in the presence of the Prc protease, structural analysis was performed and revealed that the tail-specific protease Prc forms a 2:2:4 hetero-octameric complex with the adaptor protein NlpI and the endopeptidase mMepS (**Fig. 2(a)**). Size exclusion chromatography coupled with small-angle X-ray scattering (SEC-SAXS) experiments confirmed this 2:2:4 stoichiometry, ruling out the possibility that the observed stoichiometry was an artifact of crystal packing (**Fig. 2(b)**). Furthermore, the experimental data showed that the dN36-MepS significantly reduced its interaction with NlpI and decreased the degradation efficiency of mMepS by the Prc–NlpI system (**Fig. 2(c)**). This further confirms the important role of the N-terminal region in the recognition of mMepS by NlpI and the subsequent targeting of mMepS for Prc-mediated degradation. At the cellular level, overexpression of the N-terminal 53 residues of mMepS (MepS-N53) led to a significant change in bacterial morphology, resulting in the formation of long filamentous cells (**Fig. 2(d)**).

This study provides crucial structural insights into how the lipoprotein NlpI recruits and colocalizes the endopeptidase MepS, facilitating enhanced PG hydrolysis during bacterial cell growth. The binding of NlpI induces a disorder-to-order transition in the N-terminal region of MepS, promoting its dimerization and increasing enzymatic activity. Additionally, NlpI plays a pivotal role in targeting MepS for degradation by the protease Prc during

the stationary phase, thus regulating the cellular levels of MepS and maintaining cell wall integrity. These findings advance our understanding of the molecular mechanisms underlying bacterial cell wall remodeling and highlight the functional versatility of NlpI in coordinating PG synthesis and degradation. (Reported by Chun-Hsiang Huang, NSRRC)

This report features the work of Shiou-Ru Tzeng and her collaborators published in Nat. Commun. 15, 5461 (2024).

TPS 05A Protein Microcrystallography

TPS 07A Micro-focus Protein Crystallography

- Protein Crystallography
- Biological Macromolecules, Protein Structures, Life Science

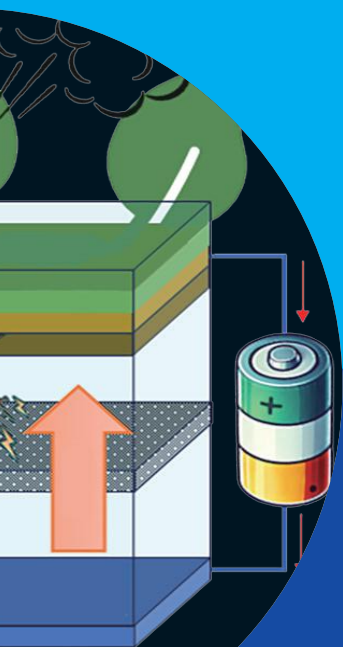
TPS 13A Biological Small-angle X-ray Scattering

- BioSAXS
- Structural Transitions of Macromolecules in Solution

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Energy Science



Synchrotron-based techniques remain indispensable in advancing energy storage and catalysis research despite significant progress in computational modeling and emerging *in situ* characterization methods. These techniques provide atomic-level insights into the structural and electronic properties of materials, thus enabling the development of high-performance batteries, electrocatalysts, and photocatalysts. At the NSRRC, several beamlines are dedicated to energy-related research, supporting breakthroughs in sodium ion batteries, zinc–air batteries, photocatalysis, methanol decomposition, and CO₂ electroreduction. Researchers have leveraged X-ray absorption spectroscopy, X-ray photoelectron spectroscopy, and X-ray diffraction to explore material transformations during operation, shedding light on reaction mechanisms and structural stability. **TPS 32A** and **TPS 44A** have been instrumental in characterizing the electronic structure of target materials and have provided crucial insights into defect-engineered nanosheets, which have optimized piezoelectric polarization in the field of sustainable electrocatalysis. **TLS 17C1** has enabled atomic-scale studies of Fe–Co and Ni–Fe bifunctional catalysts, thereby improving oxygen evolution and reduction reactions for zinc–air batteries. **TLS 09A2** and **TLS 24A1** have facilitated research on revealing the role of undercoordinated platinum sites in methanol decomposition. NSRRC’s state-of-the-art beamlines continue to support diverse scientific endeavors, enabling fundamental discoveries that drive advancements in sustainable-energy solutions.

In 2024, researchers utilizing NSRRC facilities achieved significant breakthroughs in energy storage and catalytic applications. This report highlights five outstanding contributions that advance battery technology and catalysis for sustainable-energy solutions. The first direction, led by Han-Yi Chen and Ji Liang, addressed a major limitation in current sodium ion transport technology within cathodes. Their work underscored the potential of layered structures, particularly layered metal oxide cathodes with complex elemental compositions, to enhance conductivity, stability, and overall performance. The second direction, headed by Shih-Yuan Lu and Yuan-Yao Li, introduced synergistic binary single-atom catalysts as air cathodes for high-performance and ultrastable zinc–air batteries. This dual-functional system significantly improved the efficiency and rechargeability of zinc–air and aluminum–air batteries, thus paving the way for flexible and sustainable energy storage solutions. For the third direction, Jih-Jen Wu developed dual-vacancy-engineered ZnIn₂S₄ nanosheets for hydrogen evolution. This study revealed a novel mechanism where low-frequency-vibration-induced piezoelectric polarization coupled with a static dipole field enhances photocatalytic hydrogen production. For the fourth direction, Meng-Fan Luo investigated undercoordinated Pt sites on the surface of layered PtTe₂, elucidating their role in methanol decomposition. This work provided crucial insights into catalytic activity modulation, which can lead to more efficient methanol-to-energy conversion technologies. Finally, Bing Joe Hwang’s study introduced a dual single-atom Ni/Cu catalyst that demonstrated highly selective CO₂-to-ethanol conversion. This research represents a major step forward in carbon capture and utilization, offering a promising route for sustainable fuel production. These studies underscore the vital role of NSRRC facilities in advancing battery technology and catalysis, which thereby contributes to the global pursuit of sustainable and efficient energy solutions. (by Hao Ming Chen)

Innovating Sodium-Ion Battery Cathodes: Enhancing Stability and Performance

Layered metal oxide cathode material engineering for sodium ion batteries holds significant potential for alkali-doped P2 and O3 high-capacity layered oxides.

With the rise in the global reliance on renewable energy sources, the demand for cost-effective, scalable energy-storage solutions has increased. Traditional lithium ion batteries (LIBs), while effective, face resource limitations due to lithium's limited supply and mining complexities. By contrast, sodium (Na) is the sixth most abundant element on Earth, making sodium ion batteries (SIBs) a cheaper, highly safe, and more sustainable alternative for large-scale energy-storage applications (Fig. 1).¹ The performance of SIBs is considerably influenced by the components of the cathode, anode, and electrolyte. However, achieving the energy densities and performance levels of SIBs comparable to LIBs has been challenging, necessitating extensive research into material compositions, structural arrangements, and electrochemical behaviors of Na ion systems. One of the focal points of recent research on SIBs has been the layered P2 and O3 structures in metal oxide cathode materials, which describe how sodium and transition-metal oxides are arranged within the cathode. In this context, "P" indicates sodium ions positioned at prismatic sites, while "O" indicates those located at octahedral sites based on the stacking sequences of oxide layers. The numbers 2 and 3 represent the number of Na ion layers per unit cell. Recently, most researchers have been exploring the ways to modify the layered structures and develop hybrid models to capitalize on each structure's strengths, aiming to suppress the adverse phase transition of layered oxide cathodes. For instance, inserting or doping alkali or transition-metal cations in the layered structure, designing a multi-element intention known as a high-entropy system, and switching between O3 and P2 phases during operation can, in theory, provide the high specific capacities for efficient energy storage and exhibit excellent cycling stability for reliable performance over multiple charge-discharge cycles. Variations of designs remain an area of active investigation, with potential implications for improving SIB performance. Employing a combination of advanced synchrotron X-ray techniques, including X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), and X-ray absorption spectroscopy (XAS), plays a key role in examining the structural and electrochemical properties of the layered cathode materials. These techniques allow for in-depth analysis of the materials at an atomic and molecular level, providing insights into their structural stability, chemical composition, and electronic behavior.

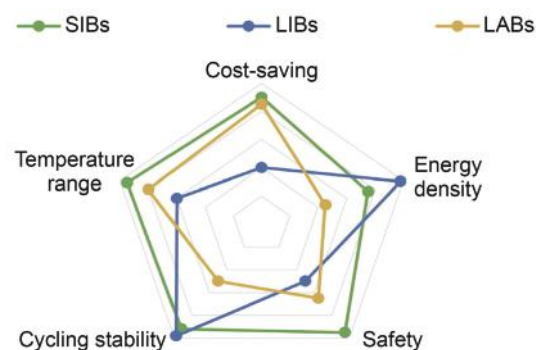


Fig. 1: Comparison of three established rechargeable battery technologies (SIBs, LIBs, and lead-acid batteries (LABs)) in the current market. [Reproduced from Ref. 1]

Most studies have tackled these challenges through innovative cathode material engineering for achieving high energy density and ensuring long-term cycling stability for SIBs. Han-Yi Chen (National Tsing Hua University) focused on a potassium (K)-doped P2-type layered $\text{Na}_{0.67}\text{Mg}_{1/3-x}\text{Cu}_x\text{Mn}_{2/3}\text{O}_2$ oxide cathode, which utilizes alkali-doped configuration to facilitate the movement of Na ions through larger diffusion pathways. This configuration enhances ionic conductivity and improves electrochemical performance.² K ions were selected for doping because of their larger ionic radius compared to Na, which thereby serves as a "pillar" that stabilizes the layered structure during Na ion intercalation and deintercalation. In particular, a doping level of $\text{K}_{0.05}$ showed the most promising results, achieving the highest specific capacity of 203 mAh/g, which represents a significant improvement compared to the undoped material's capacity of 185 mAh/g. This highlights the effectiveness of K doping in expanding ion diffusion pathways and enhancing material stability. *In situ* XRD analysis conducted at **TLS 01C2** provided insights into structural shifts within the cathode materials, revealing that moderate potassium doping (up to $\text{K}_{0.05}$) supports the P2 structure without causing unwanted phase transitions (Fig. 2, see next page). XAS analysis of transition-metal K-edges performed at **TPS 44A** and **TLS 17C1** further supported these findings, revealing that potassium doping did not alter the oxidation states of the transition metals. The structural integrity and charge balance were maintained within the material. This result is critical as it demonstrates that potassium serves as a stabilizer without disrupting the essential redox processes in the cathode material.

Another similar research conducted this year by Ji Liang (Tianjin University, China) explored a Li/Ti co-substitution strategy to address structural challenges in O3-type layered oxide cathodes for SIBs.³ By modifying the local electronic configuration of oxygen in $\text{NaNi}_{0.5}\text{Mn}_{0.5}\text{O}_2$ (NM) with an O3 structure, this approach effectively mitigates phase transitions and enhances structural stability, especially at high voltages, a common problem for layered oxides under high sodium extraction. This is a key development in stabilizing O3-phase materials through the specific elemental substitution. It demonstrates that Li/Ti co-substitution strengthens electrostatic bonding in the transition-metal layer and prevents adverse phase transitions, such as those from O3 to OP2, in a voltage range of 2.0–4.3 V. The Li/Ti-modified $\text{NaNi}_{1/9}\text{Ni}_{1/3}\text{Mn}_{4/9}\text{Ti}_{1/9}\text{O}_2$ (LNMT) cathode achieved impressive electrochemical performance with a capacity of 161.2 mAh g^{-1} at 1C and 80% retention after 100 cycles, underscoring the effectiveness of local electronic regulation in addressing phase instability (Fig. 3).

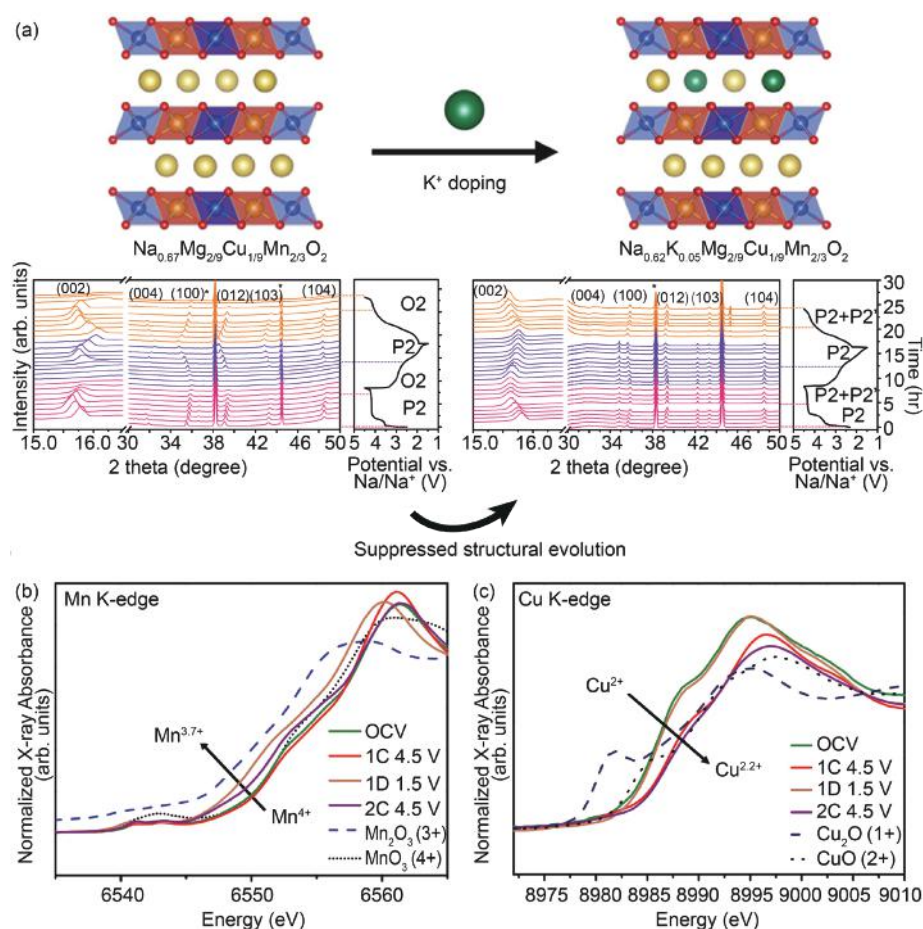


Fig. 2: Schematic K-doped P2 crystal structures and *in situ* XRD patterns of (a) $\text{K}_{0.00}$ and $\text{K}_{0.05}$. *In situ* XAS spectra of (b) Mn K-edge and (c) Cu K-edge of $\text{K}_{0.05}$ during cycling. [Reproduced from Ref. 2]

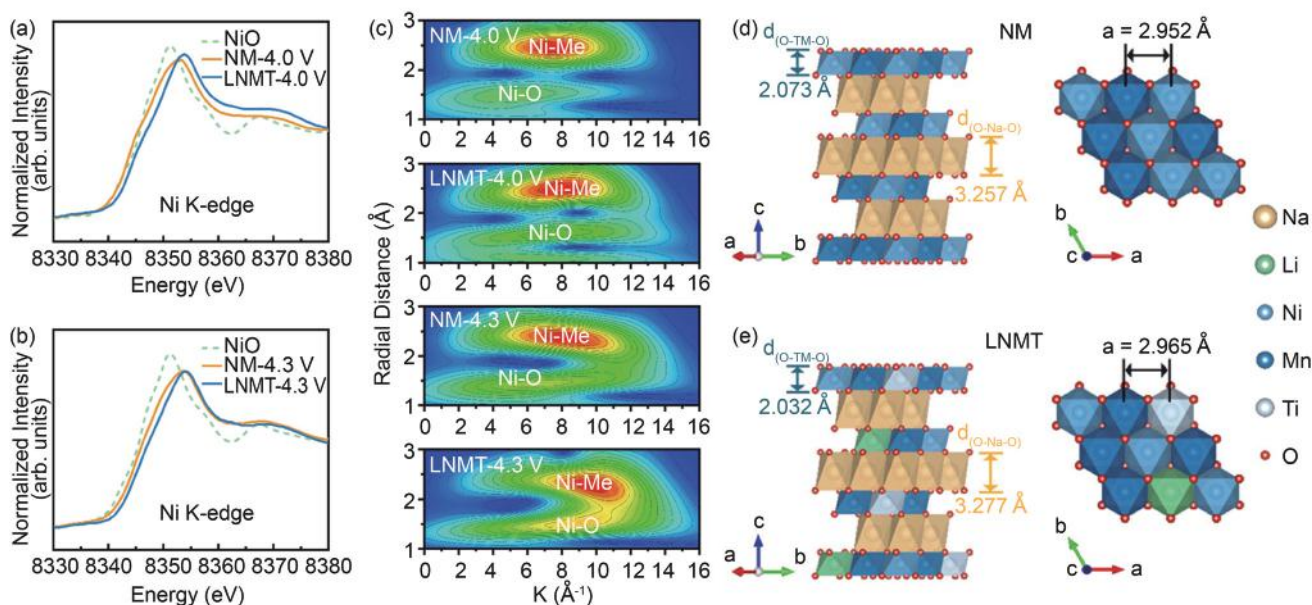


Fig. 3: (a, b) *Ex situ* XANES spectra at Ni K-edge of NM, LNMT, and NiO obtained at different charging conditions. (c) Wavelet transformation diagrams of the NM and LNMT under different voltage states. Schematic crystal structures and detailed lattice parameters of (d) NM and (e) LNMT. [Reproduced from Ref. 3]

XAS analysis conducted at **TPS 32A** revealed that LNMT promotes a stable electronic environment, mitigates drastic structural changes, and supports a more robust charge compensation mechanism. These findings highlight the importance of fine-tuning local electronic and atomic structures in designing high-capacity, high-voltage SIB cathodes. Together, these studies offer key insights into the effects of local structure on the electrochemical behavior of layered oxide cathodes, thereby paving the way for new developments in energy-storage solutions that offer enhanced capacity and cycle life.

In conclusion, while developing high-capacity and long-cycle-life SIBs remains challenging, addressing one of the major limitations of the current sodium ion transport technology in the cathodes highlights the potential of layered structures. Investigating the impact of these layered metal oxide cathodes with complex element compounds could potentially enhance the conductivity, stability, and overall performance. Applying *in situ* synchrotron X-ray techniques is essential for monitoring SIB behavior during operation. These methods provide valuable insights into changes that occur during charge–discharge cycles, enabling researchers to customize materials for improved performance. By combining the strengths of these configurations and leveraging advanced synchrotron X-ray techniques, the research lays the groundwork for more

efficient, durable, and economically viable SIBs. (Reported by Chi-Liang Chen)

This report features the work of Han-Yi Chen and her collaborators published in ACS Sustain. Chem. Eng. 12, 12795 (2024), and the work of Ji Liang and his collaborators published in ACS Nano 18, 18622 (2024).

TPS 32A Tender X-ray Absorption Spectroscopy
TPS 44A Quick-scanning X-ray Absorption Spectroscopy
TLS 17C1 EXAFS
TLS 01C2 X-ray Powder Diffraction

- XPS, XRD, XAS
- Energy Science, Chemistry, Materials Science, Condensed Matter

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Breathing Zinc–Air Batteries: Clean, Powerful, and Sustainable Energy Solutions

The development of advanced bifunctional catalysts is essential for attaining high-performance cathodes in rechargeable zinc-air batteries.

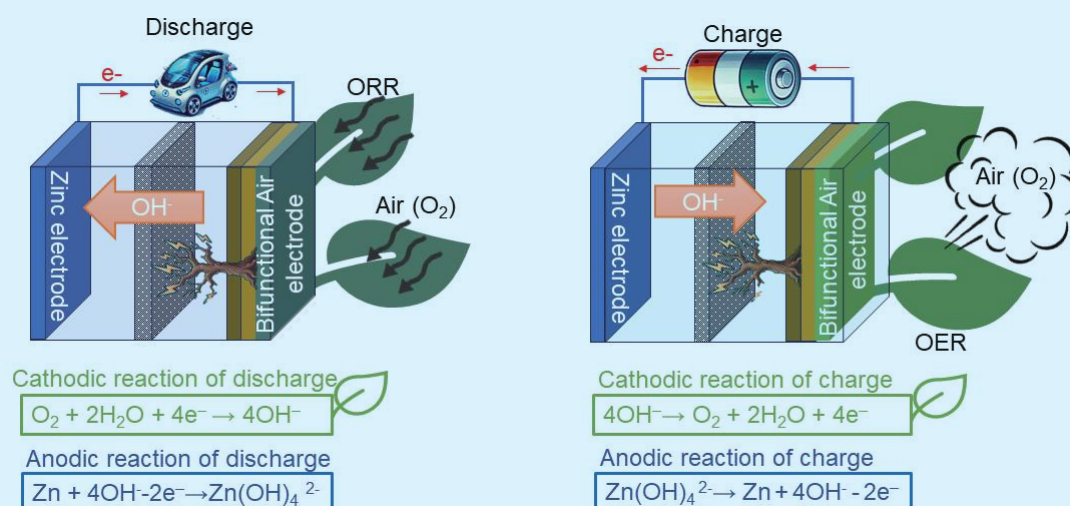


Fig. 1: A schematic configuration of zinc–air batteries and oxygen bifunctional electrocatalysts.

Zinc–air batteries (ZABs), a type of metal–air battery, generate electricity by combining zinc (Zn) with oxygen (O_2) from the air. At their core, these batteries consist of a Zn anode, a porous air cathode, and an electrolyte, which is often a strong alkaline solution. During discharge, zinc oxidizes at the anode, releasing electrons that travel through an external circuit to the cathode. Simultaneously, oxygen molecules from the air reduce at the cathode, combining with water and electrons to form hydroxide ions. These ions react with Zn ions in the electrolyte to form zincates (Fig. 1). This process releases energy that powers devices. ZABs are appealing for energy-intensive applications such as electric vehicles, grid energy storage, and portable devices. With a theoretical energy density potentially exceeding 1,000 Wh/kg, ZABs surpass lithium ion batteries. However, challenges in rechargeability and stability have historically hindered their commercial viability. Key limitations of ZABs include the rechargeability of the zinc anode and the sluggish oxygen reduction reaction (ORR) and oxygen evolution reaction (OER) kinetics at the cathode. Repeated charge–discharge cycles can lead to zinc dendrite formation, causing short circuits. In addition, reactions between zinc and the alkaline electrolyte can produce by-products that degrade performance. To address these issues, recent research has focused on improving the electrolyte, cathode catalysts, and zinc anode structure. Promising developments include the use of solid-state electrolytes to minimize side reactions and innovative anode designs to prevent dendrite formation. Particularly, enhancing cathode catalysts to improve ORR and OER efficiency is a critical area of research for bifunctional electrocatalysts. Synchrotron X-ray absorption spectroscopy (XAS), which includes X-ray absorption near edge structure (XANES) and extended X-ray absorption fine structure (EXAFS) analyses, provides the valuable insights. XAS examines the oxidation state, electronic properties, atomic coordination, and bond lengths of

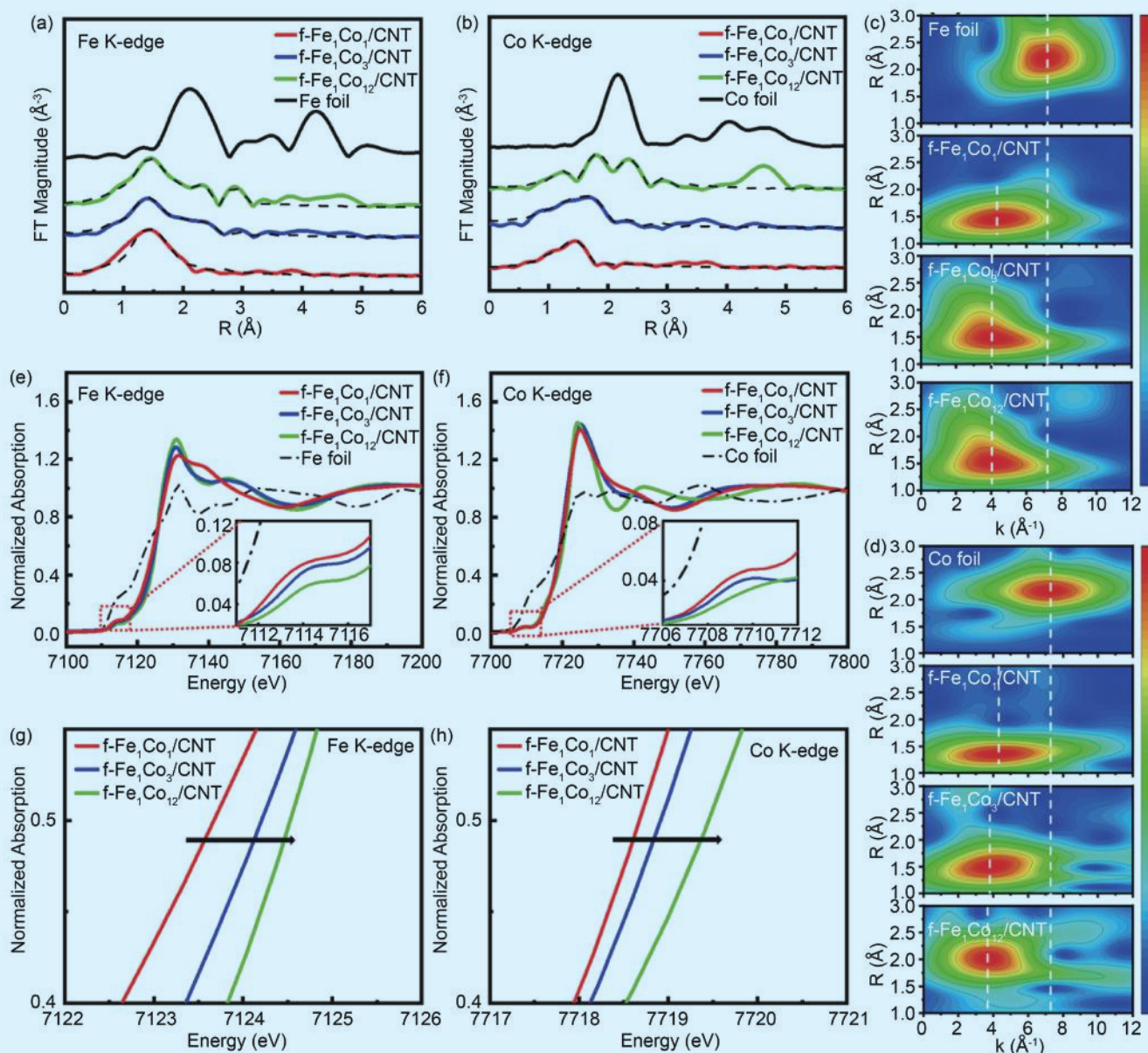


Fig. 2: EXAFS k^2 -weighted R-space results for (a) Fe K-edge and (b) Co K-edge, including fitted curves (black dashed lines). (c,d) k^2 -weighted WT images for the Fe and Co K-edges. (e,f) XAS spectra, with insets highlighting pre-edge peaks and (g,h) absorption edges. [Reproduced from Ref. 1]

catalyst atoms. This detailed understanding is essential for the rational design of more efficient catalysts by elucidating the relationships between the structure and properties, as it reveals the atomic structure and electronic properties. This analysis also sheds light on how these factors influence catalytic performance through a comprehensive analysis of the underlying mechanisms. Some ZABs have achieved impressive cycling stability and reaction efficiency this year, outperforming traditional and commercial Pt/C + IrO₂ catalysts. Therefore, if these innovations continue, ZABs could emerge as mainstream alternatives to lithium ion batteries in the near future.

Shih-Yuan Lu (National Tsing Hua University) investigated a binary Fe and Co single-atom catalyst (SAC) named f-Fe₁Co₁/CNT. This catalyst, which is synthesized *via* a formamide-assisted solvothermal approach and analyzed using XAS at **TLS 17C1**, anchors Fe and Co single atoms on nitrogen-doped carbon layers supported by carbon nanotubes (CNTs). This design achieves high atom loading and prevents aggregation, significantly enhancing ORR and OER efficiency in alkaline media. The f-Fe₁Co₁/CNT catalyst demonstrates superior ORR and OER activities and achieves the remarkable discharge peak power density and stability when paired with a carbon-paper-composited nickel foam (NF/CP) air cathode. XAS analysis, including EXAFS and wavelet-transform (WT) analysis, confirmed the changes in coordination environments and revealed synergistic activity from Fe and Co atoms, with Fe-N₄ sites identified as the primary active centers. These findings highlight the potential of SACs to achieve high catalytic activity, selectivity, and stability.

Furthermore, this year, noteworthy research conducted by Yuan-Yao Li (National Chung Cheng University) introduced a sophisticated bifunctional catalyst that integrates nickel-iron-layered double hydroxide (NiFe LDH), iron-cobalt dual single atoms (FeSACoSA), and iron-cobalt nanoalloy (FeCoAlloy) within a framework of nitrogen-doped carbon (NC) and CNTs. This combination maximizes the ORR and OER activities by leveraging the synergistic properties of its components, notably, the high density of ORR active sites provided by the FeCo alloy and FeSACoSA and the enhanced durability and OER efficiency contributed by NiFe LDH. The low ORR-OER potential gap of the catalyst and its implementation in a conductive, porous cathode structure deliver the outstanding performance in liquid-state and flexible ZABs. Such ZABs achieve impressive power densities, specific capacities, and cycling stability surpassing commercial benchmarks. **Figure 3** presents the results of XAS analysis conducted at **TLS 17C1**. The Co and Fe K-edge results revealed the presence of metal-metal bonds, confirming both metal-N coordination and the existence of an FeCo alloy within the material. Furthermore, the Ni K-edge spectra exhibited Ni-O and Ni-Ni bonds from NiFe LDH, confirming the successful coating of NiFe LDH on the material. These findings highlight the importance of both single-atom dispersion and synergistic interactions between the different active sites in achieving high catalytic efficiency in these binary SACs.

The development of ZABs highlights the potential of advanced catalyst-cathode configurations. Utilizing synchrotron XAS to analyze catalytic mechanisms can drive further advancements. With continuous improvements in bifunctional catalysts,

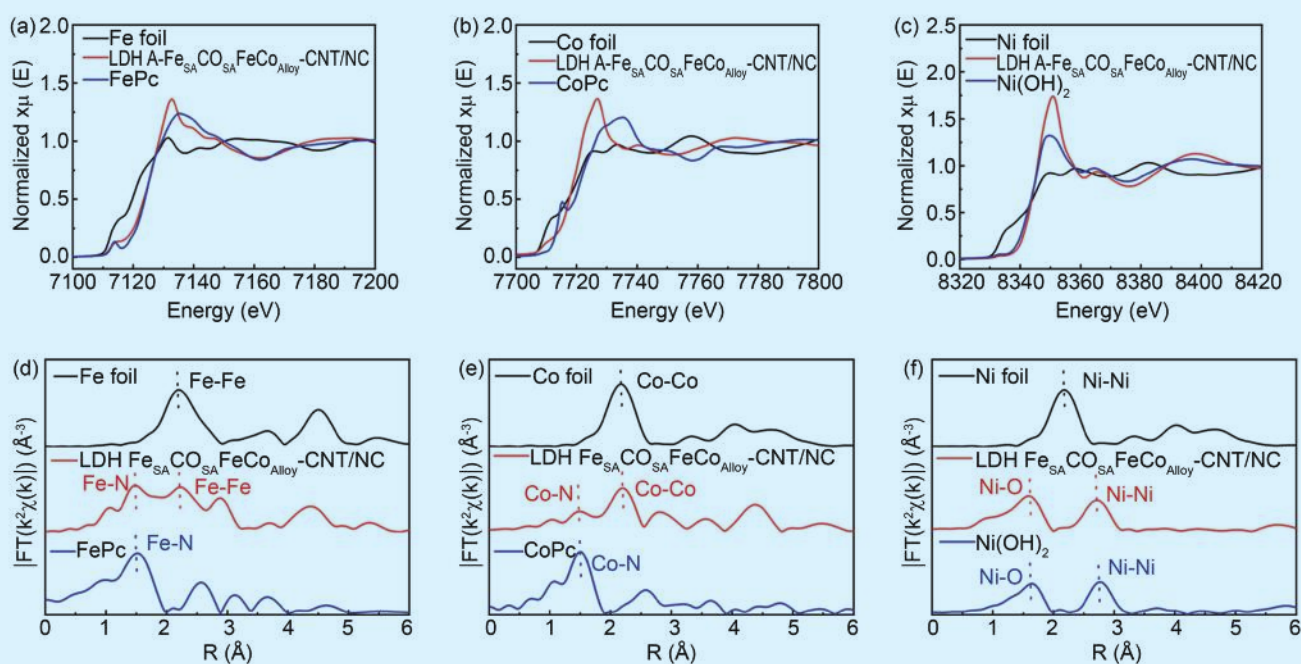


Fig. 3: XAS spectra of NiFe LDH A-FeSACoSA-FeCo Alloy CNT/NC. XANES spectra for (a) Fe K-edge, (b) Co K-edge, and (c) Ni K-edge. Fourier transform EXAFS spectra for (d) Fe K-edge, (e) Co K-edge, and (f) Ni K-edge. [Reproduced from Ref. 2]

ZABs may soon emerge as a viable solution for large-scale energy storage and transportation. Combining high energy density, eco-friendly materials, and reduced costs, ZABs hold promise as a cornerstone of sustainable energy solutions. (Reported by Chi-Liang Chen)

This report features the work of Shih-Yuan Lu and his collaborators published in Energy Storage Mater. 67, 103286 (2024), and the work of Yuan-Yao Li and his collaborators published in Nano Energy 121, 109236 (2024).

TLS 17C1 EXAFS

- XAS, WT
- Materials Science, Chemistry, Condensed-matter Physics

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Novel ZnIn₂S₄ Combines Piezopotential and Dipole Field for Hydrogen Technique

This study offers an in-depth examination of vacancy engineering in ZIS nanosheets, utilizing piezopotential and dipole fields to optimize photocatalytic performance.

Visible-light-driven photocatalysts often suffer from significant charge recombination due to their limited carrier diffusion lengths, which constrain their practical applications. A common strategy for mitigating this challenge is the development of heterostructure photocatalysts with built-in junction fields to promote charge separation. Alternatively, leveraging the dipole field within the internal bulk phase of photocatalysts has demonstrated considerable effectiveness in enhancing charge separation and transport. This dipole field arises naturally in photocatalysts with non-centrosymmetric crystal structures and can be introduced through the structural modifications. Piezoelectric polarization, a macroscopic built-in potential, is generated in piezoelectric crystals with non-centrosymmetric structures in response to applied mechanical stress. Piezo-photocatalysis synergistically integrates the piezoelectric effect with the light-harvesting capabilities of piezoelectric semiconductors, facilitating the separation and movement of photogenerated charge carriers through the macroscopic built-in potential field.

Jih-Jen Wu (National Cheng Kung University) and his coworkers recently investigated the influence of In- and S-vacancy concentrations on the photocatalytic activity of zinc indium sulfide (ZIS) nanosheets for hydrogen evolution reactions (HERs). Using X-ray absorption spectroscopy (XAS) measurements, including X-ray

absorption near edge structure (XANES) and extended X-ray absorption fine structure (EXAFS), conducted at **TPS 32A** and **TPS 44A**,¹ they gained deeper insights into the structural transformations of defective ZIS after hydrazine treatment. For clarity, the unmodified ZIS powder, as well as the modified ZIS powders that had been subjected to hydrazine treatment for 1 hour and 5 hours, are designated as ZIS-0, ZIS-1, and ZIS-5, respectively. **Figure 1(a)** presents the XANES spectra of the ZIS samples at the S K-edge, revealing a notable increase in sulfur vacancies and unoccupied states at sulfur sites following hydrazine treatment. **Figures 1(b) and 1(c)** illustrate the XANES and EXAFS spectra at the Zn K-edge for ZIS-0 and ZIS-5. The reduced coordination number and elevated electron density observed at Zn sites in ZIS-5 compared to ZIS-0 are attributed to the higher concentration of sulfur vacancies in ZIS-5. Similarly, **Figs. 1(d) and 1(e)** display the XANES and EXAFS spectra at the In K-edge for ZIS-0 and ZIS-5. Because of the contrasting trends in electron density changes driven by sulfur and indium vacancies, it was determined that sulfur vacancies have a more pronounced effect on the electron density at the indium sites in ZIS-5 than do indium vacancies, resulting in increased electron density at these sites compared to ZIS-0. Additionally, ZIS-5 exhibits a slightly diminished sulfur coordination environment relative to ZIS-0, which is attributed to a higher sulfur vacancy concentration post-treatment. The atomic arrangement of sulfur and indium vacancies within

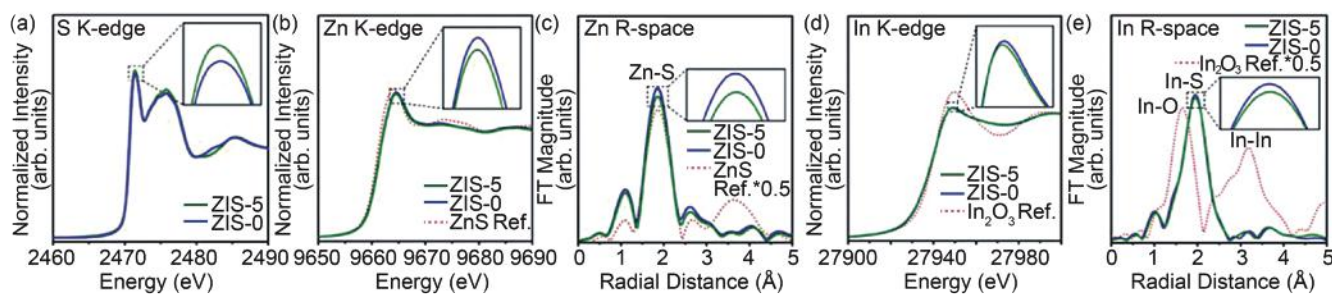


Fig. 1: (a) Normalized S K-edge XANES spectra for ZIS-0 and ZIS-5. (b) Normalized Zn K-edge XANES spectra and (c) Fourier transformed (FT) EXAFS spectra in R-space of ZIS-0, ZIS-5, and ZnS reference. (d) Normalized In K-edge XANES spectra and (e) FT EXAFS spectra in R-space of ZIS-0, ZIS-5, and In_2S_3 reference. [Reproduced from Ref. 1]

the ZIS structure plays a pivotal role in facilitating rapid charge transfer and effective charge separation, ultimately enhancing hydrogen production. This investigation meticulously explores how sulfur and indium vacancies improve charge dynamics and hydrogen evolution rates. By integrating the experimental data with multi-scale simulations, the study demonstrates how these vacancies optimize hydrogen production, offering valuable insights into the mechanisms by which structural vacancies in ZIS influence its electrochemical performance, particularly in hydrogen generation applications.

In summary, defect engineering of ZIS nanosheets with varying levels of indium and sulfur vacancies was successfully realized through hydrazine post-treatment of hydrothermally synthesized ZIS nanosheets. The photocatalytic HER rates show a positive correlation with the combined concentrations of indium and sulfur vacancies in the ZIS nanosheets. This study provides

a detailed exploration of vacancy engineering in ZIS nanosheets, leveraging piezoelectric polarization in conjunction with the dipole field to enhance photocatalytic efficiency. (Reported by Yan-Gu Lin)

This report features the work of Jih-Jen Wu and his coworkers published in Adv. Mater. 36, 2403228 (2024).

TPS 32A Tender X-ray Absorption Spectroscopy TPS 44A Quick-scanning X-ray Absorption Spectroscopy

- XAS
- Materials Science, Chemistry, Condensed-matter Physics, Environmental and Earth Science

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Defect-Engineering for Selective Methanol Decomposition

Undercoordinated Pt sites in PtTe_2 are crucial in facilitating methanol decomposition and promoting formaldehyde production.

Two-dimensional transition metal dichalcogenides (TMDs) have garnered significant attentions in catalysis due to their unique electronic properties, high surface area, and tunable surface chemistry. Unlike conventional noble metal catalysts, which exhibit disadvantages such as high costs and susceptibility to poisoning, TMDs are promising alternatives owing to their structural versatility and defect engineering capabilities.^{1,2} However, the basal planes of pristine TMDs are often inert, limiting their catalytic utility. Surface defects, particularly chalcogen vacancies, introduce the undercoordinated metal sites that can serve as highly active catalytic centers, significantly altering their chemical reactivity. Among TMDs, platinum telluride (PtTe_2) is of particular interest due to its intrinsic metallic nature, which provides superior conductivity compared to semiconducting TMDs such as MoS_2 or WS_2 . A research team led by Meng-Fan Luo (National Central University), Jyh-Pin Chou (National Changhua University of Education), and Chun-Liang Lin (National Yang Ming Chiao Tung University) is investigating the catalytic properties of PtTe_2 with engineered Te vacancies. Their study focuses on PtTe_2 , a metallic group-10 TMD, emphasizing the role of undercoordinated Pt (Pt_{uc}) sites at Te vacancies in methanol decomposition, which is a crucial reaction for hydrogen production and direct methanol fuel cells. To explore the catalytic behavior of PtTe_2 , controlled Ar⁺

bombardment was employed to selectively remove surface Te atoms, generating Pt_{uc} sites while maintaining the structural integrity of the underlying Pt lattice. **Figure 1(a)** presents the scanning tunneling microscopy (STM) images before and after ion bombardment, confirming the introduction of single and multi-Te vacancies. **Figure 1(b)** further characterizes the defect structure through synchrotron-based photoelectron spectroscopy (PES), which reveals a shift in the Pt 4f binding energy. This shift indicates a lower oxidation state at Pt_{uc} sites, which enhances their reactivity. To assess the influence of Pt_{uc} concentration on catalytic performance, **Figs. 1(c) and 1(d)** quantify the methanol conversion probability as a function of vacancy density. At low Pt_{uc} concentrations ($\leq 10\%$), methanol conversion exceeds 90%, with high selectivity toward CH_2O and CH_4 . As Pt_{uc} concentration increases (10%–20%), selectivity shifts, favoring C–O bond scission over dehydrogenation, which is further validated by near-ambient-pressure photoelectron spectroscopy (NAP-PES) and near-ambient-pressure mass spectroscopy results.

Methanol adsorption and decomposition were systematically investigated using synchrotron-based *in situ* NAP-PES and near-ambient pressure mass spectrometry (NAP-MS). As shown in **Fig. 2(a)**, the NAP-PES spectra for pristine PtTe_2 reveal that methanol does not undergo significant decomposition, confirming its catalytic inertness. In contrast, on defect-rich PtTe_2 , the spectral intensity of methanol-related species (CH_3OH) decreases with increasing temperature, indicating the progressive methanol conversion. Concomitantly, peaks corresponding to CH_2O (formaldehyde) and CH_x species are observed, which demonstrate that Pt_{uc} sites actively promote methanol dehydrogenation. NAP-MS results further confirm these findings by monitoring the gaseous products formed during the reaction (**Fig. 2(b)**). On Pt_{uc} -enriched PtTe_2 , CH_2O (formaldehyde) and CH_4 (methane) are the dominant products, whereas only trace amounts of CO and CO_2 are detected.

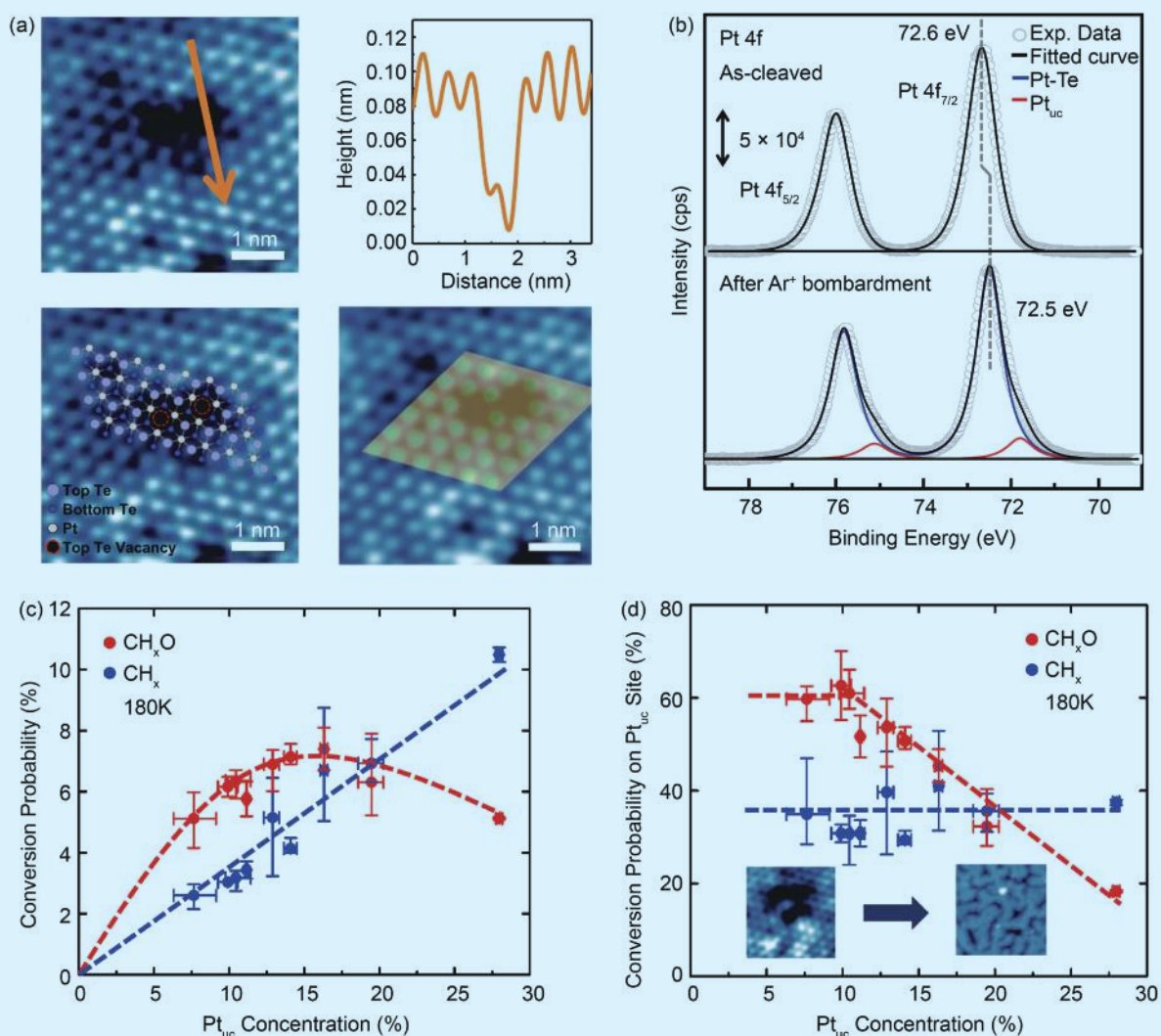


Fig. 1: (a) High-resolution image of two single-Te vacancies, the line profile across a single-Te vacancy, the overlap of the two single-Te vacancies model with the imaged ones and the overlap of the STM image with the DFT-simulated one produced based on the vacancy model. (b) PES spectra of Pt 4f core level from layered PtTe_2 as cleaved and bombarded by Ar^+ (0.5 keV, 3 mins). (c,d) Varied reaction probabilities of methanol adsorbed on a PtTe_2 surface with Pt_{uc} sites. Probabilities of conversion to CH_xO^* (red circles) and CH_x^* (blue) of monolayer methanol adsorbed on a PtTe_2 surface, and those on Pt_{uc} sites as a function of the concentration of the surface Pt_{uc} . [Reproduced from Ref. 3]

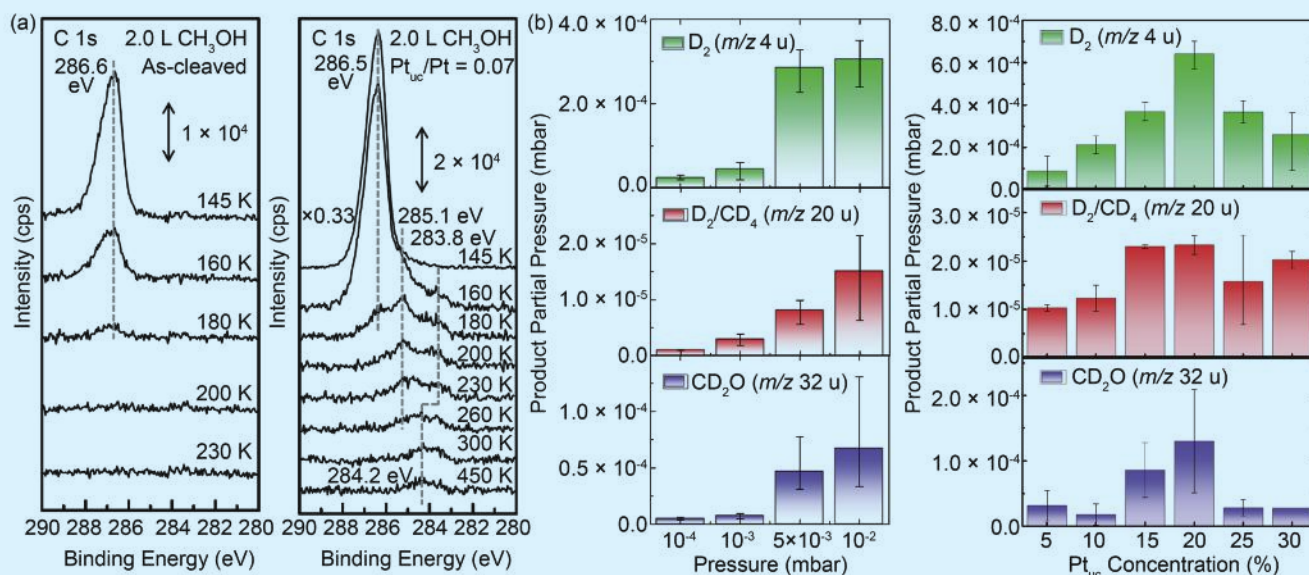


Fig. 2: (a) PES spectra of C 1s core level for methanol adsorbed on as-cleaved and Ar⁺-bombarded PtTe₂ at 145 K and annealed stepwise to selected temperatures. (b) Production of D_{2(g)} (*m/z* 4 u), D₂O(_g)/CD_{4(g)} (*m/z* 20 u), and CD₂O(_g) (*m/z* 32 u) from PtTe₂ at 300 K, as a function of methanol-d₄ pressure (Pt_{uc}/Pt ratio 0.10) and Pt_{uc} concentration (10⁻² mbar). [Reproduced from Ref. 3]

This highlights the selective nature of Pt_{uc} sites, which favor formaldehyde formation over complete C–O bond scission, reducing unwanted CO poisoning, which is an issue commonly observed for conventional Pt catalysts. Additionally, the reaction selectivity shifts with increasing Pt_{uc} concentration. At moderate defect densities (~10%), methanol decomposition predominantly follows the dehydrogenation pathway, producing CH₂O and CH₄. However, at higher Pt_{uc} densities (>10%), the C–O bond cleavage pathway becomes more competitive, leading to increased CH₄ and H₂ formation while slightly reducing CH₂O selectivity. This trend is consistent with density functional theory (DFT)-calculated reaction barriers, which suggest that Pt_{uc} sites lower the activation energy for selective dehydrogenation but can also facilitate C–O scission at higher defect concentrations.

In summary, this study establishes PtTe₂ with undercoordinated Pt sites as a highly efficient catalyst for methanol decomposition, offering superior activity and selectivity compared to conventional Pt catalysts. The unique triangular coordination and oxidation state variations of Pt_{uc} sites play a crucial role in tuning the reaction pathways. By integrating advanced synchrotron-based PES, *in situ* NAP-PES/NAP-MS at TLS 09A2 and TLS 24A1, and theoretical modeling, this work provides fundamental insights into defect engineering in TMDs. It lays the foundation for designing next-generation catalysts for methanol reforming, hydrogen production, and direct methanol fuel cells. (Reported by Hao Ming Chen, National Taiwan University)

This report features the work of Meng-Fan Luo, Jyh-Pin Chou and Chun-Liang Lin published in Nat. Commun. 15, 653 (2024).

TLS 09A2 Spectroscopy

TLS 24A1 XPS, UPS, XAS, APXPS

- *In situ* NAP-XPS
- Materials Science, Thin-film Chemistry

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Dual-Atom Catalyst for Selective CO₂ Reduction

A dual-atom NiCu catalyst achieves record-high selectivity for CO₂-to-ethanol conversion.

The electrochemical reduction of carbon dioxide (CO₂) into valuable multi-carbon fuels is a promising strategy for mitigating CO₂ emissions while producing sustainable chemical feedstocks.¹ However, achieving high selectivity and efficiency for ethanol production remains a major challenge because of the difficulty of C–C coupling and the competitive formation of C1 products. Copper-based catalysts have demonstrated the potential for facilitating C–C coupling, but their selectivity for ethanol is often low, requiring significant overpotentials.^{1,2}

To address these challenges, the teams led by Bing Joe Hwang (National Taiwan University of Science and Technology, NTUST), Wei-Nien Su (NTUST), and Meng-Che Tsai (National University of Tainan) demonstrated a dual single-atom catalyst (SAC) system featuring atomically dispersed Ni and Cu sites (NiCu-SACs/N-C), which synergistically enhance ethanol selectivity and catalytic efficiency. **Figure 1(a)** illustrates the rational design and synthesis of NiCu-SACs/N-C prepared *via* hydrothermally assisted pyrolysis using zeolitic imidazolate frameworks.

The synthetic strategy ensures the atomic dispersion of Ni and Cu in a nitrogen-doped carbon matrix, creating abundant accessible active sites. To further elucidate the role of cooperative dual active sites, control experiments in CO-saturated electrolytes were conducted. The results confirm that Ni sites generate labile CO intermediates, which diffuse to adjacent Cu sites for C–C coupling, thereby forming ethanol. A key highlight of this work is the use of Ni–Cu cooperative heteroactive sites, which uniquely facilitate CO₂ reduction to ethanol with an unprecedented Faradaic efficiency (FE) of 92.2% at –0.6 V vs. reversible hydrogen electrode (RHE) (**Fig. 1(b)**). To the authors' knowledge, this represents the highest selectivity reported to date for direct electrochemical CO₂-to-ethanol conversion. The dual-atom catalyst also exhibits the lowest onset potential for ethanol production at –0.4 V vs. RHE, surpassing the performance of single-atom Cu or Ni catalysts. This exceptional performance is attributed to the cooperative entanglement of adjacent Ni and Cu sites, where CO generated on Ni–N₃ sites is efficiently transferred to Cu–N₄ sites for C–C coupling, ultimately leading to ethanol formation.

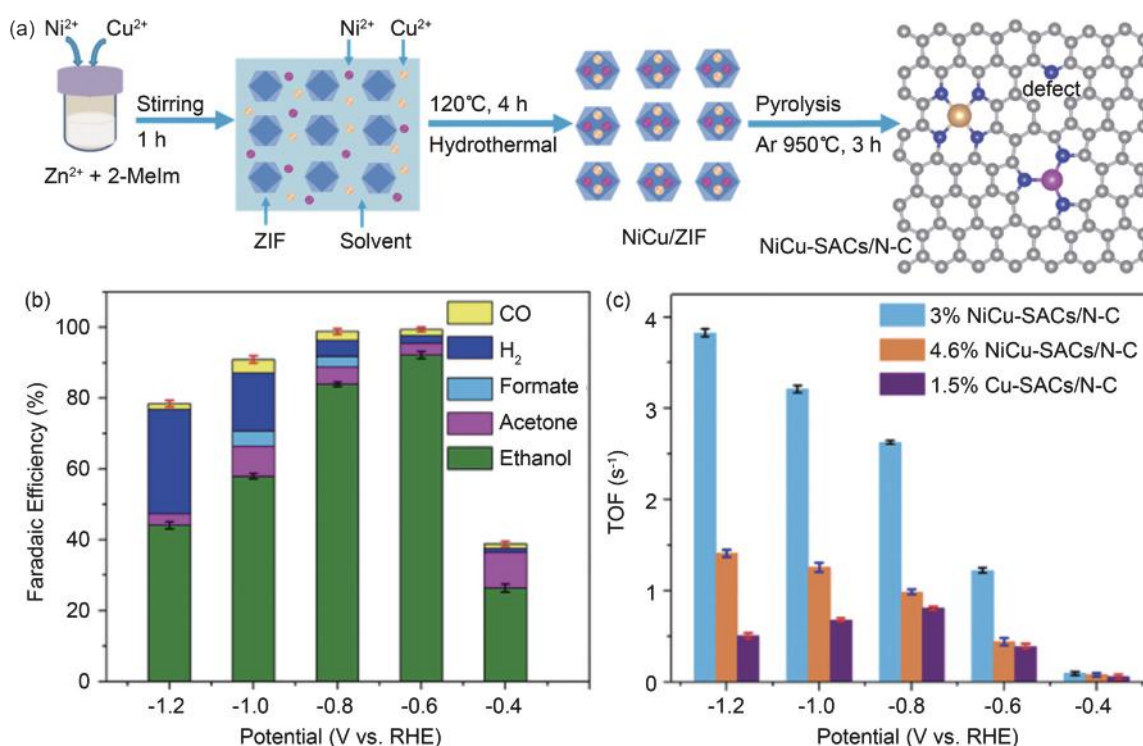


Fig. 1: (a) Schematic representation of the synthesis of dual SACs *via* hydrothermally assisted pyrolysis method. (b) FEs and product distributions over the catalysts of 3% NiCu-SACs/N-C electrodes at different potentials. Error bars indicate the standard deviation. (c) TOF of ethanol over NiCu-SACs/N-C hybrids with different metal loadings, compared with Cu-SACs/N-C at different applied potentials. [Reproduced from Ref. 3]

Figure 1(c) highlights the turnover frequency (TOF) of CO₂-to-ethanol conversion, with the NiCu-SACs/N-C catalyst exhibiting a TOF four times higher than single-atom Cu catalysts. The cooperative synergy between Ni and Cu is also evident in the significantly enhanced electrochemical active surface area and reduced charge transfer resistance (R_{ct}), indicating more efficient electron and mass transport. The computational modeling further supports the experimental findings by demonstrating that the energetically favorable C–C coupling step occurs at the Cu–N₄ sites, which are enhanced by neighboring Ni–N₃ sites. At greater negative potentials, the formation of Cu clusters increases the energy barrier for C–C coupling, leading to a decline in ethanol selectivity. This mechanistic insight explains the volcano-shaped ethanol FE trend observed in the electrochemical studies.

The role of *in situ* X-ray absorption spectroscopy (XAS) is crucial in unraveling the mechanistic insights of this dual-site catalyst. **Figure 2** presents *in situ* extended X-ray absorption fine structure (EXAFS) and X-ray absorption near-edge structure (XANES) analyses. The results reveal a key structural transformation during CO₂ reduction. Notably, while Ni remains in its single-atom Ni–N₃ coordination environment throughout the reaction, Cu undergoes dynamic restructuring, forming potential-induced Cu clusters at high negative potentials. These dynamic Cu clusters play a critical role in modulating catalytic activity, but excessive clustering leads to decreased

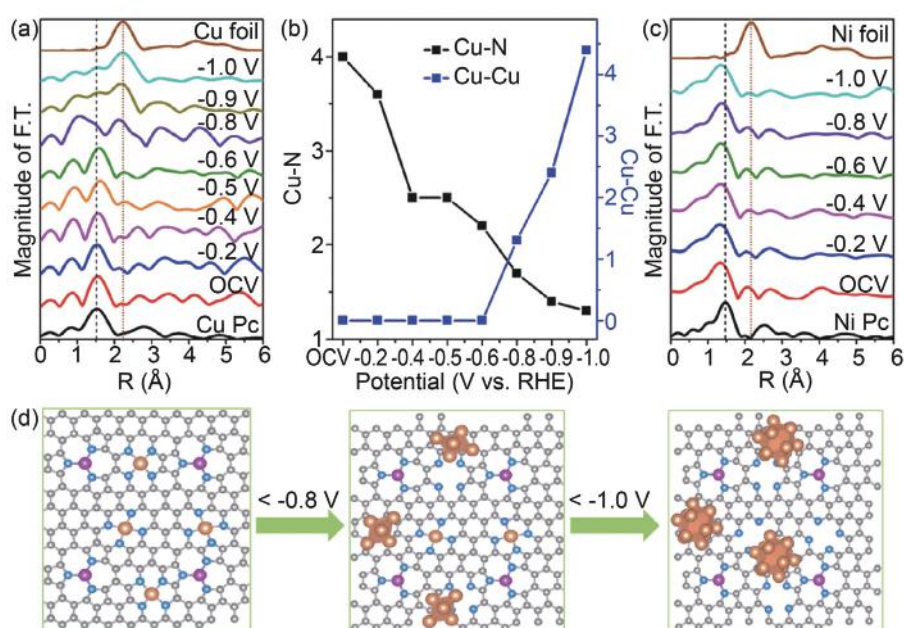
ethanol selectivity. This observation, which is supported by density functional theory (DFT) calculations, confirms that C–C coupling occurs preferentially on atomically dispersed Cu–N₄ sites rather than Cu clusters, explaining the decline in ethanol FE at highly negative potentials.

In summary, this study establishes NiCu-SACs/N-C as a groundbreaking dual-site catalyst for CO₂-to-ethanol conversion, achieving record-high FE and low onset potential. The findings underscore the importance of cooperative single-atom site engineering and the synergistic role of Ni and Cu in promoting selective C–C coupling. The insights gained from *operando* XAS at **TPS 44A**, electrochemical analysis, and DFT modeling pave the way for designing next-generation electrocatalysts for carbon-neutral fuel production. This work provides a scalable and sustainable strategy for high-selectivity electrochemical CO₂ conversion, contributing to advancements in renewable energy and carbon capture technologies. (Reported by Hao Ming Chen, National Taiwan University)

This report features the work of Bing Joe Hwang, Wei-Nien Su and Meng-Che Tsai published in Appl. Catal. B-Environ. 358, 124420 (2024).

TPS 44A Quick-scanning X-ray Absorption Spectroscopy

- *In situ* XAS
- Materials Science, Electrocatalyst

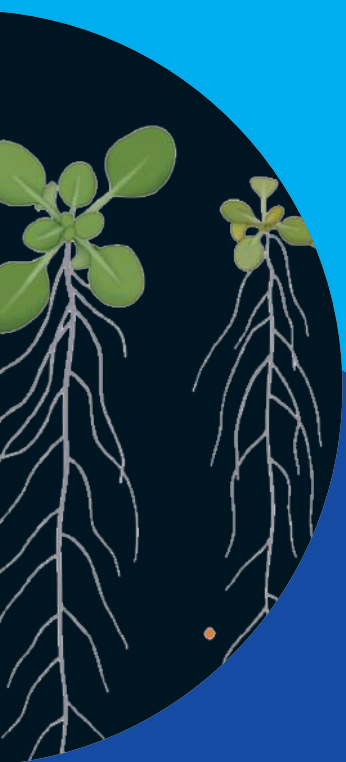


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Fig. 2: (a) *In situ* EXAFS spectra and (b) the corresponding average coordination number for Cu–N and Cu–Cu shells of the Cu K-edge for 3% NiCu-SACs/N-C hybrid recorded during the CO₂ electrolysis at different applied potentials in CO₂-saturated 0.5 M KHCO₃. Data for CuPc and Cu foil are included for reference. (c) *In operando* EXAFS spectra of Ni K-edge for 3% NiCu-SACs/N-C hybrid recorded during the CO₂ electrolysis at different applied voltages. Data for NiPc and Ni foil are included for reference. (d) The structural models of atomically distributed Ni and Cu sites, as well as the potential-induced dynamic Cu clusters from Cu single atoms, as suggested by *in operando* analysis during the CO₂ electrolysis. [Reproduced from Ref. 3]

Environmental and Earth Sciences



Plants and their environment exhibit complex interactions. Through their relationship with soil, plants absorb nutrients and heavy metals, improving soil quality and fixing carbon. By absorbing and transpiring water, plants regulate the water cycle and purify water. In their interaction with air, plants absorb carbon dioxide for photosynthesis, regulate gas exchange, and reduce air pollution. Plants also form symbiotic relationships with microorganisms to enhance nutrient utilization and disease resistance, while interactions with animals support pollination and seed dispersal. Additionally, plants mitigate the greenhouse effect by absorbing carbon and adapting to extreme climates, thereby maintaining ecological balance. In 2024, research on plant–environment interactions using synchrotron radiation technologies at the NSRRC achieved significant progress.

Plants' ability to absorb and accumulate toxic substances makes them essential indicators for evaluating soil conditions. Understanding their absorption mechanisms is crucial for preventing harmful toxins from entering crops and improving soil health. Shan-Li Wang at National Taiwan University used X-ray absorption near-edge structure (XANES) technology to explore how soil conditions affect molybdenum (Mo) chemical forms and their absorption by rice.

Kuo-Chen Yeh at Academia Sinica applied the X-ray Nanoprobe beamline for X-ray fluorescence (XRF) analysis of *Arabidopsis*, mapping element distribution under varying soil conditions. This provided insights into element absorption and transport mechanisms.

Samir Gamil Mohammad Al-Solaimani at King Abdulaziz University (Saudi Arabia) used XANES and XRF imaging to study the effects of humic acid (HA) on nutrient availability, toxic elements, and plant growth. The results showed that HA improves soil fertility and reduces the absorption of toxic metals, offering practical solutions for enhancing soil and crop health.

Yu-Ting Liu at National Chung Hsing University focused on using plants and their derivatives to remove toxic metals from the environment. They demonstrated that thiol-functionalized biochar (BC) effectively removes hexavalent chromium [Cr(VI)] and uncovered how thermophilic acidic microalgae *Cyanidiales* detoxify trivalent arsenic [As(III)]. These findings highlight BC's effectiveness in Cr(VI) removal and *Cyanidiales*'s efficiency in As(III) detoxification, offering sustainable solutions *via* materials science and bioremediation.

Yu-Min Tzou at National Chung Hsing University investigated phosphorus regulation in soil to promote plant growth. His study examined citric acid adsorption on HA–ferric hydroxide co-precipitates (HAFHCPs) and competition between citric acid and phosphate (P) at different carbon-to-iron (C/Fe) ratios. Results revealed that HAFHCPs regulate phosphorus availability, which is influenced by C/Fe ratios, HA composition, and citric acid presence, as observed by XANES technology. Optimizing phosphorus availability is vital for sustainable phosphorus resource use.

These studies advance our understanding of plant–environment interactions and soil remediation strategies, providing critical insights for ecosystem sustainability. (by Chun-Chieh Wang)

Assessing Soil Conditions Through Plant Accumulation of Toxic Substances

Research reveals how absorption of soil toxins by plants can indicate soil health, guiding effective management and remediation strategies.

Plants can serve as the direct indicators of soil environmental conditions because they readily absorb and accumulate toxic substances from the soil. By studying the mechanisms through which plants accumulate chemical or toxic substances, we may prevent crops from taking up these harmful substances. Additionally, analysis of plants may be used to evaluate the effectiveness of soil remediation. Here, we present the research findings from 2024 conducted at the NSRRC, which focused on how plants accumulate toxic substances from the soil and how this process can be used to assess the effectiveness of soil

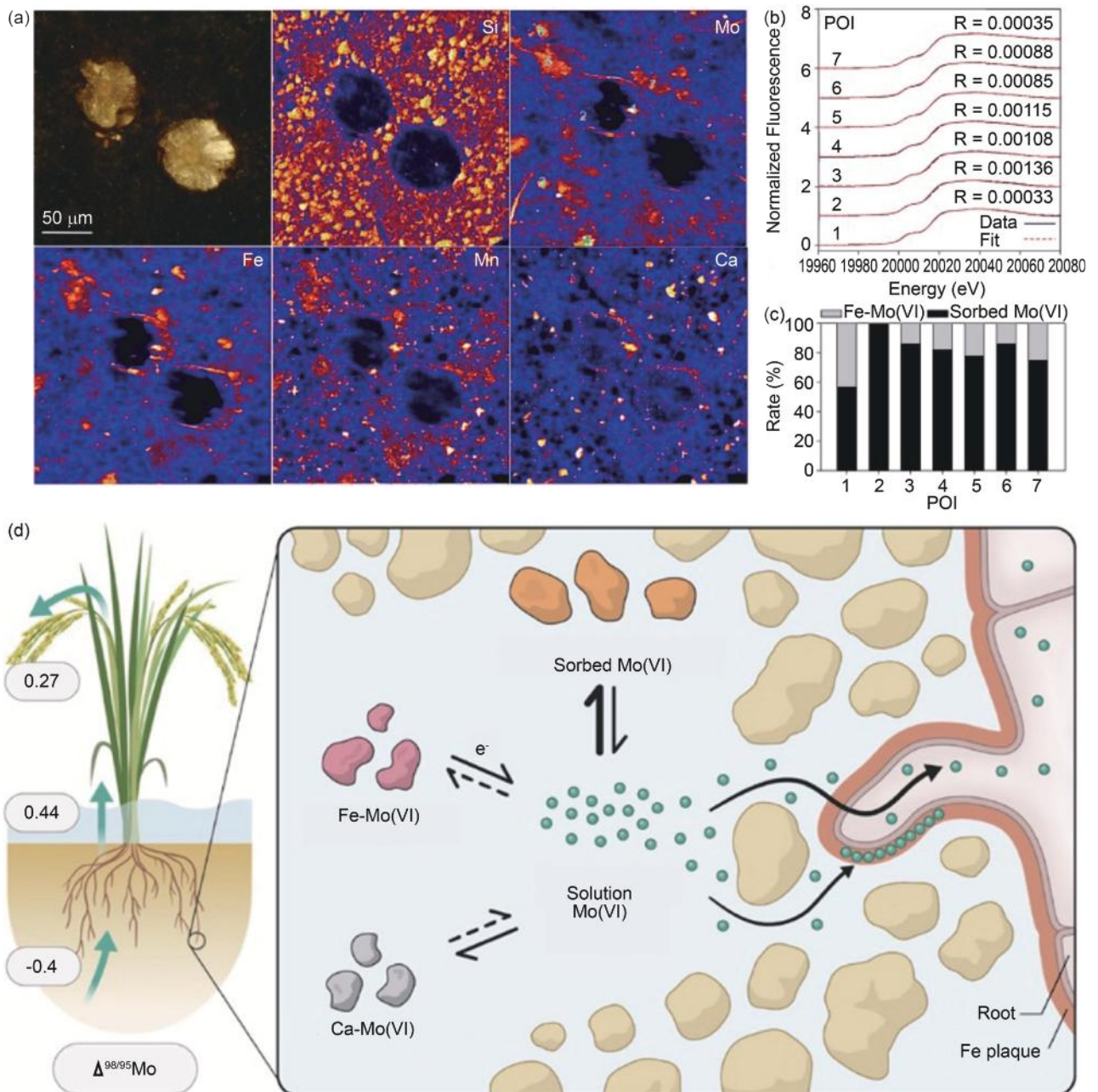


Fig. 1: (a) Microscopic view of rice roots, and spatial distributions of Si, Mo, Fe, Mn, and Ca; (b) μ -XANES spectra and (c) linear combination fits of the points of interest (POIs) indicated in (a). (d) Mechanism of rice uptake of soil molybdenum. [Reproduced from Ref. 1]

modification. With these discoveries, we aim to deepen our understanding of the interactions between plants and soil and to develop more effective soil management and remediation strategies.

Chemical Speciation and Uptake of Molybdenum by Rice

Molybdenum (Mo), a vital micronutrient for organisms, facilitates the metabolism of nitrogen, carbon, and sulfur and is crucial in enzyme catalysis. Although generally present in low concentrations in the Earth's crust, Mo levels can be elevated near industrial areas, potentially contaminating soil and accumulating in crops. This poses the health risks, including livestock poisoning and, in humans, conditions such as infertility and gout-like symptoms.

Rice, a major Mo dietary source for humans, often grows in submerged conditions that enhance Mo solubility and availability. This availability is further influenced by continuous flooding, which contrasts with the reduced Mo uptake seen in alternating wet–dry cycles. Key factors include soil redox processes driven by microbial activity and the dissolution of iron hydroxides, which affect the mobility of Mo compounds.

Shan-Li Wang (National Taiwan University) and his co-workers utilized X-ray absorption near edge structure (XANES) at **TPS 44A** and **TLS 16A1** and other microanalytical techniques to investigate how soil conditions affect Mo chemical forms and uptake in rice (**Fig. 1**). Their findings highlight the strong associations between Mo and Fe in the rice rhizosphere, which are facilitated by Fe plaques on root surfaces. This association promotes Mo dissolution/desorption, which is crucial for root absorption and subsequent transport to plant shoots, though only minimal amounts reach the grains.

The study reveals that Mo can accumulate significantly in rice without apparent toxicity, even in contaminated soils, raising concerns about Mo levels in consumed rice. Advanced studies in soil solution chemistry and Mo speciation provide the deeper insights into the complex dynamics of Mo availability and uptake, influencing the future research directions, particularly in Mo isotope fractionation. This study provides further understanding of the mechanisms affecting Mo's behavior in soil and its broader environmental and health impacts.

Physiology and Molecular Basis of Thallium Toxicity and Accumulation in *Arabidopsis thaliana*

Thallium (Tl) is a heavy metal with extensive applications across several industries, including chemical, pharmaceutical, optical, electronics, energy, and aerospace, as well as in superconducting materials and high-energy physics. Despite its limited annual production—approximately 10 tons globally—industrial processes inadvertently release an estimated 2,000 to 5,000 tons into the environment each year. This can significantly elevate Tl concentrations in contaminated soils, with well above the typical background level of less than 1 mg/kg found in most uncontaminated soils. Recognized for its extreme toxicity, Tl has been classified as a priority pollutant, which necessitates thorough research into its environmental impact, exposure routes, and toxicity.

Tl is particularly concerning because of its ability to be readily absorbed by plants, where it can disrupt potassium (K)-dependent biological processes and accumulate in the edible parts of plants, such as the roots and leaves. This accumulation poses the significant risks of food chain contamination. The model plant *Arabidopsis thaliana* (hereafter *Arabidopsis*), which is known for its fully sequenced genome, provides an excellent subject for mutagenesis studies aimed at understanding the physiological and molecular impacts of Tl. These studies are crucial for developing strategies to mitigate Tl toxicity and accumulation in plants.

Kuo-Chen Yeh (Academia Sinica) and his co-workers utilized the NSRRC **TPS 23A** X-ray nanoprobe beamline to perform X-ray fluorescence (XRF) analyses on *Arabidopsis* (**Fig. 2(a)**). These studies reveal that different concentrations of Tl not only inhibit growth and cause leaf chlorosis but also result in Tl accumulation in both roots and shoots, illustrating the plant's transport capabilities and the mechanisms of Tl toxicity.

Further investigations showed that Tl absorption occurs primarily through the roots, moving to the stems with increasing concentrations in the growth media. At peak levels, Tl concentrations reached 1,775 mg/kg in roots and 1,219 mg/kg in shoots. The plant demonstrated a significant transfer factor from roots to aerial parts, but this accumulation adversely affects the growth of both. The similarity in the distribution patterns of Tl and K within *Arabidopsis* suggests that they might share transport proteins or channels.

In addition to physiological studies, genetic and transcriptomic analyses aim to identify mutants with altered responses to TI and to explore the genes involved in its uptake and transport. Early transcriptional response studies have identified several TI-responsive genes associated with oxidative stress, antioxidant defense, K channel activity, and photosynthesis. These findings are integral to understanding TI's behavior in plants and the potential mechanisms of bioaccumulation (Fig. 2(b)), paving the way for future research and mitigation strategies.

Lemongrass and Sage Fertilized with Humic Acid Accumulate Toxins in Soil Treated with Heavy Oil Fly Ash

Saudi Arabia relies on over 40 million tons of heavy oil annually

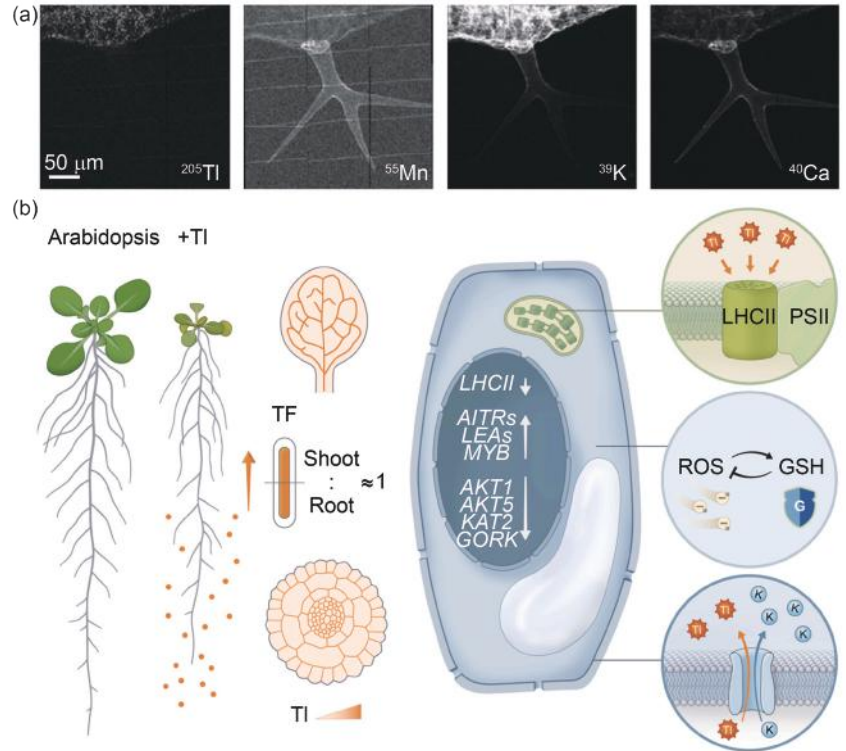


Fig. 2: (a) Synchrotron μ-XRF images of TI, Mn, K, and Ca distribution in TI-treated leaves. The elemental signal is shown in white. (b) A schematic representation of the uptake, toxicity, and accumulation of TI in Arabidopsis plants. [Reproduced from Ref. 2]

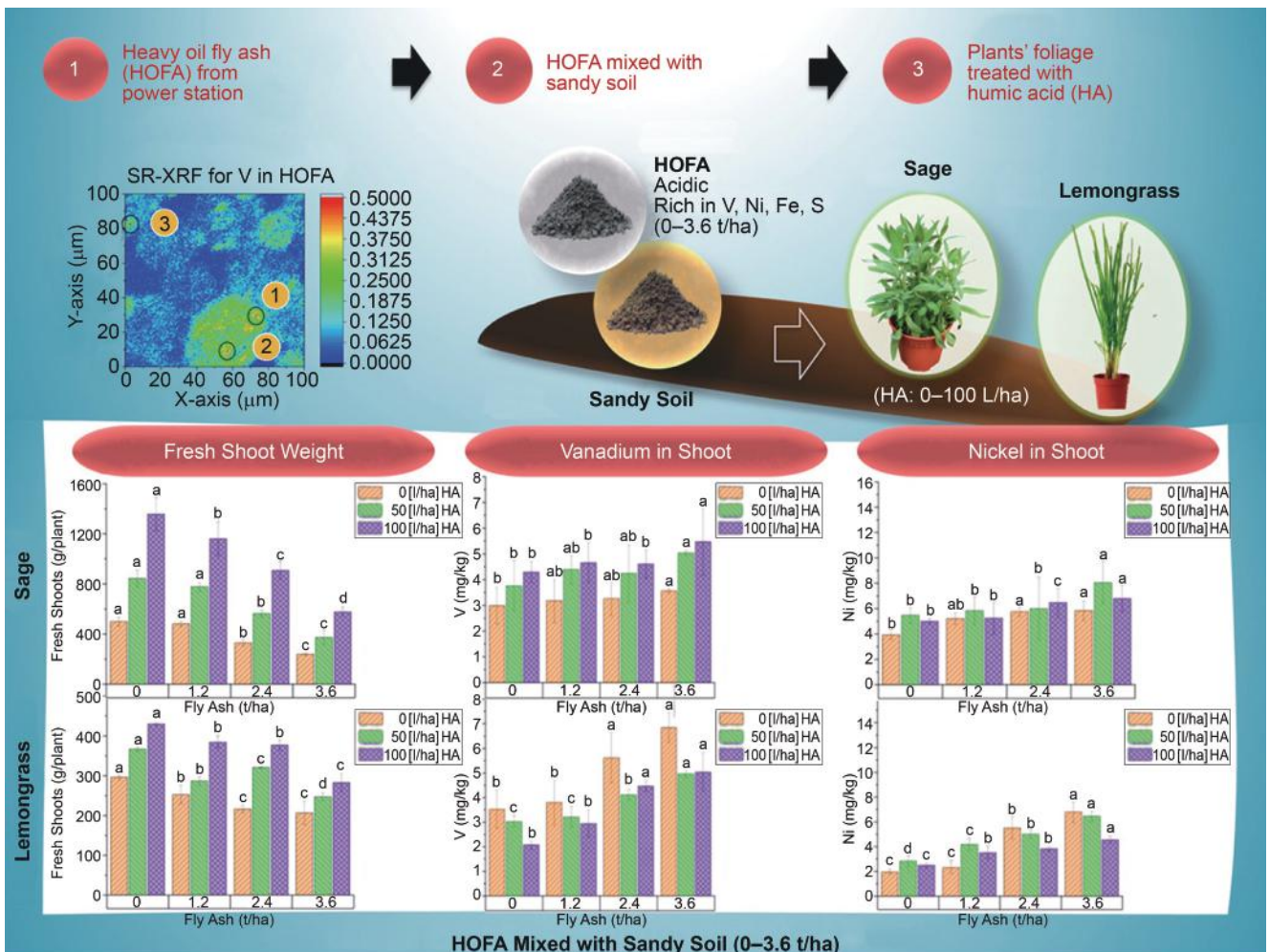


Fig. 3: Evaluating the effect of soil amendment with HOFA using sage and lemongrass as indicators. [Reproduced from Ref. 3]

for electricity and desalination, which contribute to 70% of its energy output, and it consumes about 320 million barrels of oil each year. This process results in the production of roughly 250,000 tons of heavy oil fly ash (HOFA) annually, with the individual facilities like the Rabigh power plant generating around 10,000 tons. Predominantly, HOFA is disposed of in landfills, a method that increases the risk of air, surface water, and groundwater pollution due to its content of unburned carbon and toxic inorganic compounds like vanadium and nickel.

Efforts to manage and mitigate HOFA's environmental risks are crucial. It has found uses in construction as a stabilizing material in cement, concrete blocks, asphalt mixtures, and in synthesizing glass ceramics. Additionally, HOFA serves economical roles in recovering valuable metals and as a water treatment adsorbent. However, its application in agriculture as a soil amendment is less explored because of the high levels of toxic elements it contains, which can harm soil quality and plant growth.

Samir Gamil Mohammad Al-Solaimani (King Abdulaziz University, Saudi Arabia) and his co-workers conducted research using the NSRRC **TPS 23A** beamline, including XRF imaging and XANES analysis. These techniques aided in assessing the impact of varying HOFA doses on sandy soils' nutrient and toxic element profiles and explored the effects of humic acid on plant growth and elemental uptake (**Fig. 3**).

Results indicated a significant presence of vanadium, nickel, iron, and sulfur in HOFA, with lower levels of chromium and manganese and minimal silicon. Notably, about 17.3% of vanadium exhibited high mobility, potentially increasing the environmental risks. Experiments demonstrated that low HOFA doses (1.2 to 3.6 tons per hectare) slightly affected soil pH, electrical conductivity, and element content, while higher doses adversely impacted plant growth. Treatment with humic acid significantly improved plant resilience to HOFA-induced stress.

Among the species tested, common sage showed the superior capability in accumulating elements compared to lemongrass, though both remained below critical toxicity thresholds. The findings suggest that controlled application of HOFA could enhance nutrient levels in low-fertility soils without severely altering their properties, particularly when combined with humic acid. Common sage also displayed potential for phytoremediation in HOFA-treated soils containing nickel and vanadium. These insights confirm the potential of HOFA as a resource for improving soil health in arid environments and offer new strategies for its disposal that are relevant both within Saudi Arabia and globally.

These studies underscore the critical role of plants in monitoring and improving soil health, providing the valuable data that can inform soil management strategies and mitigate pollution. The findings from the NSRRC in 2024 emphasize the complex dynamics of substance accumulation in plants and offer new insights into effective environmental management practices. (Reported by Chun-Chieh Wang)

*This report features the work of Shan-Li Wang and his collaborators published in *Sci. Total Environ.* **949**, 175141 (2024); the work of Kuo-Chen Yeh and his collaborators published in *Ecotox. Environ. Safe.* **276**, 116290 (2024); and the work of Samir Gamil Al-Solaimani and his collaborators published in *Sci. Total Environ.* **945**, 173998 (2024).*

TPS 23A X-ray Nanoprobe

TPS 44A Quick-scanning X-ray Absorption Spectroscopy

TLS 16A1 Tender X-ray Absorption, Diffraction

- Quick-scanning XAS, XRF, XANES
- Environmental and Earth Sciences, Physics, Materials Science, Chemistry

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Sustainable Remediation Materials for Toxic-Metal Removal: Insights into Arsenic and Chromium Detoxification Mechanisms

Heavy metal remediation is achieved using an environmentally sustainable approach that involves thiol-functionalized black carbon and thermoacidophilic Cyanidiales.

The remediation of toxic metals, such as chromium (Cr) and arsenic (As), is vital for public health and ecological balance. The 2024 studies at the NSRRC explore the advanced materials and biological pathways for hexavalent Cr(VI) and arsenite [As(III)] removal. Utilizing synchrotron-based techniques, the research highlights thiol-functionalized black carbon for Cr(VI) remediation and thermoacidophilic Cyanidiales for As(III) detoxification, offering effective and sustainable solutions through materials science and bioremediation advancements.

Thiol-Functionalized Black Carbon as Effective and Economical Materials for Cr(VI) Removal:

Hazardous Cr(VI) continues to raise critical environmental and public health concerns, necessitating the development of effective remediation methods. Yu-Ting Liu (National Chung Hsing University) and her collaborators recently discovered the removal mechanisms of Cr(VI) by modifying black carbon (BC), which is synthesized from rice straw residue and contains thiol groups. This is the first study that alters BC with thiol groups to target Cr(VI) removal. Here, the designated samples were evaluated: i) BC and ii) thiol-functionalized black carbon (S-BC) with BC/thioglycolic acid ratios (g mL⁻¹) of 1:20 (S-BC20), 1:30 (S-BC30), and 1:40 (S-BC40).

The research team conducted X-ray absorption spectroscopy (XAS) at TLS 17C1 and TPS 44A to determine the related Cr speciation on solid samples.

The results revealed that Cr species on solid samples primarily transformed from Cr(VI) to Cr(III), as shown in Fig. 1. At pH 3.5, over 86.2% of Cr(VI) was reduced to Cr(III), with S-BC retaining only 4.1–6.8% Cr(VI) compared to 13.8% for BC. At pH 5.5, all Cr(VI) was converted to Cr(III) on both materials. Finally, at pH 7.5, S-BC retained no Cr(VI), while BC retained 13.2% Cr(VI).

Sorption isotherms confirmed that S-BC40 demonstrated high Cr(VI) sorption capacities—201.2, 145.8, and 106.6 mg g⁻¹ at pH 3.5, 5.5, and 7.5—exceeding BC sorption capacities by 2.0–2.3 times. Notably, S-BC40 converted all sorbed Cr into Cr(III) at pH ≥ 5.5, forming Cr(OH)₃ and organic Cr(III) complexes. These findings highlight thiol functionalization as a promising strategy for effective Cr(VI) remediation and waste reutilization.

Accumulation and Bio-Oxidation of Arsenite Mediated by Thermoacidophilic Cyanidiales:

As contamination from geogenic and anthropogenic sources poses a critical

environmental and public health threat due to the high toxicity and mobility of As(III). Liu and her team unveiled the molecular mechanisms of As(III) removal by thermoacidophilic microalga Cyanidiales. This red alga, thriving in acidic, metal-rich environments, exhibits unique detoxification characteristics ideal for As remediation. This pioneering study evaluated As(III) removal by *Cyanidium caldarium* (Cc) and *Galdieria partita* (Gp) across a pH range of 2.0–7.0.

The team employed transmission X-ray microscopy (TXM) at TLS 01B1 to reveal the related As distribution on Cyanidiales, as shown in Fig. 2 (see next page). 3D tomography revealed that As present on Cc was distributed both near their surface and internally, particularly at pH 5.0, which suggests intracellular tolerance mechanisms. In contrast, Gp relied on surface-level As immobilization. These findings indicate that Cc is more resilient to As stress, particularly at pH ≤ 5.0 due to its superior intracellular detoxification capacity.

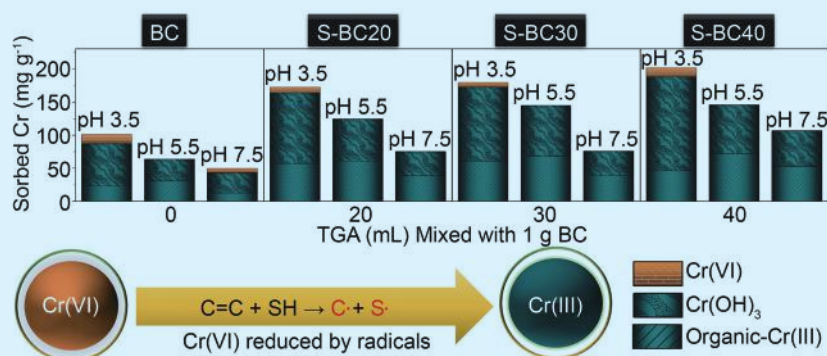


Fig. 1: Proposed removal mechanisms for Cr(VI) by thiol-functionalized black carbon. [Reproduced from Ref. 1]

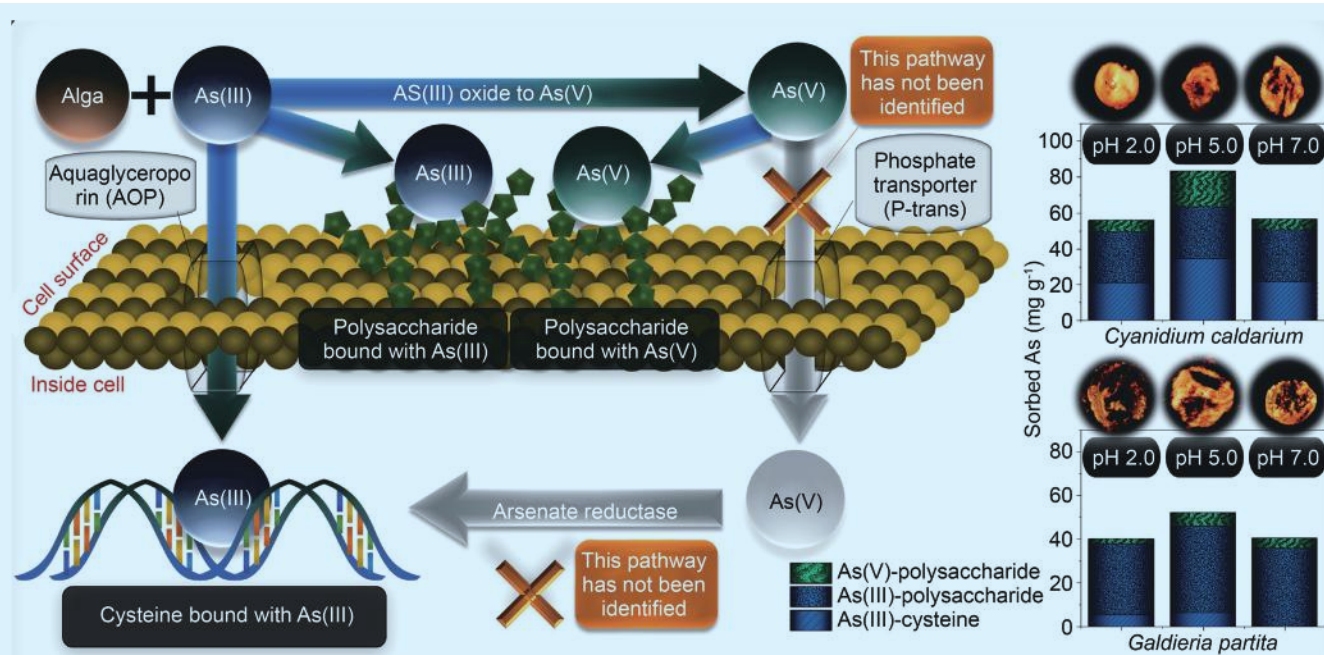


Fig. 2: Proposed mechanisms for As(III) removal by Cyanidiales. [Reproduced from Ref. 2]

The team conducted XAS analysis at **TLS 17C1** and **TPS 44A** to determine the related As speciation on Cyanidiales. As species were identified as arsenate [As(V)]-polysaccharide, As(III)-polysaccharide, and As(III)-cysteine, as illustrated in **Fig. 2**. At pH 5.0, Cc showed the highest sorption capacity that was dominated by As(III)-cysteine, while Gp retained mainly As(III)-polysaccharide. At pH 2.0 and 7.0, Cc exhibited balanced As(III)-polysaccharide and As(III)-cysteine with minimal As(V). These results highlight Cyanidiales' reliance on surface complexation and intracellular sequestration, with Cc exhibiting superior detoxification capacity.

To study As interactions in Cyanidiales, the team conducted synchrotron-based Fourier transform infrared spectroscopy at **TLS 14A1** to examine the changes in functional groups and protein secondary structures under As(III) exposure. The results show that α -helix/ β -strand ratios correlated with sorbed As levels. However, Gp exhibited a negative correlation between α -helix/ β -strand ratios and sorbed As levels, indicating structural fragility. In contrast, Cc displayed positive correlations at $\text{pH} \leq 5.0$, reflecting strong protein

adaptation. Notably, Cc exhibited fewer unordered structures, suggesting intracellular protein proliferation as a key response to As toxicity, further highlighting its superior resilience.

Cc demonstrated superior sorption capacity, achieving 83.2 mg g^{-1} at pH 5.0. Cc outperformed Gp across all tested conditions, driven by mechanisms such as As(III) oxidation, surface complexation with polysaccharides, and intracellular formation of As(III)-cysteine complexes, as shown in **Fig. 2**. These findings highlight Cyanidiales' ability to transform and immobilize As, paving the way for innovative and effective detoxification strategies.

In summary, this report showcases two sustainable approaches for heavy-metal remediation: i) thiol-functionalized BC for Cr(VI) removal and ii) thermoacidophilic Cyanidiales for As(III) removal. Leveraging NSRRC's synchrotron-based techniques, both methods demonstrated high levels of toxic-metal removal, offering promising solutions to environmental contamination. (Reported by Yen-Lin Cho, National Sun Yat-sen University, and Kamonchanok Huangmee, National Chung Hsing University)

This report features the work of Yu-Ting Liu and her collaborators published in *J. Environ. Manage.* **360**, 121074 (2024) and *Bioresource Technol.* **406**, 130912 (2024).

TPS 44A Quick-scanning X-ray Absorption Spectroscopy
TLS 01B1 X-ray Microscopy
TLS 14A1 IR Microscopy
TLS 17C1 EXAFS

- TXM, FT-IR, EXAFS, XAS
- Environmental and Earth Sciences, Biological Science, Chemistry

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Citric Acid Enhances Phosphate Release from Humic Acid-Iron Hydroxide Coprecipitates

The presence of citric acids could mildly obstruct the structural development of the Fe domain in humic acid-iron hydroxide coprecipitates, as shown by X-ray absorption spectroscopy techniques.

Recent research led by Yu-Min Tzou (National Chung Hsing University) and his team has demonstrated the sorption of citric acid onto two humic acid-iron hydroxide coprecipitates (HAFHCPs), as well as the mutual effects of citric acid and phosphate (P) sorption on these HAFHCPs at different C/Fe ratios. Their findings show that as the C/Fe ratio increases to 0.5, the maximum sorption capacity (MSC) of citric acid on HAFHCP-Y50 and HAFHCP-A50 decreases by 9.1–16.7%

(Fig. 1). In addition, the citric acid sorption capacity of HAFHCP containing humic acid was extracted from volcanic soil of Yangmingshan (HAFHCP-YHA) is approximately 92% of that of HAFHCP containing the Sigma-Aldrich humic acid (HAFHCP-AHA). This trend is likely due to competition for sorption sites on iron hydroxide (FH) surfaces between YHA and citric acid, which is driven in part by the electrostatic repulsion between negatively charged HA and citric acid at pH 5.5.

Using X-ray absorption spectroscopy (XAS) at TLS 16A1, Tzou and coworkers observed through Fe K-edge X-ray absorption fine structure (EXAFS) analysis that the k^3 -weighted $\chi(k)$ data for iron hydroxide (FH) and FH with sorbed citric acid were largely similar. However, slight differences emerged at $k \approx 5.0, 7.5,$ and 8.5 \AA^{-1} between free HAFHCP-Y50/A50 and HAFHCP-Y50/A50 with sorbed citric acid. These findings suggest that citric acid impedes the structural development of Fe domains in HAFHCP, increasing dissolved Fe content and reducing the sorption capacity for citric acid (Fig. 1). A previous study by Tzou and his colleagues showed that HAFHCP-Y50 likely contains a relatively homogeneous distribution of C and Fe domains. In contrast, HAFHCP-A50 appears to have a ferrihydrite core with humic acid growing on its surface and possesses fewer polar functional groups. This arrangement results in greater Fe dissolution when citric acid is sorbed.

Understanding how HAFHCPs influence P availability is critical, particularly when organic acids and P coexist. To explore this, three experimental systems were developed to

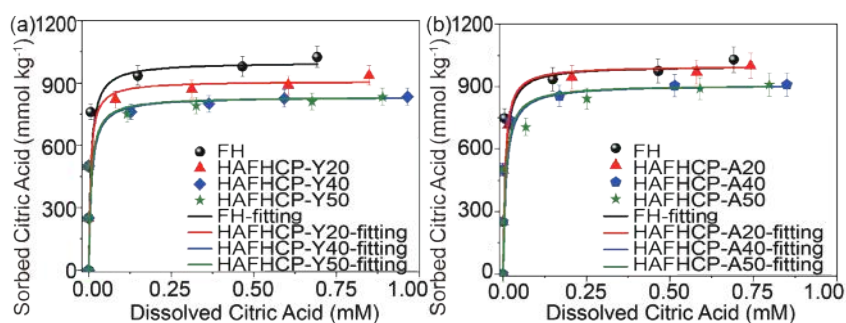


Fig. 1: Citric acid sorption isotherms for iron hydroxide (FH) and HAFHCP containing (a) YHA and (b) AHA with initial C/Fe ratios of 0.2, 0.4, and 0.5 (HAFHCP-Y20, Y40, Y50, A20, A40, and A50) fitted with the Langmuir isotherm model (solid lines). [Reproduced from Ref. 1]

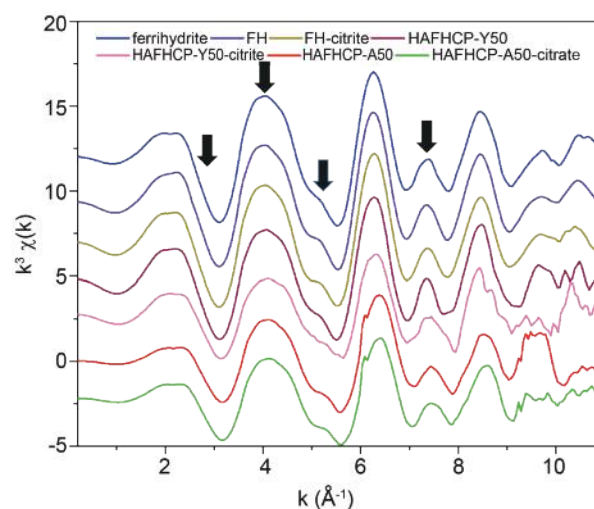


Fig. 2: Fe K-edge EXAFS spectra after sorption of citric acid on FH, HAFHCP-Y50, and HAFHCP-A50. Initial amounts of added citric acid were 0 and 2500 mmol kg⁻¹. [Reproduced from Ref. 1]

assess the cross-competitive sorption of P and citric acid on FH and HAFHCPs. Across all three systems, P sorption onto FH, HAFHCP-YHA, and HAFHCP-AHA followed the same order: P-C > S > C-P (Fig. 3, see next page). In the P-C, S, and C-P systems, P sorption on FH, HAFHCP-Y50, and HAFHCP-A50 differed significantly, having a decreasing trend of 1669–1780, 1245–1365, and 1018–1132 mmol kg⁻¹, respectively (Fig. 3). Notably, in the C-P system, pre-sorbed citric acid reduced P sorption by 50% compared with the MSC of P on FH (2250 mmol kg⁻¹) likely because of citric acid acting as a diffusion barrier for P. In

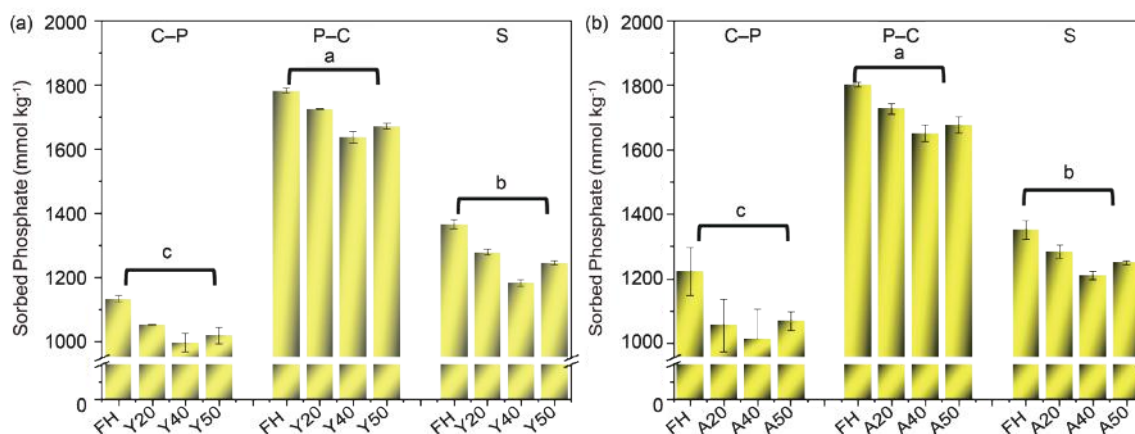


Fig. 3: The P sorption capacity for FH and HAFHCP containing (a) YHA and (b) AHA with initial C/Fe ratios of 0.2, 0.4, and 0.5 (HAFHCP-Y20, Y40, Y50, A20, A40, and A50) on cross-competitive sorption systems of C-P, P-C, and S. The order of addition for the different sorption systems are as follows: (1) For the C-P system, 1000 mmol kg⁻¹ of citric acid was first added into FH/HAFHCP suspensions for 42 h, and then 2250 mmol kg⁻¹ of P was added and allowed to react for another 42 h. (2) For the P-C system, 2250 mmol kg⁻¹ of P was added and allowed to react with FH/HAFHCP for 42 h prior to the addition of 1000 mmol kg⁻¹ of citric acid. The mixtures were then allowed to react for another 42 h. (3) In the S system, both P and citric acid were added simultaneously and allowed to react for 42 h. Statistical analysis was performed using one-way analysis of variance followed by Fisher's least-significant-difference (LSD) multiple-comparisons test at a significance level of $P < 0.05$. Significant differences between groups are denoted by different letters (a, b, c), as determined by Fisher's protected LSD test at a significance level of $P = 0.05$. [Reproduced from Ref. 1]

contrast, in the P-C system (where P was pre-sorbed), the presence of citric acid reduced P sorption by approximately 20%. Such effects may result from dissolved Fe functioning as a bridging agent between citric acid and P. Overall, these results indicate that citric acid strongly enhances P release in the C-P system.

In summary, this study emphasizes the role of HAFHCPs in the cross-competitive sorption of citric acid and P. The findings show that factors such as C/Fe ratios, the organic composition of humic acids, and the presence of citric acids (as observed through XAS) are key to understanding P availability. Consequently, the implementation of soil management strategies that foster favorable conditions for P availability is crucial for the sustainable utilization of P resources. (Reported by Kai-Yue Chen, National Chiayi University)

This report features the work of Yu-Min Tzou and his collaborators published in Environ. Res. 240, 117517 (2024).

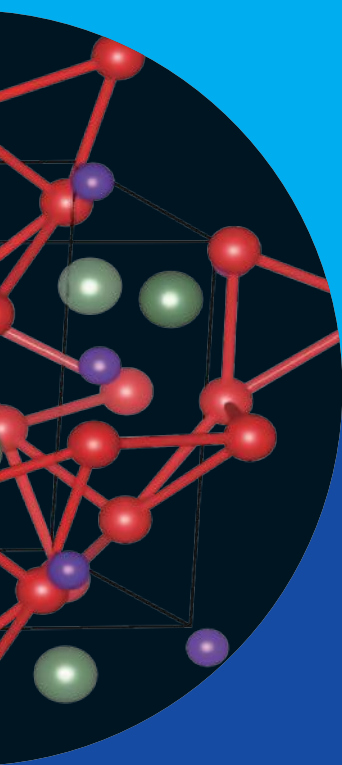
TLS 16A1 Tender X-ray Absorption, Diffraction

- EXAFS
- Environmental and Earth Sciences, Chemistry

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Neutron Science



Recent research conducted at the Australian Nuclear Science and Technology Organization (ANSTO) has employed neutron scattering techniques to explore the properties of various materials, yielding critical insights into their behavior and potential applications. One investigation examined the hyperkagome antiferromagnet Mn_3RhSi using low- and high-energy inelastic neutron scattering to analyze its magnetic excitations. Led by Shin-ichi Shamoto, the research team determined that, despite the manganese magnetic moment being approximately $5 \mu\text{B}$, only $2.6 \mu\text{B}$ orders below the Néel temperature of 190 K, suggesting substantial fluctuations attributable to geometric spin frustration. The calculated effective exchange interaction predicts a transition temperature aligning closely with the observed magnetic short-range order temperature of 720 K. Additionally, the study characterized the magnon dispersion, revealing an unusual form that results in a minimal magnon contribution to the electronic specific heat at low temperatures, underscoring the efficacy of neutron scattering in clarifying the magnetic properties of complex materials.

Another study utilized neutron reflectometry at ANSTO's **SPATZ** instrument to assess the thermal stability of non-fullerene organic solar cells (OSCs). Tzu-Yen Huang and colleagues investigated the morphology of bulk heterojunctions (BHJs) comprising PffBT4T-2OD as the donor and ITIC as the acceptor under thermal annealing. Their results revealed that the vertical morphology of the BHJ layers is initially well-mixed; however, annealing above 150°C triggers interface diffusion, predominantly due to ITIC molecule aggregation within the blends. This research highlights the critical need to address the thermal instability of acceptor materials like ITIC to improve the performance and longevity of OSCs.

The third investigation utilized neutron powder diffraction at ANSTO's **ECHIDNA** and **WOMBAT** instruments to explore the tunability of magnetic properties in the oxygen-deficient double perovskite YBaCuFeO_5 (YBCFO). Chao-Hung Du and his team demonstrated that introducing small amounts of Fe/Cu self-doping effectively modulates magnetic transition temperatures and the nature of magnetic order. Notably, a mere 1% iron addition increased the lower magnetic transition temperature (T_{n2}) by 65 K. This study offers a viable approach to tailoring YBCFO's magnetic properties by simultaneously inducing lattice effects and adjusting the Cu/Fe distribution.

Magnetic Excitation in the Hyperkagome Antiferromagnet Mn_3RhSi

Low- and high-energy inelastic neutron scattering measurements of a hyperkagome antiferromagnet revealed little magnetic specific heat capacity contribution in the enhanced electronic specific heat γ term.

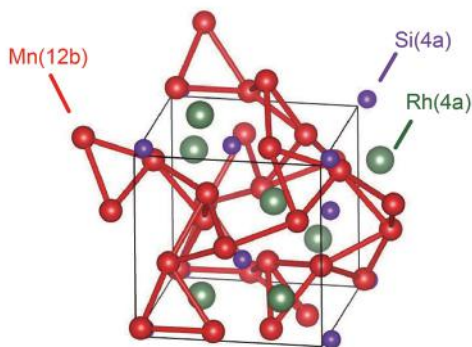


Fig. 1: Mn_3RhSi cubic crystal structure with a lattice parameter of $a = 6.467 \text{ \AA}$ at 293 K. The red bond network is a hyperkagome lattice of the Mn site (12b), where the Mn–Mn bond distance ranges from 2.66 to 2.76 \AA . [Reproduced from Ref. 3]

The hyperkagome antiferromagnetic semimetal Mn_3RhSi lacks spatial inversion symmetry in its β -Mn-type crystal structure (Fig. 1). The material exhibits intriguing magnetic behavior in which magnetic diffuse scattering due to magnetic short-range order is observed in the paramagnetic state up to 720 K, approximately 500 K higher than its Néel temperature of 190 K. The hyperkagome lattice is also found in well-studied β -Mn, which is a known spin liquid candidate and is a heavy Fermion $3d$ material with an electronic specific heat coefficient (γ) of about 70 mJ/MnK^2 .¹ In ordered β -Mn-type alloys, a quantum critical behavior is observed in the enhanced γ term.² Here, the team of Shin-ichi Shamoto (Comprehensive Research Organization for Science and Society, Japan) studied the magnetic excitation of Mn_3RhSi by low- and high-energy inelastic neutron scattering measurements to elucidate the magnetic contribution to the enhanced γ term. Their research results provided the fluctuating magnetic moment size and the magnetic specific heat capacity at low temperatures for Mn_3RhSi .

In this study, the team measured a 1.65 g Mn_3RhSi single crystal and observed one magnon mode up to the maximum energy, as shown in Fig. 2. The observed intensity in Fig. 3(a) revealed that the magnitude of the magnetic moment of Mn $3d^5$ was approximately $5 \mu_B$ and is almost localized. The magnetic moment of the long-range order state below the Néel point was only $2.6 \mu_B$ at 4 K,⁴ indicating that most of the magnetic moment fluctuates without being ordered. This fluctuation is thought to be due to the geometrical spin frustration of the hyperkagome

lattice. Interestingly, by using a magnetic moment of $2.6 \mu_B$ and the Néel temperature (190 K), the effective exchange interaction can be estimated, yielding a predicted transition temperature of 700 K from the magnetic moment of $5 \mu_B$ based on classical mean field approximation theory. The predicted temperature is very close to the temperature of the magnetic short-range order temperature 720 K mentioned above. The ability to measure low- and high-energy spin fluctuations even with a small crystal has allowed us to better understand mysterious magnetic materials.

Figure 3(b) shows the magnon dispersion of the low-energy mode in Mn_3RhSi . The solid line is fit using the equation $E = E_{\text{top}} \sin^{\alpha}(\pi h)$, where the best fit was obtained with $E_{\text{top}} = 39.5(3) \text{ meV}$ and $\alpha = 0.53(3)$. The peculiar dispersion leads to the magnon specific heat capacity of $C_{\text{mag}} = dU/dT \propto (T/E_{\text{top}})^{3/\alpha}$. As for the electronic specific heat coefficient γ of Mn_3RhSi , the magnon heat capacity becomes small at low temperatures because of the large exponent of $\sim (T/E_{\text{top}})^{\epsilon}$, resulting in little magnon effect on the T -linear electronic specific heat. The team confirmed that the magnon dispersion continues to the low-energy region below 1 meV at SIKa without an appreciable spin gap. The combination of the low- and high-energy inelastic

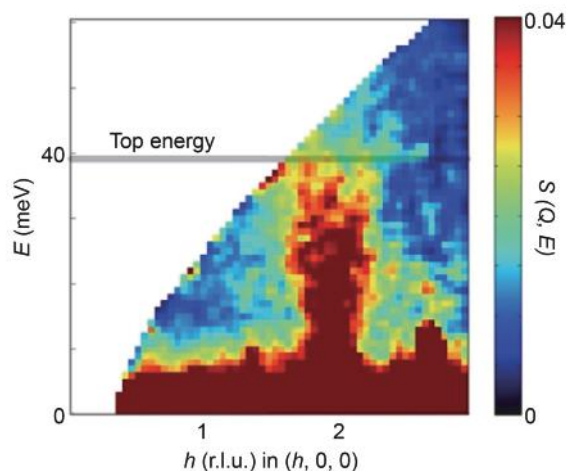


Fig. 2: Dynamical structure factor divided by the Bose factor $\{n(E) + 1\}$ in the low-Q region measured with $E_i = 95 \text{ meV}$. The single-crystal scattering intensity at $T = 6 \text{ K}$ is averaged vertically in the range of $-0.2 < k < 0.2$ and $-0.2 < l < 0.2$ (r.l.u.). The horizontal scattering plane is $[h, 0, 0]$ - $[0, k, k]$. The top energy of the low-energy magnon mode is shown by a solid line. [Reproduced from Ref. 3]

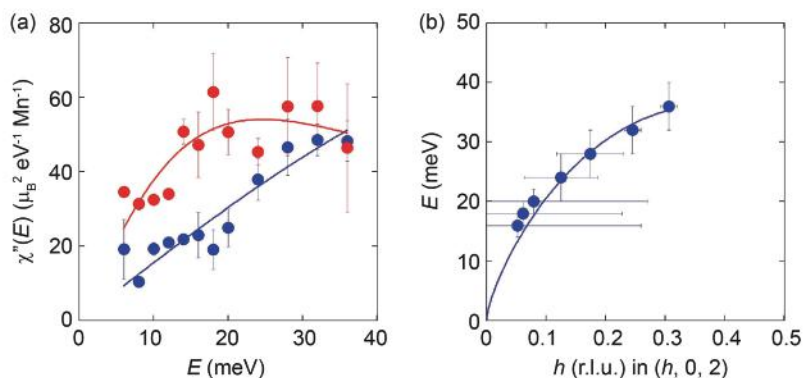


Fig. 3: (a) The dynamical spin susceptibilities are plotted as a function of energy at $T = 6 \text{ K}$ (blue open circles) and 200 K (red closed circles). (b) Magnon dispersion observed at $T = 6 \text{ K}$. Solid lines are used to aid with visual inspection. [Reproduced from Ref. 3]

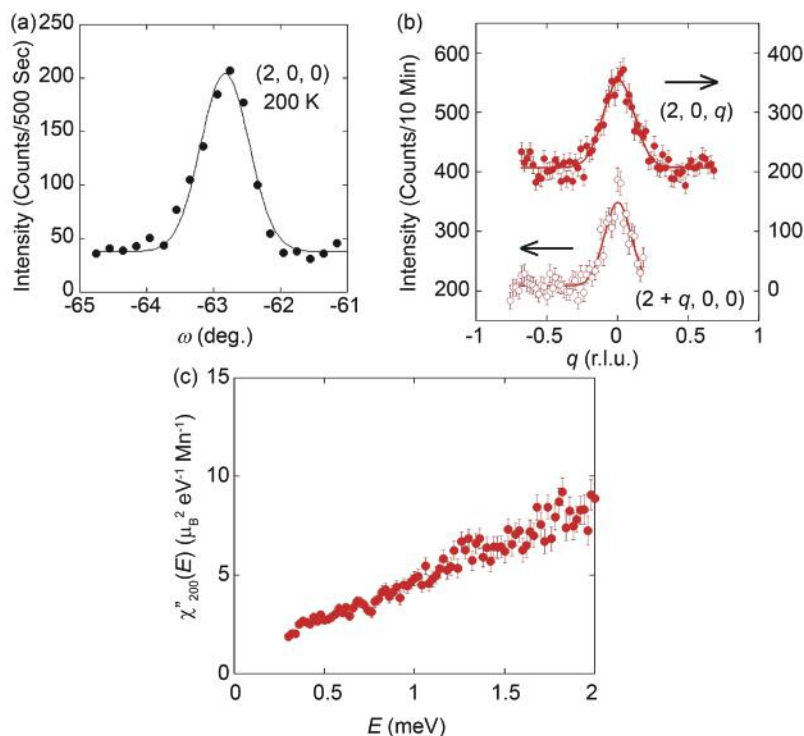


Fig. 4: (a) ω -scan at $(2, 0, 0)$ of the Mn_3RhSi crystal. (b) The constant-energy q -scan in $(2 + q, 0, 0)$ (open circles) and $(2, 0, q)$ (closed circles) of $E = 1 \text{ meV}$ at $T = 200 \text{ K}$. The horizontal scattering plane is $[h, 0, 0] - [0, 0, l]$. (c) Low-energy Q -integrated local dynamical spin susceptibility $\chi''_{200}(E)$ at $Q = (2, 0, 0)$ measured at $T = 200 \text{ K}$. [Reproduced from Ref. 3]

neutron scattering measurements of a hyperkagome antiferromagnet revealed how magnetic excitation can contribute to the magnetic specific heat capacity in the enhanced electronic specific heat γ term. (Reported by Shin-ichi Shamoto, Comprehensive Research Organization for Science and Society, Japan)

This report features the work of Shin-ichi Shamoto and his collaborators published in Phys. Rev. Research 6, 033303 (2024).

ANSTO SIKa – Cold Neutron Triple-axis Spectrometer

- Neutron Inelastic Scattering
- Materials Science, Condensed-matter Physics

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The Non-Fullerene Acceptor ITIC Induced the Morphology of Bulk Heterojunctions Through Thermal Annealing

The key component affecting the thermal stability of non-fullerene organic solar cells is explored.

The nanomorphology of bulk heterojunctions (BHJs) is a critical factor influencing the performance of non-fullerene organic solar cells (OSCs). Thermal annealing is widely used in enhancing device performance by reorganizing the donor and acceptor phases within BHJs. A team led by Tzu-Yen Huang (NSRRC) and Yan-Ling Yang (National Taiwan University of Science and Technology) systematically examined the vertical morphology of BHJ blend films, which utilized poly[(5,6-difluoro-2,1,3-benzothiadiazol-4,7-diyl)-alt-(3,3'-di(2-octyldodecyl)-2,2';5',2'';5'',2'''-quaterthiophen-5,5'''-diyl)] (PffBT4T-2OD) polymer as the donor and 3,9-bis(2-methylene-(3-(1,1-dicyanomethylene)-indanone))-5,5,11,11-tetrakis(4-hexylphenyl)-dithieno[2,3-d:2',3'-d']-s-indaceno[1,2-b:5,6-b']dithiophene (ITIC) as the acceptor through thermal annealing.

The team made use of the neutron reflectometry (NR) instrument (SPATZ) at the Australian Nuclear Science and Technology Organisation (ANSTO) to study the blend morphology of BHJ thin films. **Figures 1(a) and 1(c)** illustrate the NR patterns and scattering length density (SLD) profiles of PffBT4T-2OD:ITIC BHJs with various blend ratios deposited on quartz-coated glass substrates. The SLD values of PffBT4T-2OD monomers and ITIC molecules were calculated to be 0.65 and $1.44 \times 10^{-6} \text{ \AA}^{-2}$, respectively. The NR curves of the 1:9 ratio revealed distinct Kiessig fringes spanning a Q -range of 0.02 to 0.10 \AA^{-1} , indicating high-quality thin films. As the ratio of PffBT4T-2OD is increased, the clear fringes are preserved, demonstrating good film quality across different blend ratios. The fringes of the BHJ layer (9:1 ratio) became less defined due to the proximity of the SLD value of PffBT4T-2OD to that of the atmosphere, which reduced contrast. For all samples, the quartz layer on the glass substrate exhibited a consistent thickness of about 200 \AA with a roughness of 13 \AA . The SLD value of different BHJ layers ranged from $1.39 \times 10^{-6} \text{ \AA}^{-2}$ (1:9 ratio) to $0.70 \times 10^{-6} \text{ \AA}^{-2}$ (9:1 ratio). These findings suggest that the vertical morphology of the BHJ layers is well-mixed, without distinct phase segregation.

Thermal annealing is a common method for optimizing phase separation in a BHJ layer, facilitating the formation of the desired morphologies for efficient charge transport. To avoid surpassing the recrystallization temperature of ITIC at 170°C , the thermal treatment was carried out incrementally, with annealing temperatures reaching up to 150°C . **Figures 1(b) and 1(d)** present the NR patterns and the SLD profiles of the PffBT4T-2OD:ITIC BHJ layers (5:5 blend ratio) deposited on Si wafers, which were subjected to annealing at temperatures ranging from 90 to 150°C . The pristine PffBT4T-2OD:ITIC BHJ layers exhibit fringes in the Q -range of 0.010 to 0.040 \AA^{-1} , indicating the formation of smooth and uniform thin films. At an annealing temperature of 130°C , the fringes remain intact, signifying that

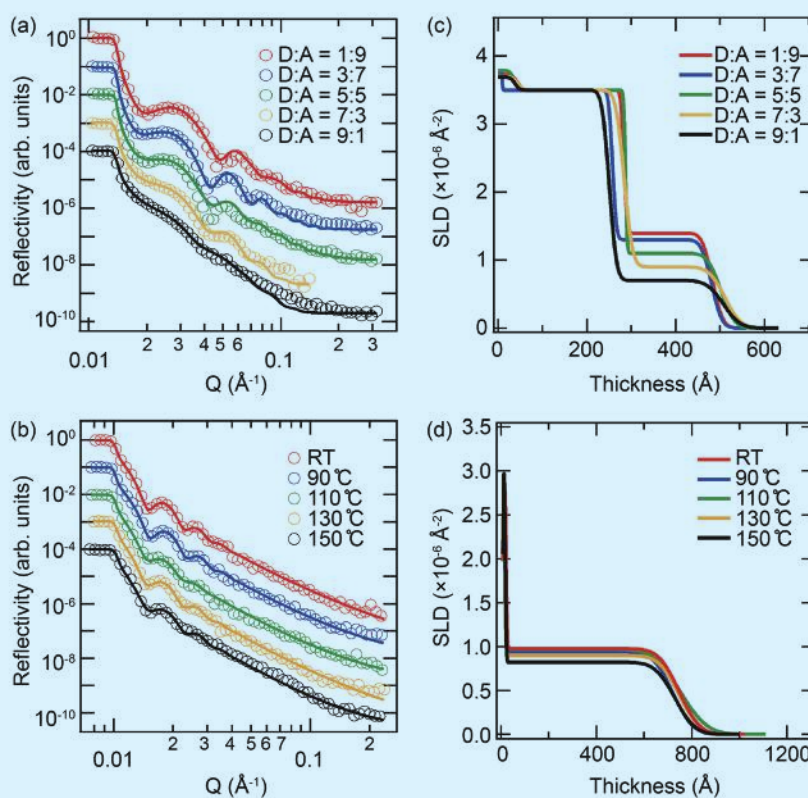


Fig. 1: (a) NR profiles and (c) corresponding SLD profiles of PffBT4T-2OD:ITIC BHJ films with different donor/acceptor ratios (1:9, 3:7, 5:5, 7:3, and 9:1) on the quartz-coated glass substrates. The individual data points and solid lines represent the experimental data and fitting results, respectively. The NR curves have been offset for clarity. (b) NR profiles and (d) corresponding SLD profiles of PffBT4T-2OD:ITIC (5:5) BHJ films on the Si wafers annealed from RT to 150°C . [Reproduced from Ref. 1]

the interface of the film is maintained. The corresponding SLD value ($0.96 \pm 0.05 \times 10^{-6} \text{ \AA}^{-2}$) of the PffBT4T-2OD:ITIC BHJ layer also remains unchanged at this temperature. At 150°C , the fringes near $Q = 0.025 \text{ \AA}^{-1}$ become indistinct, suggesting an increased diffusive interface between the air and the film, accompanied by a decrease in the SLD value to $0.81 \times 10^{-6} \text{ \AA}^{-2}$. These results indicate that interface diffusion of the PffBT4T-2OD:ITIC BHJ layers is initiated after annealing at 150°C .

To understand the phase behavior of BHJ blends, the individual donor and acceptor materials were examined under the same annealing conditions. The NR curves and SLD profiles of pristine PffBT4T-2OD films on the Si wafer with different annealing temperatures are shown in **Figs. 2(a) and 2(c)**. The fringes remain unchanged as the annealing temperature increases. The SLD profiles show that a thickness of $614 \pm 9 \text{ \AA}$ and surface roughness of $30 \pm 1 \text{ \AA}$ are retained after thermal treatment. These results suggest that even when the annealing temperature exceeds 90°C , the highly active side chains of PffBT4T-2OD do not induce significant vertical morphological changes in the films. On the other hand, **Figs. 2(b) and 2(d)** display the NR patterns and SLD profiles of pure ITIC thin films. The fringes are observed within the Q -range of 0.01 to 0.03 \AA^{-1} for temperatures between 90 and 130°C . The corresponding film thickness and roughness are about 554 ± 10 and $106 \pm 9 \text{ \AA}$, respectively. The NR patterns become less distinct after thermal annealing at 150°C . In addition, there is little change in the position of the Kiessig fringes, indicating that there is little change in the ITIC film thickness. The thickness was set to 555 \AA with limits of $\pm 5 \text{ \AA}$, and then the SLD value and interfacial roughness were fitted. The SLD value decreased to $0.75 \times 10^{-6} \text{ \AA}^{-2}$ along with an increase in roughness to 139 \AA . These results suggest that interface diffusion begins at 150°C , likely driven by intrinsic agglomeration of the ITIC molecules rather than by interactions with the substrate. Additionally, even brief annealing at this temperature appears to induce ITIC nanocrystal reorganization, leading to variations in the vertical morphology of the thin film.

In summary, the study demonstrated the diffusive interfaces of the PffBT4T-2OD:ITIC BHJ thin films emerging when the annealing temperature exceeds 150°C primarily because of ITIC aggregation within the blends. Thermal annealing serves as a critical post-treatment process for removing residual solvent and optimizing phase distribution in BHJ thin films. Within a brief annealing period, the morphology of PffBT4T-2OD:ITIC BHJs are influenced by ITIC nanoclusters. The findings suggest that the initial unfavorable mixing contributes to the instability of ITIC molecules during annealing, adversely

affecting power conversion efficiency and device stability. It is essential to consider thermal instability and processing conditions for the enhanced performance of OSCs for promising BHJs incorporating ITIC-based small molecules. (Reported by Tzu-Yen Huang)

This report features the work of Tzu-Yen Huang and his collaborators published in ACS Appl. Nano Mater. 7, 17588 (2024).

ANSTO SPATZ – Neutron Reflectometer

- NR
- Polymers, Morphology, Interfaces, Thin Films

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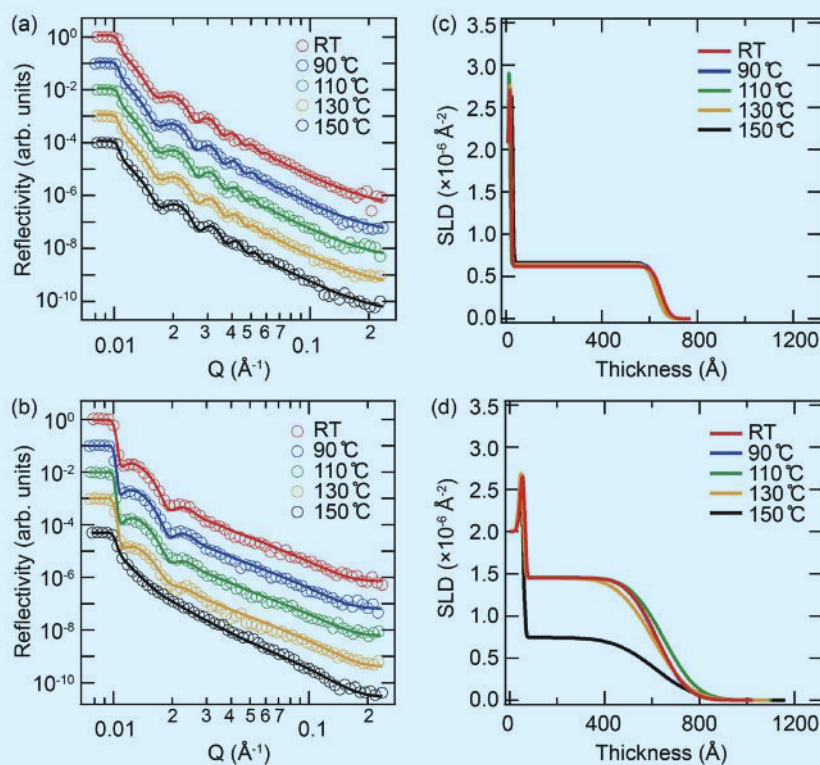


Fig. 2: NR curves and the corresponding SLD profiles for pristine (a,c) PffBT4T-2OD and (b,d) ITIC films on the Si wafers annealed from RT to 150°C . [Reproduced from Ref. 1]

Effective Modulation of the Magnetic Properties of YBaCuFeO_5

Very low concentrations of Cu/Fe self-doping can effectively change the magnetic properties of YBaCuFeO_5 .

Multiferroic materials, in which ferroelectricity and magnetic ordering coexist, have been actively studied with interests in fundamental and applied sciences. Several decades of investigations reveal that the incommensurate orientation-modulated spin structures, such as spiral and cycloidal, are good candidates for hosting ferroelectricity. In such materials, termed type II ferroelectrics, the electrical polarization is coupled with the magnetic structures triggered by the Dzyaloshinskii–Moriya interaction or magnetic frustration. Nevertheless, the magnetic ordering temperature T_N and ferroelectric Curie temperature T_C are generally well below the ambient temperature and are unfavorable for daily-life applications. While pursuing multiferroic materials with high T_C , the oxygen-deficient double perovskite, YBaCuFeO_5 (YBCFO), arouses wide attention. YBCFO possesses the layered structure of $\text{MM}'\text{O}_5$ bipyramids (Fig. 1(a)), and Cu/Fe resides within the double pyramids. The Cu/Fe spins undergo antiferromagnetic transition to a commensurate, collinear antiferromagnetic order characterized by the propagation vector of $k_{c1} = (1/2, 1/2, 1/2)$ at T_{N1} . Below the second magnetic phase-transition temperature T_{N2} , the spin structure transits into an incommensurate chiral structure, with propagation vector $k_i = (1/2, 1/2, 1/2 \pm \delta)$.¹ The reported values of T_{N1} and T_{N2} are inconsistent in the literature, and the differences are as large as several tens of degrees. Such differences are unlikely due to the experimental errors but imply a tunable magnetic property of the compound. It is demonstrated that T_{N2} is successfully tuned by chemical substitution and a post-sintering cooling rate.^{2,3} Chemical substitution can tune the lattice, change the interlayer distances, and subsequently alter the strength of interlayer interactions. Increasing the cooling rate or quenching the sample after the sintering has been proven to be an effective way of controlling the Cu/Fe distribution within the bipyramids. Theoretical studies show that disorder in the Cu/Fe distribution is crucial for the onset of commensurate to incommensurate magnetic transition (CM–ICM). At the same time, the CM phase persists down to low temperature without transiting into the ICM phase in the sample with highly ordered cation distribution.⁴

Magnetic transition temperatures are correlated with the degree of disorder. T_{N2} increases while T_{N1} is slightly suppressed when the Cu/Fe distribution is more disordered. Therefore, the ICM order temperature can be enhanced by tuning the Cu/Fe distribution. In the noncentrosymmetric $P4mm$ structure, the bipyramids are occupied by one Fe and one Cu, with ordered arrangement. The in-plane nearest-neighbor interactions are strongly antiferromagnetic despite the Cu/Fe distribution; therefore, the spins order into the checkerboard arrangement in the ab-plane.

Meanwhile, the interplane interactions alternate the sign along the c -axis and are ferromagnetic within the bipyramids and antiferromagnetic between bipyramids, resulting in the $[+--+]$ ground state characterized by the propagation vector of $k_{c1} = (1/2, 1/2, 1/2)$. By increasing the post-annealing cooling speed or quenching, disorder is introduced. Fe–Fe and Cu–Cu pairs could occupy the bipyramid sites locally. Theoretical works suggest that the Fe–Fe pairs occupying the bipyramid impurities serve as “impurities”, introducing antiferromagnetic interactions to the matrix of ferromagnetic coupling. When the concentration is sufficiently high and the temperature is sufficiently low, the incommensurate spiral structure sets in.

Chao-Hung Du (Tamkang University) proposed an alternative way to tune the magnetic properties of YBCFO by directly modifying the B site. By introducing additional Fe/Cu to the B sites, the local Fe/Cu arrangement could be changed to avoid the potential effects of the foreign

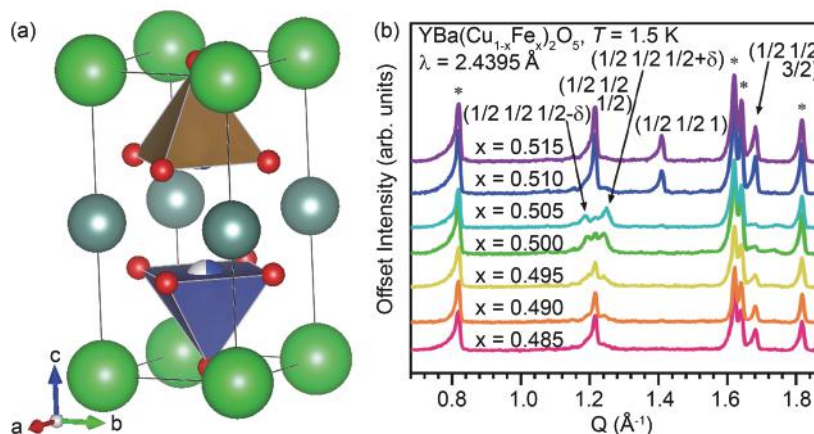


Fig. 1: (a) Crystal structure of YBCFO. (b) NPD patterns of selected $\text{YBa}(\text{Cu}_{1-x}\text{Fe}_x)_2\text{O}_5$ samples. The asterisks indicate the nuclear reflections, and the Miller indexes of magnetic peaks are labeled. [Reproduced from Ref. 6]

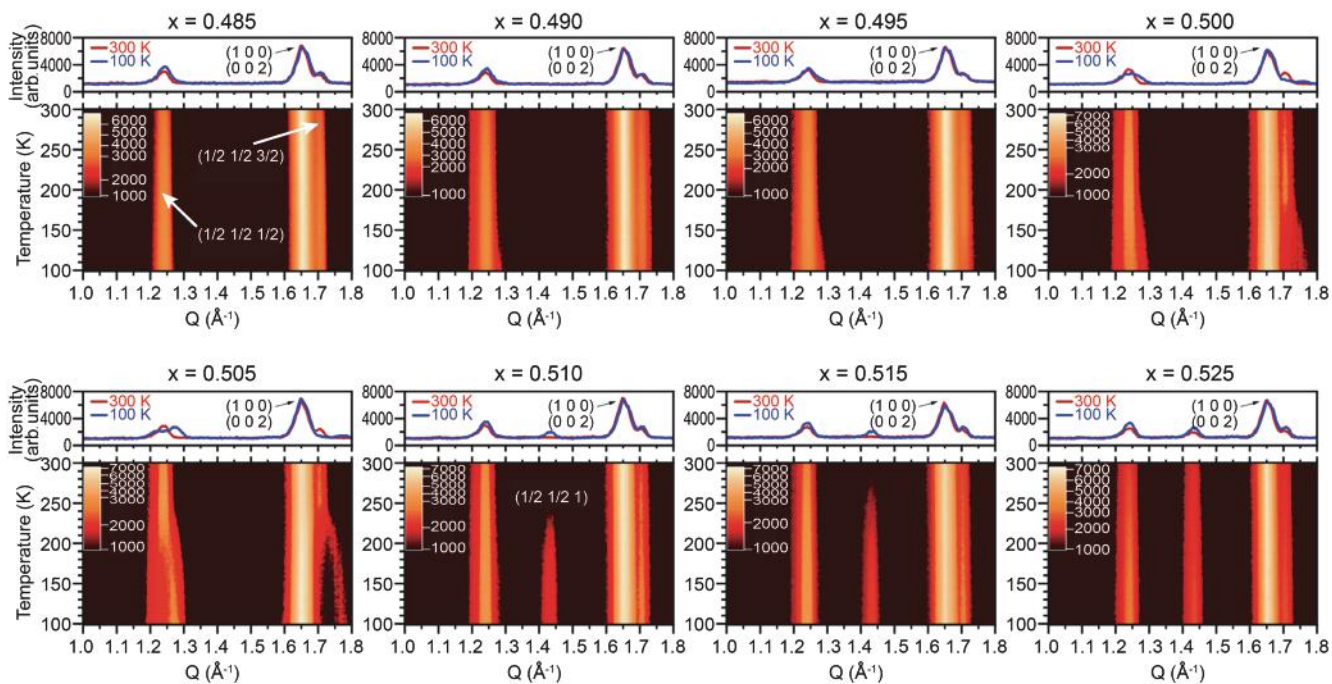


Fig. 2: Two-dimensional contours of eight $\text{YBa}(\text{Cu}_{1-x}\text{Fe}_x)_2\text{O}_5$ samples with distinct Cu/Fe ratios illustrate the temperature dependence of NPD patterns measured from 100 to 300 K. These figures focus on the low- Q region where the magnetic reflections appear NPD-pronounced. [Reproduced from Ref. 6]

element dopants. Polycrystalline $\text{YBa}(\text{Cu}_{1-x}\text{Fe}_x)_2\text{O}_5$ with x ranging between 0.485 and 0.525 were synthesized. **Figure 1(b)** shows the neutron powder diffraction (NPD) patterns collected at 1.5 K, employing the high-resolution neutron powder diffractometer, **ECHIDNA**. The patterns reveal various magnetic structures. The thermal evolution of magnetic structures was studied on the high-intensity neutron powder diffractometer, **WOMBAT**. **Figure 2** displays the temperature evolution of the magnetic structures of $\text{YBa}(\text{Cu}_{1-x}\text{Fe}_x)_2\text{O}_5$ and is summarized in the phase diagram shown in **Fig. 3**. The k_{c1} and k_{icm} magnetic phases coexisted at 1.5 K in the $x = 0.500$ sample. Noticeably, the incommensurate phase was most pronounced in the $x = 0.505$ sample, and the associated peaks were weaker because the iron content increased

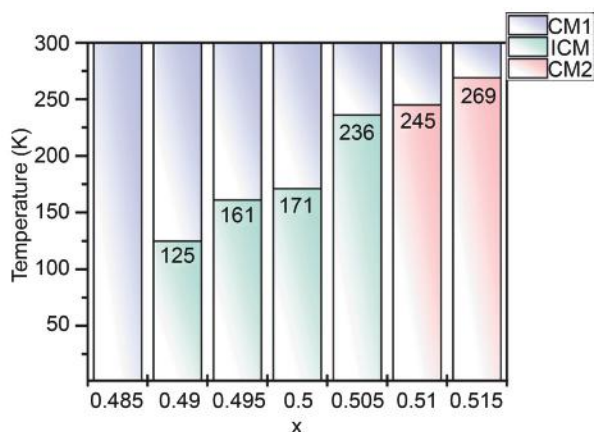


Fig. 3: Phase diagram of $\text{YBa}(\text{Cu}_{1-x}\text{Fe}_x)_2\text{O}_5$. [Reproduced from Ref. 6]

above 0.505. Compared to the parent YBCFO sample, T_{N2} was enhanced by 65 K by adding merely 1% iron. When the iron content was reduced from $x = 0.505$, the CM-ICM transition temperature, T_{N2} , gradually decreased, suggesting that the impurity bond density was gradually reduced. By contrast, the k_{icm} phase diminished much more quickly, and there were only extremely weak peaks of k_{icm} in the NPD of $x = 0.510$ and 0.515 samples. In samples with high iron content ($x = 0.510$ and 0.515), a new commensurate magnetic phase $k_{c2} = (1/2, 1/2, 1)$ emerged. In the k_{c2} phase, the checkerboard arrangement was preserved in the ab -plane, and the sequence of spin arrangement along the c -axis was $[+-+]$, contrary to $[+++]$ in the k_{c1} phase. The additional iron content likely introduced excessive antiferromagnetically coupled Fe-Fe pairs to the bipyramid layers and destroyed the delicate balance for the emergence of the incommensurate spiral structure. The coexistence of k_{c1} , k_{c2} , and k_{icm} phases was observed in isostructural NdBaCuFeO_5 as magnetic texturing due to the partial ordering of Fe/Cu^{5+} and is similar to the behavior of highly ordered YBCFO.⁴

The magnetic properties of the oxygen-deficient double perovskite YBCFO can be tuned by A-site doping by adjusting the crystal structure to affect the magnetic coupling strength. By controlling the post-sintering cooling rate, the cation distribution on the B sites where the magnetic species Cu/Fe resides was determined. The current work proposed a more effective route that can simultaneously introduce the lattice effect and tune the Cu/Fe distribution. (Reported by Chin-Wei Wang)

This report features the work of Chao-Hung Du and his collaborators published in *Phys. Rev. Mater.* **8**, 054404 (2024).

ANSTO ECHIDNA – High Resolution Powder Diffractometer

ANSTO WOMBAT – High-Intensity Powder Diffractometer

- NPD
- Materials Science, Chemistry, Condensed-matter Physics

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Inelastic Neutron Scattering Reveals Strong Electron–Phonon Interactions Driving Ultrahigh zT Values

Through inelastic neutron scattering, strong electron–phonon interactions in Sb–Bi codoped GeTe crystals are revealed, unlock ultrahigh zT values. This breakthrough redefines thermoelectric innovation and accelerates the development of materials such as PbTe, SnTe, and SnSe.

Imagine a world where we can recover and utilize heat that would normally be lost. This includes the heat produced by automobile engines, the warmth radiating from industrial machines, and even the heat generated during physical exercise. The possibility of turning this heat into usable electricity could radically change how we consume energy. This is the promise of thermoelectric materials, which have the remarkable ability to convert thermal energy into electrical energy.¹ The range of potential applications is extensive—everything from making vehicles more fuel-efficient to developing more energy-efficient homes and appliances.

A significant advancement in the understanding and improvement of thermoelectric materials was achieved through the research conducted by Yang-Yuan Chen and his team at the Institute of Physics, Academia Sinica. By utilizing the powerful SIKa Cold Neutron Triple-axis Spectrometer at the Australian Nuclear Science and Technology Organisation (ANSTO),² Chen and his team discovered a novel mechanism in thermoelectric materials: the presence of ultrahigh zT , which serves as a figure of merit for thermoelectric performance driven by strong electron–phonon (EP) interactions and a low-dimensional Fermi surface. These discoveries offer new possibilities for enhancing thermoelectric performance, paving the way for applications that may revolutionize how we capture and use energy in our everyday lives.

Thermoelectric materials are designed to capture heat and convert it into electrical energy. However, they face a challenge: the heat usually travels through the material in a way that reduces its efficiency in generating electricity. The zT value is a key metric that indicates the efficiency of a thermoelectric material; the higher the zT , the better the material can convert heat into electrical power. By studying the intricate interactions between electrons (which carry electricity) and phonons (which transport heat), Chen's team identified a method to develop a material with an exceptionally high zT , enabling it to convert heat into electricity with remarkable efficiency.

The discovery is based on EP interactions, which occur at the atomic level, as shown in **Figs. 1(a) and 1(b)**. In many thermoelectric materials, heat dissipates too quickly because of weak coupling between electrons and phonons. However, in the materials studied by Chen's team, the strong EP interaction allows efficient electrical conduction while maintaining a temperature gradient that drives the thermoelectric process. Such as behavior leads to an ultrahigh zT , which is a game-changer in the thermoelectric field.

Moreover, Chen and his team found that the low-dimensional Fermi surface of the material—essentially, the arrangement of the electrons within the material—further enhances its thermoelectric properties. A low-dimensional Fermi surface allows electrons to travel with less resistance, which in turn improves the material's ability to generate electrical energy from heat, as shown in **Figs. 1 (c) and 1(d)**. This is a crucial insight for developing new thermoelectric materials suitable for real-world applications.

Further investigation into Sb–Bi codoped GeTe by Chen's team, using the SIKA Cold Neutron Triple-axis Spectrometer, revealed even more remarkable findings as shown in **Figs. 1(e) and 1(f)**. They found a strong Kohn anomaly in the phonon dispersion of this material, which indicates the strong electron-phonon interaction. This anomaly occurs when the Fermi surfaces of the material exhibit significant nesting, which implies that the two parallel Fermi surfaces overlap significantly. This nesting enhances the EP interactions, which are crucial for thermoelectric efficiency. The Sb–Bi codoped GeTe material exhibited a distinctive Kohn anomaly that was absent in the pristine GeTe, suggesting that codoping with Sb and Bi strengthens the EP interaction, thereby improving the material's thermoelectric properties.

The Kohn anomaly plays a pivotal role in enhancing the thermoelectric efficiency of materials. In Sb–Bi codoped GeTe, the anomaly occurs because the Fermi level is slightly below the D maximum of the material, resulting in an electronic structure that behaves in a one-dimensional manner. This feature allows electrons to move more efficiently along a single axis, which contributes to improved thermoelectric performance. The strong EP interactions in Sb–Bi codoped GeTe result in superior heat-to-electricity conversion, leading to a material with a higher zT value.

How does this discovery impact our daily lives? Consider the substantial energy we waste. In the modern world, nearly everyone has experienced some form of energy loss, whether from excessive heat in homes, industries, or vehicles. For example, in a typical car engine, a considerable portion of the energy from the fuel is lost as heat. Imagine if a car could capture even a small portion of that wasted heat and convert it into electricity to power its electronic systems or improve fuel efficiency. Thermoelectric materials with ultrahigh zT values could facilitate this, creating more efficient vehicles that require less fuel and produce lower emissions.

Similarly, industries generate enormous amounts of heat during the production processes. Factories could use thermoelectric materials to capture and convert this waste heat into usable electricity, thus powering their operations or reducing the need for external energy sources. This approach could lead to more sustainable manufacturing practices, reduce energy consumption, and lower costs for businesses, ultimately benefiting consumers as well.

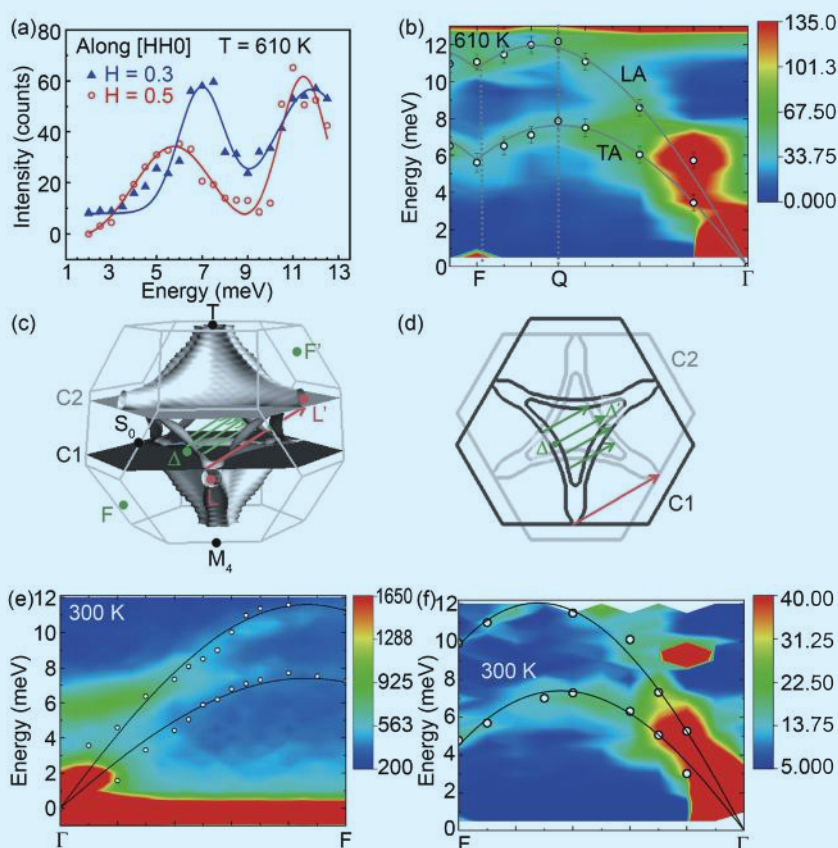


Fig. 1: Relationship between phonon dispersion and density of states. (a) Phonon excitation spectra measured in constant-Q scans along the crystallographic [HH0] direction at 610 K, with $H = 0.3$ represented by filled triangles and $H = 0.5$ represented by open circles. (b) Phonon dispersion of $(\text{Ge}_{0.86}\text{Sb}_{0.08}\text{Bi}_{0.06})\text{Te}$ at 610 K, measured along the crystallographic [110] direction. (c) Calculated Fermi surface of p-type GeTe at the Fermi energy, E_F . (d) The two corresponding 2D cross-sections (C1 and C2) of the Fermi surface. The short green arrows and the long red arrows indicate the wave vector difference Dk between the initial and final states due to the electronic transition in the [110] direction (G-F direction). Images of (c) and (d) were prepared using FermiSurfer. (e) Phonon dispersion of pristine GeTe at 300 K. (f) Phonon dispersion of Sb–Bi codoped GeTe at 300 K. [Reproduced from Ref. 3]

Thermoelectric materials have the potential to revolutionize energy consumption in our daily lives. In household appliances such as refrigerators, air conditioners, and heating systems, these materials could recycle heat generated during their operation, either by directly converting it into electricity or by improving efficiency. This advancement would enhance energy efficiency of everyday appliances, reduce reliance on external power sources, and lower electricity expenses.

In the renewable-energy sector, thermoelectric materials with ultrahigh zT values could also play a vital role. Although solar panels are proficient at capturing sunlight, they frequently lose excess heat. By integrating thermoelectric materials into solar panels, we could recover and convert this wasted heat into additional electricity, further boosting the overall efficiency of solar energy systems.

The research conducted by Chen's team, supported by the advanced technology of the **SIKA** Cold Neutron Triple-axis Spectrometer, paves the way for the development of more efficient thermoelectric materials. By understanding how electrons and phonons interact on a microscopic level, we are progressing toward a future where energy is used more efficiently, waste is minimized, and a greater number of devices can be powered by everyday heat. The implications of this research are profound, offering the potential to improve energy efficiency across transportation, industrial production, and daily appliances, all while contributing to a more sustainable and energy-conscious society. (Reported by Chun-Ming Wu)

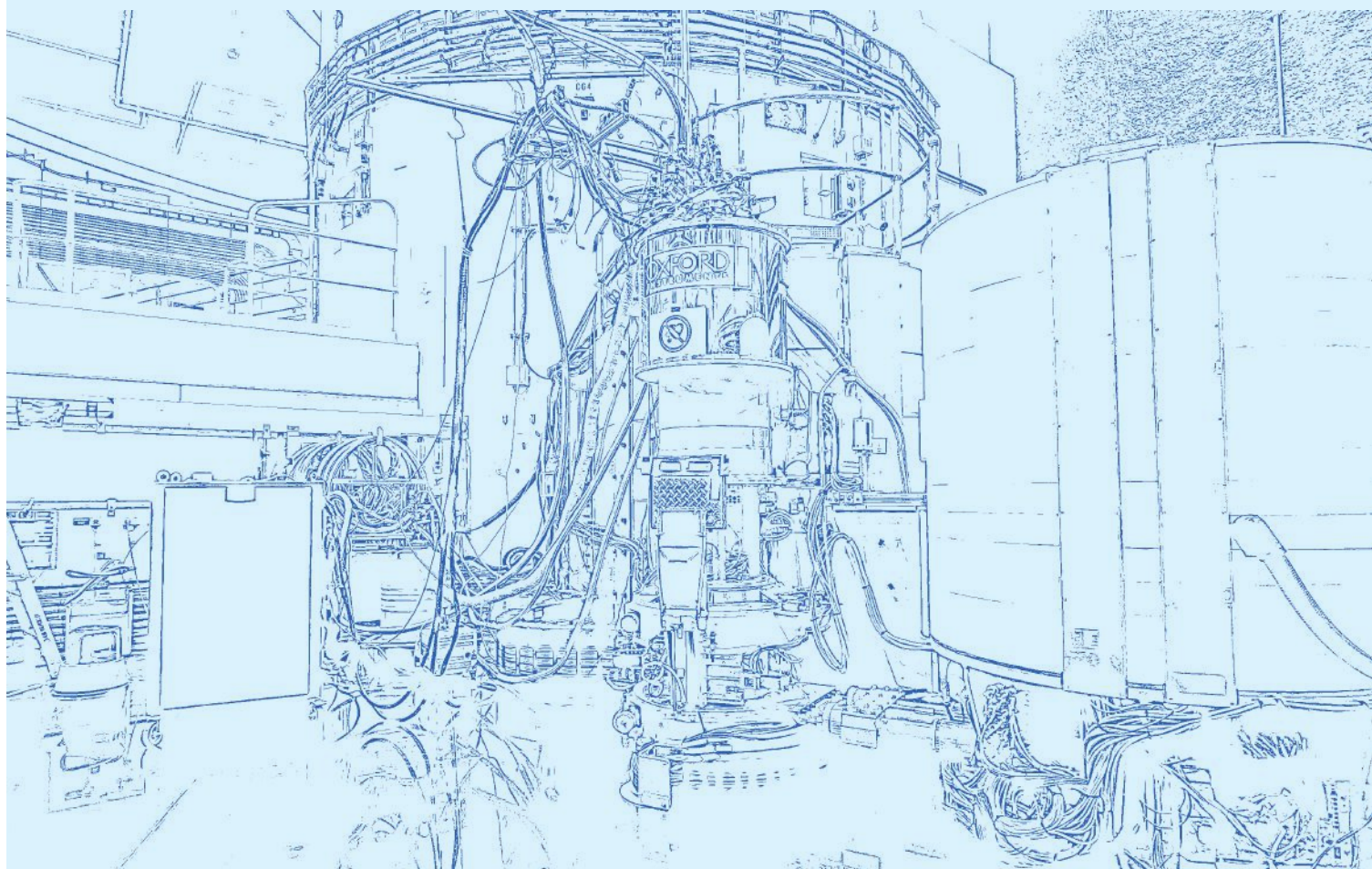
This report features the work of Yang-Yuan Chen and his collaborators published in Energy Environ. Sci. 17, 1904 (2024).

ANSTO SIKA – Cold Neutron Triple-axis Spectrometer

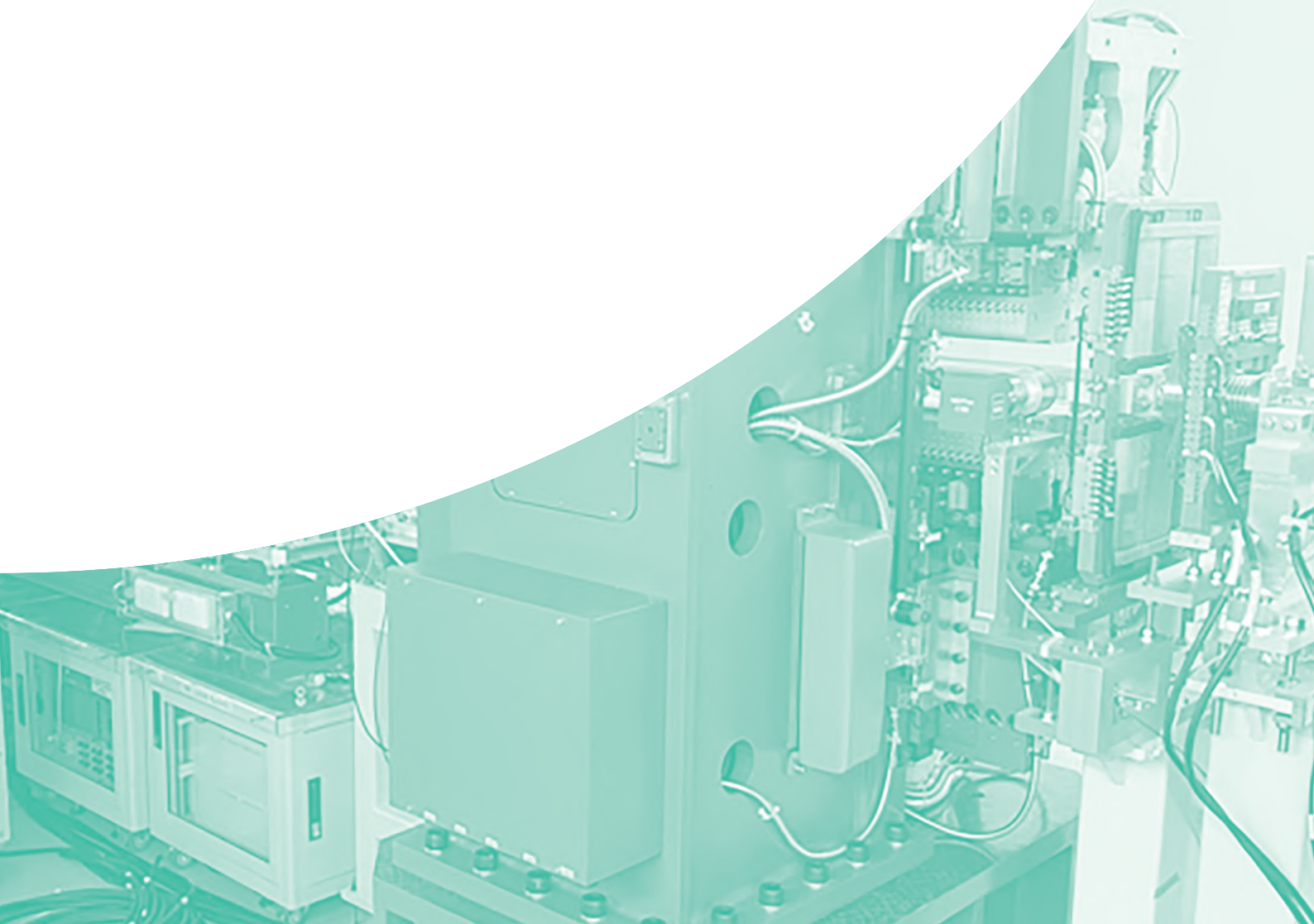
- Inelastic Neutron Scattering
- Thermal Electric Materials, Ultrahigh zT , Condensed Matter, Energy Materials, Quantum Physics

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Facility Development and Status



Status of TLS and TPS Accelerators

Taiwan Light Source (TLS)

Machine Parameters of the TLS

The Taiwan Light Source (TLS) celebrated its 30th anniversary of its first operation in 2023. Since its commissioning in 1993, TLS has invited experimental proposals and opened its facilities to users, initially featuring three soft X-ray beamlines: HSGM, LSGM, and Seya. The original TLS design was based on a triple bend achromatic lattice with a beam energy of 1.3 GeV and a beam current of 200 mA. Following several phases of upgrades, the accelerator has now achieved a beam energy of 1.5 GeV, a maximum stored beam current of 360 mA, top-up injection capabilities, a superconducting radio-frequency (SRF) cavity, a liquid-helium cryogenic system, superconducting wigglers (SCWs), and advanced feedback systems for orbit and bunch-to-bunch stability. Many of these advancements were pioneering and unique in the low-energy synchrotron community. The key parameters of TLS are presented in [Table 1](#).

The storage ring, which is designed with sixfold symmetry, features four room-temperature undulators, one wiggler, and five SCWs, giving resulting in the most densely packed SCW configuration in the community for the TLS. SCWs enable the generation of high-energy photons to support X-ray users. The specifications of the insertion devices are listed in [Table 2](#).

Statistics of TLS Machine Operation

During the initial top-up injection phase, the stored beam current was limited to 200 mA in early 2005 because of the constraints of the radio frequency system capabilities and beam stability. Following the installation of the SRF module and the upgrade of the feedback system, TLS gradually increased the stored beam current to 360 mA after 2010. [Figure 1](#) presents the performance metrics of TLS operations from 2011 to 2024. Availability is defined as the ratio of actual user time to scheduled user time; mean time between failures (MTBF) is defined as the ratio of scheduled user time to the number of system faults; and the beam stability index is evaluated based on photon intensity variation in the diagnostic beamline, maintained within 0.1%.

Table 1: Main parameters of the TLS storage ring.

Beam Energy (GeV)	1.5
Number of Buckets	200
Current (mA)	360
Horizontal Emittance (nm-rad.)	22
Vertical Emittance (pm-rad.)	88
Tunes (ν_x/ν_y)	7.303/4.175
Lifetime (hour)	> 6

Table 2: Main parameters of the insertion devices used in the TLS.

	W200	U50	U90	EPU56	SWLS	SW60	IASWA	IASWB	IASWC
Type	Hybrid	Hybrid	Hybrid	Pure	SC	SC	SC	SC	SC
Period length (mm)	200	50	90	56	250	60	61	61	61
Photon energy (eV)	800–15k	60–1.5k	5–500	80–1.4k	2k–38k	5k–20k	5k–20k	5k–23k	5k–20k



Fig. 1: Annual beam stability index of 0.1%, availability, and MTBF of the TLS.

In 2024, the annual availability of the TLS reached 99.4%, with scheduled user time totaling 4,578 hours, a second-highest MTBF of 286.1 hours, and a beam stability of 99.36%. After switching of the pulsed klystron supplier from Thales to Canon in 2023, the operational reliability and stability of the TLS linear accelerator system have significantly improved in 2024.

Downtime and Failure Analysis of the TLS

In 2024, there were 15 beam trips and a total of 28.35 hours of downtime. The SRF system, which provides high power to the stored beam and operates at 4.5 K, is complex and requires a strict interlock protection system. This system accounted for the largest portion of the annual downtime with a fast recovery time. The contributions from each subsystem of the TLS facility are shown in Figs. 2 and 3. The primary causes of downtime were force majeure and unknown events, including earthquakes and voltage drops at the power station. The second-most common cause of downtime was related to the instrumentation and control group (I&C), including issues such as FOFB, failures in the power supply of the 500 MHz master clock RF amplifier, and ILC failures. Alternative solutions for replacing failed and aging systems are currently being evaluated.

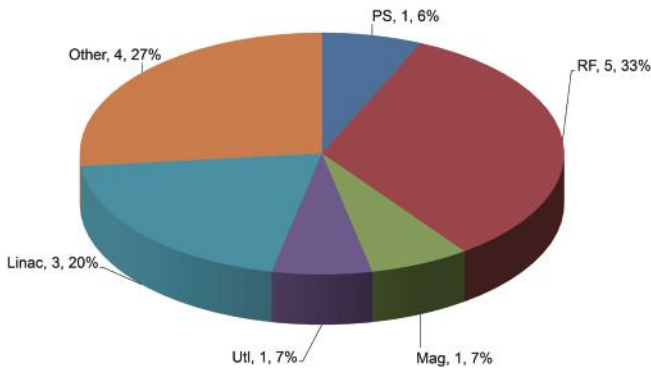


Fig. 2: Proportions of beam trips for the TLS accelerators in 2024 (15 trip events in total). RF stands for radio frequency; PS stands for power supply; and I&C stands for instrumentation & control. “Other” includes 1 earthquake, 1 voltage drop caused by Taiwan Power Company (TPC), and 2 unknown partial beam losses.

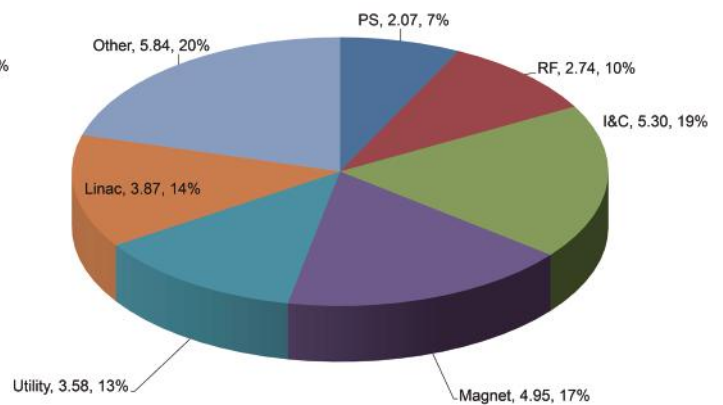


Fig. 3: Downtime distribution for the TLS accelerators in 2024 (totaling 28.35 hours). The major failure times were as follows: other, 5.84 hours; I&C, 5.3 hours; magnet, 4.95 hours; and Linac, 3.87 hours.

Taiwan Photon Source (TPS)

Machine Parameters of the TPS

The Taiwan Photon Source (TPS) has been operational for eight years, having officially been opened to users in 2016. The TPS storage ring incorporates a strong focusing double bend achromatic lattice, which features low emittance, top-up injection, SRF module operation, long straight sections, and high stability. The major parameters of the TPS storage ring for current operation are listed in Table 3. The TPS accelerators consist of concentric storage rings and booster rings within the same tunnel, a design choice made considering the limited space on the campus and energy conservation.

Table 3: Main parameters of the TPS storage ring.

Beam Energy (GeV)	3
Circumference (m)	518.4
Current (mA)	500
Number of Buckets	864
Beam Emittance (ϵ_x/ϵ_y) (nm-rad.)	1.6/0.016
Momentum Compaction (α_1/α_2)	0.0024/0.0021
Tunes (ν_x/ν_y)	26.15/14.23
Lifetime (hour)	> 8

Statistics of TPS Machine Operation

The TPS began operations for users in the last quarter of 2016, with a beam current of 300 mA, which increased to 400 mA in December 2017. The system continued to operate regularly until it reached 450 mA on the last day of 2020. In 2021, the stored beam current reached an operating current of 500 mA. The COVID-19 pandemic caused delays in the delivery of several key components for the Phase-II and Phase-III beamlines. Despite these challenges, through dedicated collaboration between vendors and NSRRC staff, 18 beamlines were available for user operation in 2024.

Figure 4 on the next page shows the scheduled and delivered user times and availability on a quarter-to-quarter basis since 2017. The scheduled user time in 2024 was 4,890 hours. Because of frequent earthquakes in Hualien from April to May, a total of 8 beam trips and a recovery time of 12.01 hours were recorded. As shown in Fig. 5 on the next page, the annual availabilities with and without seismic induction statistics were 97.7% and 97.92%, and MTBF of 163 hours and 221.2 hours, respectively.

Downtime and Failure Analysis of the TPS

In 2024, there were 29 beam trips and a total downtime of 113.36 hours. With the 8 beam trips caused by earthquakes excluded, the contributions of each subsystem within the TPS facility to these beam trips and downtime are illustrated in **Figs. 6 and 7**. The subsystems most frequently involved in beam trips and downtime are the SRF and LINAC systems. The higher failure rate of the SRF system is attributed to sensor aging and solid-state module damage resulting from prolonged operation at a high current of 500 mA. For the LINAC system, the primary issue is the long recovery time caused by klystron failure. Nevertheless, excluding trips caused by earthquakes, the overall reliability of these subsystems has significantly improved in recent years, enabling stable operation and extending the MTBF. (Reported by Hung-Jen Tsai)

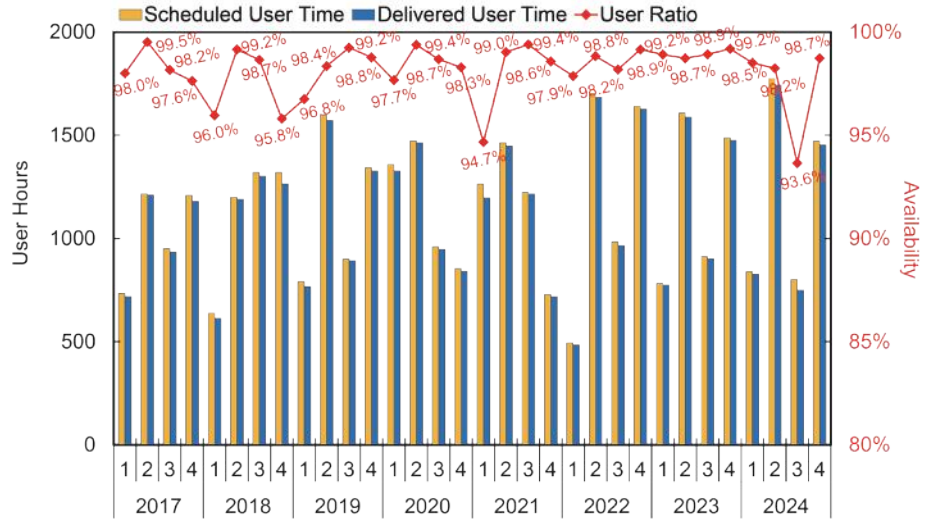


Fig. 4: User time and beam availability of the TPS from 2017 onward.

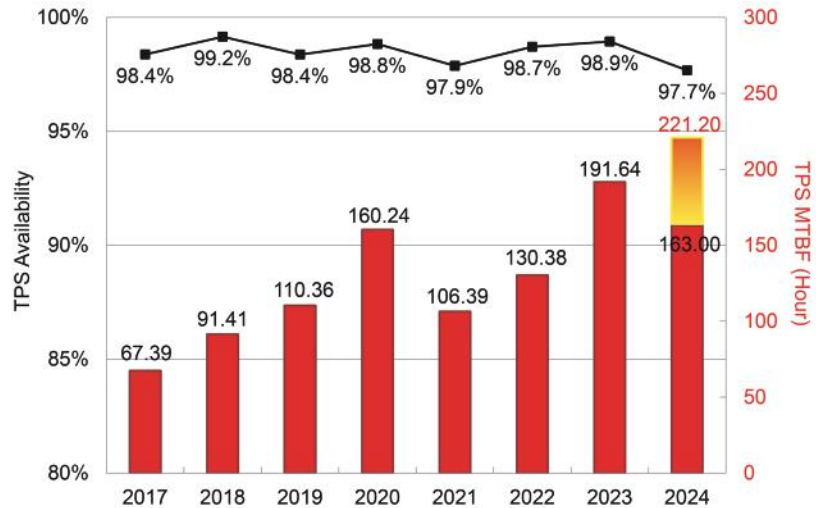


Fig. 5: MTBF and beam trip statistics of the TPS from 2017 onward. In 2024, the MTBF with and without seismic induction statistics was 163 hours and 221.2 hours, respectively.

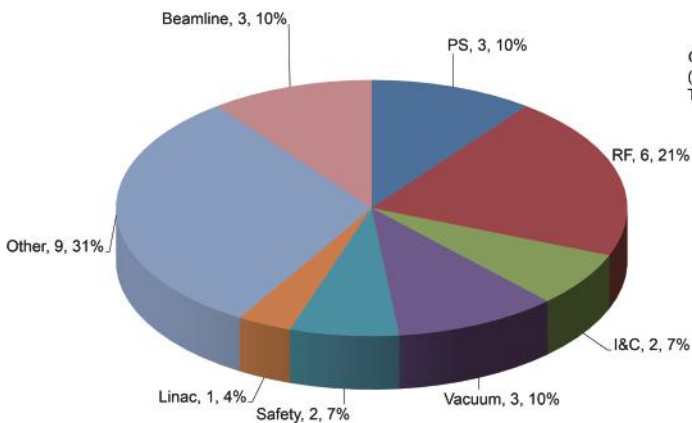


Fig. 6: Proportions of beam trips of the TPS accelerators in 2024. There were 29 trip events in total. "Other" includes 8 earthquake events and 1 TPC voltage drop.

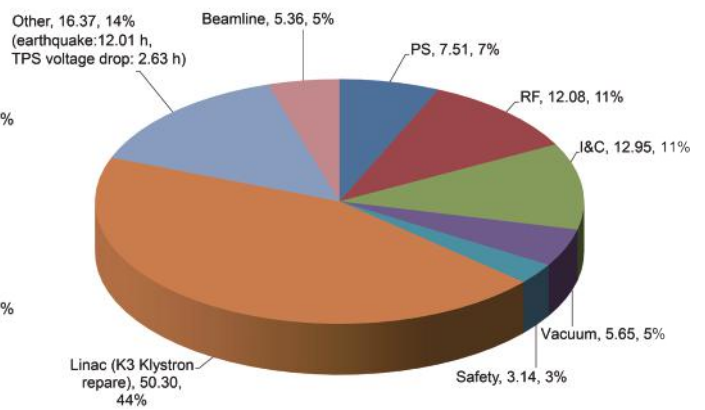


Fig. 7: Proportions of downtime in the TPS accelerators in 2024 (totaling 113.36 hours). The major failure times were as follows: Linac, 50.3 hours; other, 16.37 hours; I&C, 12.95 hours; and RF, 12.08 hours.

Elliptically Polarized Undulator Coupling Correction at the Taiwan Photon Source

Introduction

Five APPLE-II types of elliptically polarized undulators (EPUs) are installed in the Taiwan Photon Source (TPS) to produce elliptically polarized light, namely, EPU46-45, EPU48A-41, EPU48B-41, EPU168-39, and EPU66-27. An APPLE-II-type EPU comprises four identical quadrants of magnet arrays. By enabling the translation of two diagonal quadrants parallel to the magnetic axis while keeping the other two fixed, adjustments can be made to vary the strength of the horizontal and vertical magnetic field components (B_x and B_y), consequently influencing the ellipticity of the electron beam. Because of manufacturing imperfections in the EPU, it generates a skew quadrupole component that varies at different gaps and phases.

The residual skew quadrupole component couple horizontal betatron motion and dispersion to the vertical plane and then results in changes in beam size. When the electron beam encounters the residual skew quadrupole component of the EPU and subsequently traverses a region where vertical acceptance is minimal around the entire ring, there is a risk of beam scraping and loss.

To mitigate the coupling effects from the EPU and to maintain vertical beam size, each EPU is equipped with a pair of skew quadrupole magnets, named upstream skew quadrupole (USQ) and downstream skew quadrupole (DSQ), located at the entrance and exit of the EPU, respectively. This type of coupling correction is similar to the three-

magnet bump used for orbit correction.¹ Since the residual skew quadrupole of the EPU varies with gaps and phases, a 2D coupling feed-forward table that accounts for gap and phase for both USQ and DSQ is needed to compensate for the residual skew quadrupole from the EPU and to maintain the beam size. To build such a 2D coupling feed-forward table, an indicator beam loss monitor (BLM) is used to monitor the variations in the beam size. This method is fast and sensitive. If the beam size exceeds the vertical acceptance level, then the beam will be scraped and lost. Conversely, if the beam size remains smaller within the vertical acceptance, then the electron beam will not be scraped.

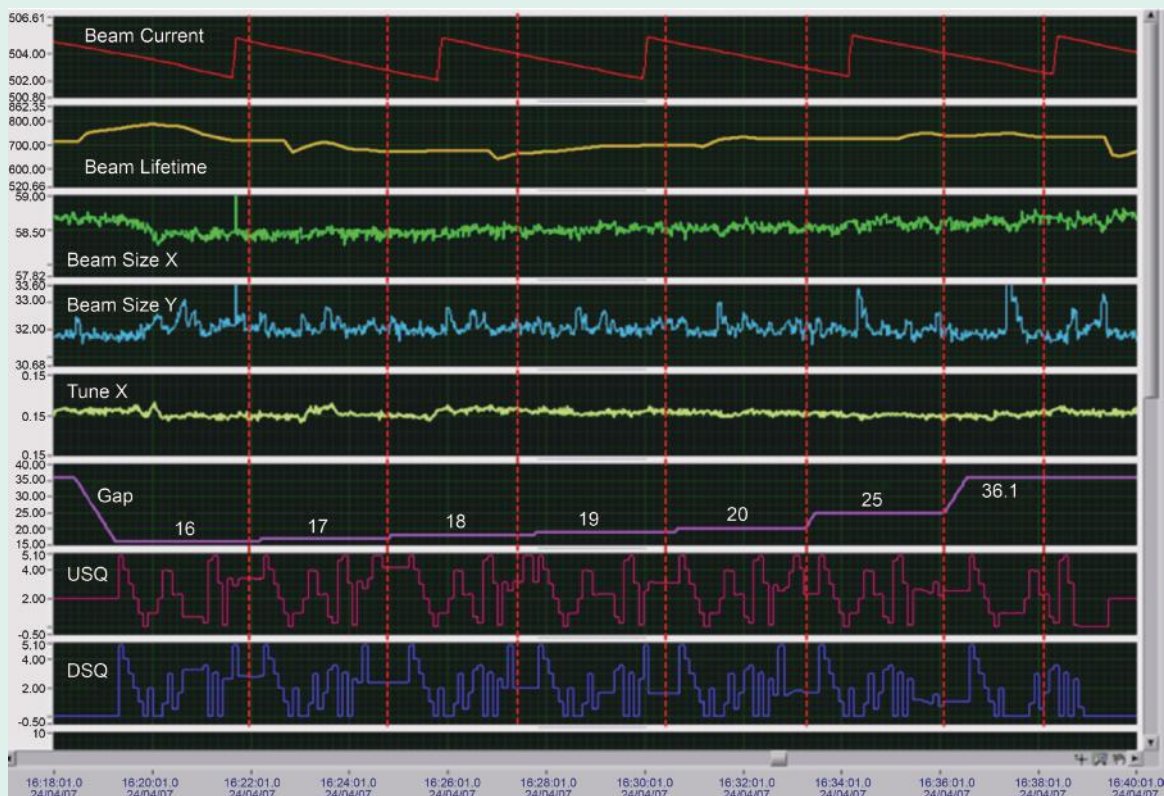


Fig. 1: Optimization process for EPU66-27 coupling feed-forward table by Bayesian optimization.

Coupling Feed-Forward Table

To counteract the undesired coupling effects caused by the residual skew quadrupole component of the EPU, it is essential to construct a 2D coupling feed-forward table based on the gap and phase of the EPU. For this purpose, an automatic measurement program that uses two nested loops to automatically adjust the gap and phase of the EPU has been developed. At each step of the gap and phase, the currents for the USQ and DSQ of the EPU are optimized to minimize the BLM reading using Bayesian optimization. The procedures for building the EPU coupling feed-forward table are outlined as follows:

1. The gaps of all EPUs are ensured to be minimized to their lowest settings to provide appropriate vertical acceptance, while the TPS is operated in top-up injection mode with a beam current of 500 mA.
2. Two nested loops are used to gradually adjust the gap and phase of the EPU to a specific setting and thus modify one gap or phase at a time.
3. For each gap and phase setting, the program automatically adjusts the currents of the USQ and DSQ of the EPU to minimize the BLM reading. The optimization should occur outside of injection periods.
4. Once the minimum BLM reading is achieved, the USQ and DSQ currents corresponding to that gap and phase setting are recorded by the program.
5. This process for each subsequent gap and phase is repeated, with the skew quadrupole currents adjusted and recorded at each step of gap and phase.

This procedure effectively builds the coupling feed-forward table, minimizing the coupling effects across all gap and phase settings of the EPUs. A shell script is then used to convert the 2D feed-forward table into a waveform format to enable

the soft IOC to read it and perform interpolation. This allows the soft IOC to adjust the currents of the USQ and DSQ at 200 Hz when the EPU gap or phase varies.

Figure 1 shows the optimization process of the EPU66-27 coupling feed-forward table through Bayesian optimization. The optimization process is described as follows: The program reduces the gap to 16 mm and the phase to 0 mm. In the beginning, 10 sets of initial points are given (the different sets of USQ and DSQ currents and their associated BLM reading). On the basis of these initial measured points, the Bayesian optimization algorithm varies the USQ and DSQ currents to minimize the BLM reading.

Verification of Coupling Correction by Closest-Tune Approach

After the creation of the 2D coupling feed-forward table, it is important to verify the performance of the coupling correction. The closest-tune approach² was applied to check whether the minimum gap (tune-split) between the two normal modes was minimized after the correction had been applied. This was achieved by simultaneously scanning the currents of 36 defocusing quadrupoles (referred to as QS1) from -0.49 to -0.88 A relative to their

nominal values, with a step size of 0.01 A. These 36 quadrupoles are located on both sides of the 18 short straight sections. At each step, the turn-by-turn data for 7000 turns from 172 beam position monitors were recorded and converted into betatron tunes using fast Fourier transform. A pulsed pinger magnet was used to excite betatron oscillations to ensure that all 172 BPMs were synchronized with the trigger event initiated by the pinger magnet, allowing the TbT signals to be recorded in sync with the revolution period.

The betatron tunes of the two normal modes were plotted against the QS1 currents relative to their nominal values. The normal mode (eigenmode) tunes can be expressed in terms of uncoupled betatron tunes (ν_x, ν_y) and the coupling strength by using the following equation:

$$\nu_{\pm} = \frac{\nu_x + \nu_y - l}{2} \pm \frac{1}{2} \sqrt{\Delta^2 + G_{1,-1,l}^2}$$

, where $\Delta = \nu_x - \nu_y - l$, and $G_{1,-1,l}$ represents the minimum separation between the normal mode tunes. The integer parts of the betatron tunes for the TPS storage ring are 26 and 14 for the horizontal and vertical planes, respectively, resulting in $l = 12$. The minimum gap $G_{1,-1,l}$ between these two normal modes represents the coupling strength.

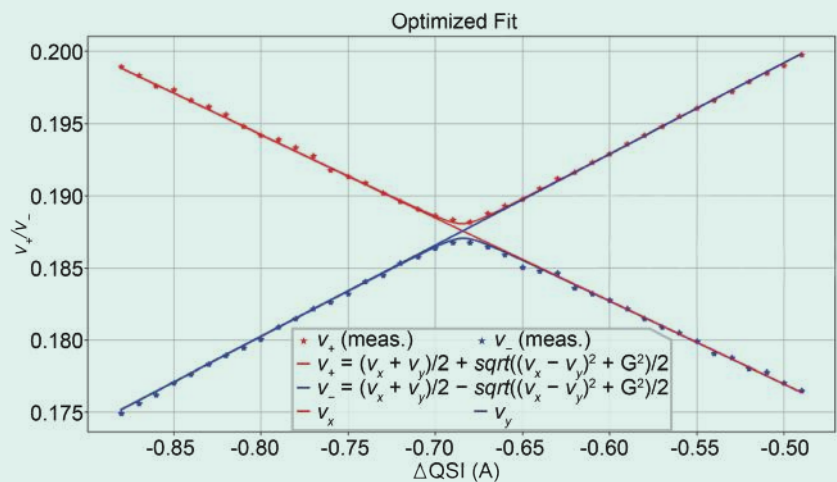


Fig. 2: Measured betatron tunes of two normal modes versus the currents of the quadrupole QS1 relative to their nominal values for all EPU ID gaps that are open.

Figure 2 shows the measured betatron tunes of normal modes against the currents of the quadrupole QS1 relative to their nominal values when gaps of all EPU IDs are open. The tune-split $G_{1,-1,l}$ between the two normal modes is 0.001.

Figure 3 shows the measured betatron tunes of normal modes against the currents of the quadrupole QS1 relative to their nominal values when the EPU66-27 gap is closed to 18 mm without coupling correction. The tune-split $G_{1,-1,l}$ between the two normal modes is 0.0046.

Figure 4 shows the measured betatron tunes of normal modes against the currents of the quadrupole QS1 relative to their nominal values when the EPU66-27 gap is closed to 18 mm after coupling correction by USQ and DSQ. The tune-split $G_{1,-1,l}$ between the two normal modes is reduced from 0.0046 to 0.001.

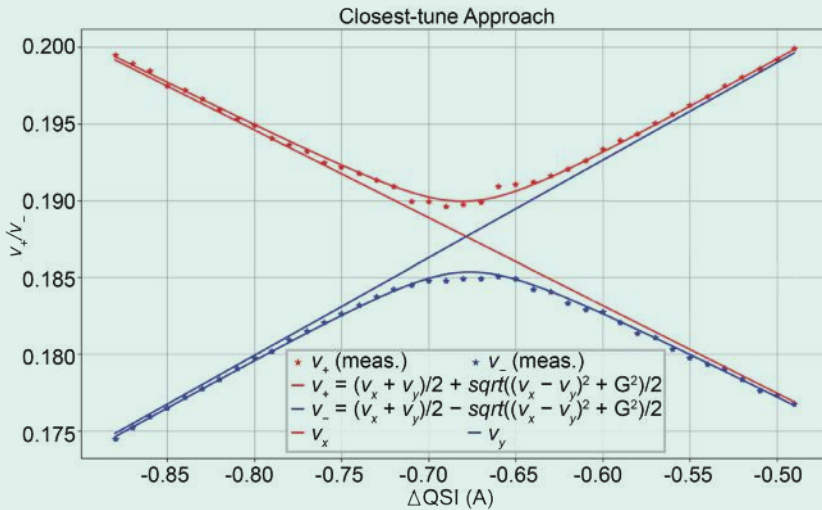


Fig. 3: Measured betatron tunes of two normal modes *versus* the currents of the quadrupole QS1 relative to their nominal values when the EPU66-27 gap is closed to 18 mm.

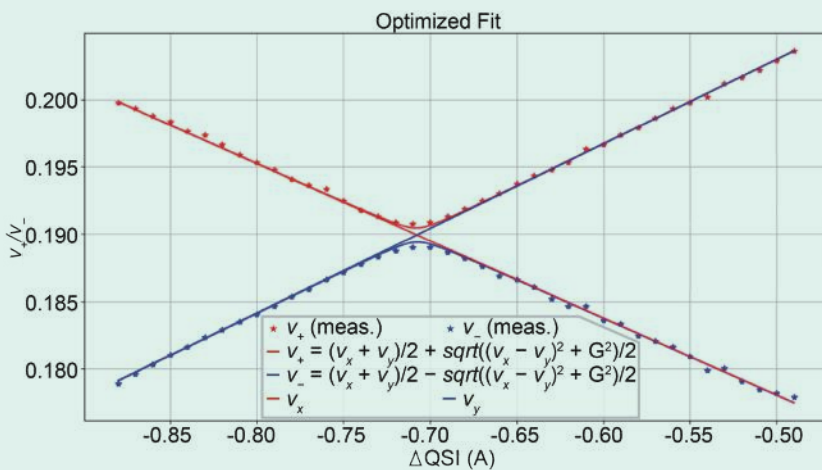


Fig. 4: Measured betatron tunes of two normal modes *versus* the currents of the quadrupole QS1 relative to their nominal values when the EPU66-27 gap is closed to 18 mm after coupling correction by USQ and DSQ.

Conclusion

Manufacturing imperfections in the EPUs inevitably result in the formation of a residual skew quadrupole component. To mitigate the coupling effects caused by the EPUs and to maintain the vertical beam size, each EPU is equipped with a pair of skew quadrupole magnets. This report demonstrates the feasibility of constructing a 2D coupling feed-forward table based on the gap and phase of EPUs to compensate for the coupling effects using Bayesian optimization. After building this 2D table, the closest-tune approach was employed to verify its performance by checking whether the minimum gap between the two normal modes (tune-split) was minimized after the correction had been applied. (Reported by Mau-Sen Chiu)

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The Path to Sustainability: Energy-Saving Achievements and Future Plans of the NSRRC

The concentration of carbon dioxide in the atmosphere rose from 280 ppm before the Industrial Revolution to 420 ppm. Fortunately, humanity has recognized the climate change problem caused by this issue and has been actively transitioning energy systems. This includes using low-carbon natural-gas power generation as a transitional step and introducing zero-carbon renewable energy sources.

The International Energy Agency compiled data on global power generation for 2020 and 2021 and predicted the energy mix for 2030 and 2050. As shown in Fig. 1, while energy demand continues to rise, the share of fossil fuels is expected to drop from 67.5% in 2020 to 25.8% in 2050. Meanwhile, renewable energy is projected to increase from 19.7% to 65.2%,¹ meaning that in 30 years, the roles of renewable and fossil energy will reverse.

Figure 2 illustrates the power usage and share of major facilities and buildings of the NSRRC in summer. The two accelerators, TLS and TPS, are the largest power consumers, with a combined usage of 4,341 kW. This accounts for 45.3% of the NSRRC total power consumption. The top three subsystems of the accelerators in terms of energy usage are the magnet power supply, radio-frequency (RF) systems, and cryogenic systems. Other major power consumers include the utility systems (3,241 kW, 33.8%), building electricity usage (1,378 kW, 14.4%), and beamlines and laboratories (629 kW, 6.6%).

Using the ISO 14064-1 standard, we record the direct carbon emissions and indirect emissions from purchased electricity of the NSRRC, excluding indirect emissions from employee commuting, business trips, supplier transportation, or service provisions. The annual carbon emissions are about 35,265 tons of CO₂ equivalent, with 95.5% stemming from purchased electricity. The remaining 4.5% comes from kitchen liquid petroleum gas burning, emergency generator operation, official vehicle emissions, refrigerant leaks from air conditioning systems, and experimental processes. Therefore, reducing electricity use is key to decreasing carbon emissions.

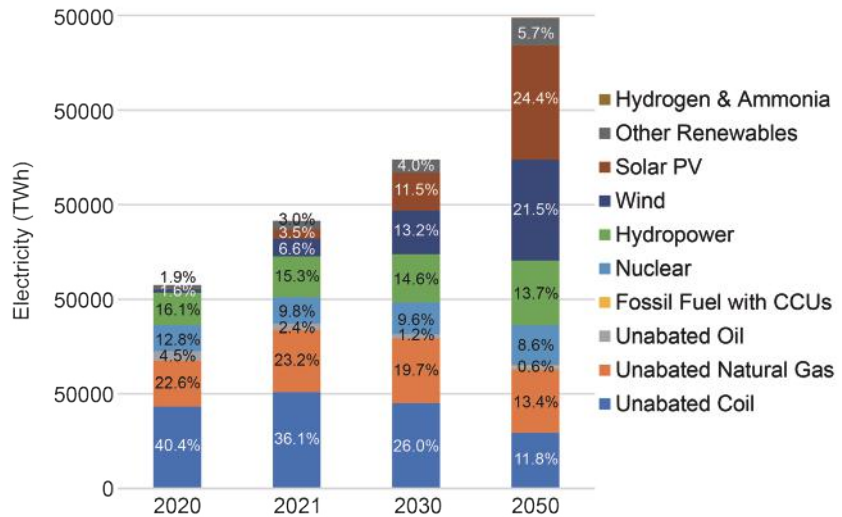


Fig. 1: Global power generation by energy type and percentage.¹

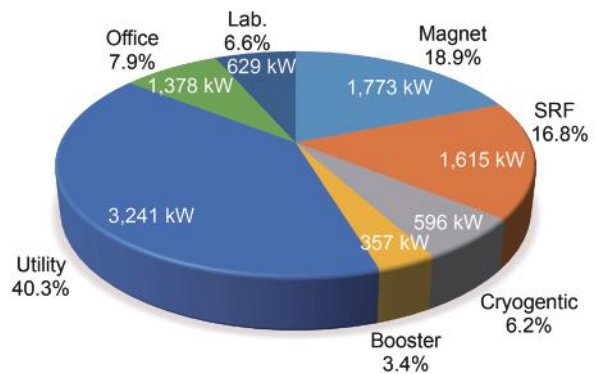


Fig. 2: Power usage and share by major facilities and buildings in summer 2021 at the NSRRC. [Reproduced from Ref. 2]

From previous energy-flow statistics, it is clear that the accelerators are the most significant power consumers. However, before implementing energy-saving measures, we must consider whether the chosen technologies can affect reliability, safety, investment costs, and economic returns from lower electricity bills. Despite these challenges, recent breakthroughs have been made, such as the energy-efficient operation of booster ring dipole magnets and the implementation of solid-state RF systems in the TPS. Other advancements, such as using permanent magnets, have improved accelerator performance while saving energy, promising further progress.

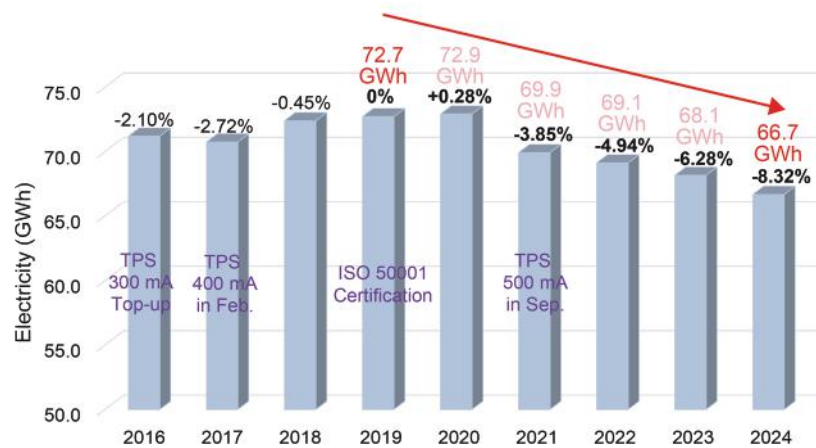


Fig. 3: Yearly electricity usage statistics for the NSRRC. [Reproduced from Ref. 2]

The second-largest power consumer is the utility system. During the construction of the TPS, energy-saving features like variable-frequency drives for pumps, air-conditioning fans, and air compressors were planned. The NSRRC recently implemented an energy management system, earning ISO 50001 certification. This recognition highlights the energy-saving efforts of the NSRRC and supports the development of new techniques, including:

- A. **Optimizing Chiller Operations:** Chillers are the core of air conditioning and accelerator cooling systems and the most energy-intensive machines. Connecting the cooling systems with chilled water pipelines across the TLS and TPS allows chillers to be centrally managed for optimal operation and energy efficiency.
- B. **Heat Pump Energy Recovery:** Waste heat from various equipment and air-conditioning systems can be recovered using heat pumps. This recovered heat can reheat dehumidified cool air to provide the dry, room-temperature air, reducing energy consumption from electric heaters by 70%.
- C. **Upgrading to light-emitting diode (LED) in the TPS Experimental Hall:** Previously, this area used 384 metal halide lamps, each rated at 400 W. These have been replaced with 153 W LED lights, leveraging improvements in LED lighting technology.

Despite the addition of new equipment and record-high accelerator operation hours, our net electricity usage has decreased yearly thanks to collective efforts. As shown in Fig. 3, starting from the TPS's official launch in 2016, and using 2019 as the baseline, the annual electricity consumption of the NSRRC dropped from 72.7 GWh (about 7% of Hsinchu City's household electricity usage) to 66.7 GWh in 2024. This reduction saves at least 6.0 GWh annually, equivalent to TWD 26.5 million in electricity costs. Additionally, the NSRRC has installed solar panels with a total capacity of 1,187 kWp on building

roofs, generating about 1.5 GWh of renewable energy yearly—2.1% of our annual electricity usage. Although the electricity generated is sold to the Taiwan Power Company and cannot be counted toward energy savings or carbon reductions, it provides considerable revenue for the NSRRC.

Saving energy protects the environment and addresses practical concerns such as rising electricity costs and potential carbon fees. From July 2022 to present, electricity rates have increased by 74.8%, and this year's summer rates were extended for an additional month. Energy costs have also been driven up by the war in Ukraine, and carbon fees may be introduced in the future. Under this stress, the NSRRC will continue to implement energy-saving measures such as:

- A. Detecting pipeline leaks
- B. Optimizing operational parameters of mechanical equipment
- C. Recovering heat energy
- D. Replacing outdated equipment
- E. Improving building energy efficiency³
- F. Maintaining heat exchangers
- G. Generating renewable energy
- H. Utilizing electrical energy or ice storage systems

By taking these actions, we aim to fulfill our corporate social responsibility and accelerate the transition to green energy. (Reported by Wen-Shuo Chan and Chin-Kang Yang)

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Design and Fabrication of a 4 K Helium Phase Separator

Liquid helium (LHe) is transported from the cryogenic system to superconducting devices through multi-channel transfer lines. However, unavoidable heat loss during transmission causes the LHe to transition into a two-phase flow, which can significantly impact the performance of cryostats in superconducting or other devices not continuously filled with LHe. Helium phase separators were developed at the NSRRC to re-condense the two-phase helium flow from a liquid-helium transfer line and ensure a stable supply of LHe to users.^{1,2}

A G-M cryocooler (Sumitomo, model RDK415D) with a cooling capacity of 1.5 W at 4.2 K was integrated into the phase separator to re-condense and liquefy helium while storing it in a 100 L vessel. Practical improvements to reduce the heat load on the helium phase separator were also discussed and implemented.

Figure 1 depicts the configuration of the helium phase separator.^{3,4} The second-stage cold head of the cryocooler, which provides a maximum cooling capacity of 1.5 W at 4.2 K, is connected to an oxygen-free copper condenser mounted at the top of the 100 L LHe vessel. To minimize heat transfer through radiation and convection, the LHe vessel is wrapped in multi-layer insulation (MLI) and housed within a vacuum vessel.

The vessel is supported by four G-10 fiberglass-epoxy rods installed at its base, with the opposite ends secured to the outer vessel to bear the weight of the LHe. Three transfer pipes at the top of the vessel act as inlets and outlets for LHe and as a vent for gaseous helium. Vacuum barriers are placed between the transfer pipes and the outer vessel to mitigate conductive heat loss from room temperature.

A thermal shield made of 30 layers of MLI is connected to the first-stage cold head of the 4 K cryocooler. Positioned between the outer vessel and the LHe vessel, this shield blocks radiative heat loss. Additionally, a thermal bridge made of copper connects the thermal shield to the cryogenic valves, further reducing conductive heat loss. The thermal shield and bridge are constructed of copper, while the outer vessel, LHe vessel, and transfer pipes are made of stainless steel, ensuring durability and effective thermal isolation.

Figure 2 illustrates the piping and instrumentation diagram of the helium phase separator, outlining its operational process. In the first step, the thermal shield was cooled to below 80 K using the first-stage cold head of the cryocooler. Helium gas from the bundle entered the LHe vessel through the pre-cooler, where its temperature was initially reduced. Further cooling occurred *via* heat exchange in the condenser, which was thermally connected to the second-stage cold head of the cryocooler, which brought the helium to its liquefaction temperature.

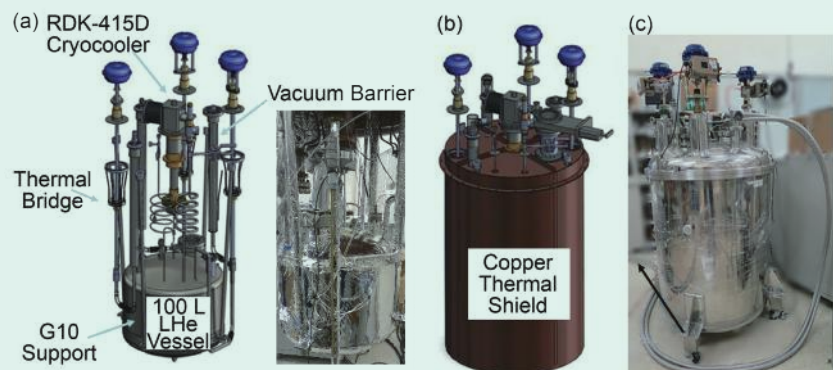


Fig. 1: Configuration of the helium phase separator. (a) Components after removal of the thermal shield; (b) outer vessel removed to reveal the thermal shield; (c) prototype of the helium phase separator. [Reproduced from Ref. 3]

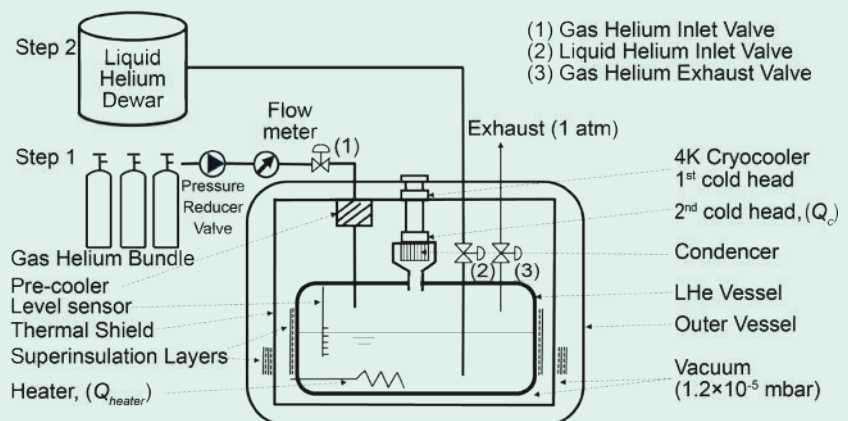


Fig. 2: Configuration of the experiment. Step 1: the separator was cooled with the cryocooler. Step 2: the LHe vessel was filled with LHe; the inner heater was then activated to ensure stable operation in this closed system. [Reproduced from Ref. 3]

The pressure in the LHe vessel was maintained at approximately 1.67 bar-a, and key temperatures, including those of the thermal shield, LHe vessel, condenser, and cold head, were monitored. This was done to confirm that the condenser temperature had reached or dropped below the helium liquefaction temperature of 4.8 K at 1.67 bar-a. Achieving this condition ensured helium liquefaction and confirmed that the separator's heat load was within the cryocooler's cooling capacity. This step established the initial cooling and liquefaction process critical for the subsequent liquid-helium operations.

In the second step, LHe was transferred from the dewar to the LHe vessel under the established operating conditions. During the transfer, a portion of the LHe was naturally consumed and vented to the atmosphere through the exhaust valve, which remained open to allow for pressure regulation.

Once the desired operating level of LHe in the LHe vessel was reached, all inlet and exhaust valves were closed, converting the system into a closed configuration. To maintain stable operating conditions, a heater installed in the LHe vessel was activated. The heater's power was precisely adjusted to stabilize both the LHe level and the internal pressure of the vessel. This heating power played a critical role in determining the overall heat load of the system, providing the valuable data for evaluating the thermal performance and efficiency of the helium phase separator.

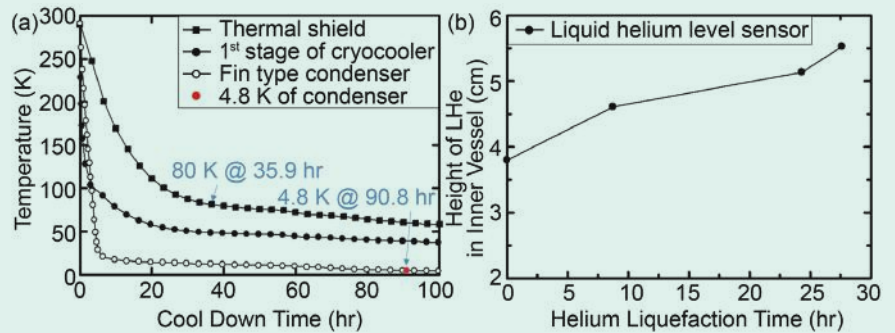


Fig. 3: Experimental results. (a) Cooling-down curve; (b) helium liquefaction rate. [Reproduced from Ref. 3]

Table 1: Thermal parameters.

m	T _e	h _e	T ₁	h ₁	Q ₁	h _{2G}	h _{2L}	Q _s	Q _L	Q _{2nd}	Q _{1st}
(g/s)	(K)	(J/g)	(K)	(J/g)	(W)	(J/g)	(J/g)	(W)	(W)	(W)	(W)
0.0026	295	1532	54	281	3.25	14.06	-1.34	0.69	0.04	0.73	13.8

Helium liquefaction: **Figure 3(a)** illustrates the cooling process of the helium phase separator. The temperature of the thermal shield dropped below 80 K after approximately 35.9 hours. The condenser temperature reached the liquefaction point of 4.8 K at 1.67 bar-a after approximately 90.8 hours, as indicated by the red point in **Fig. 3(a)**. This marked the initiation of the helium liquefaction process. **Figure 3(b)** shows the measured helium liquefaction rate, which was approximately 1.4 cm/day, equivalent to approximately 1.8 L/day. The associated thermal parameters were calculated based on data obtained from the *Helium Material Handbook* and relevant thermodynamic principles, as summarized in **Table 1**. These data provide insights into the thermal efficiency and performance of the separator during its operational cycle.

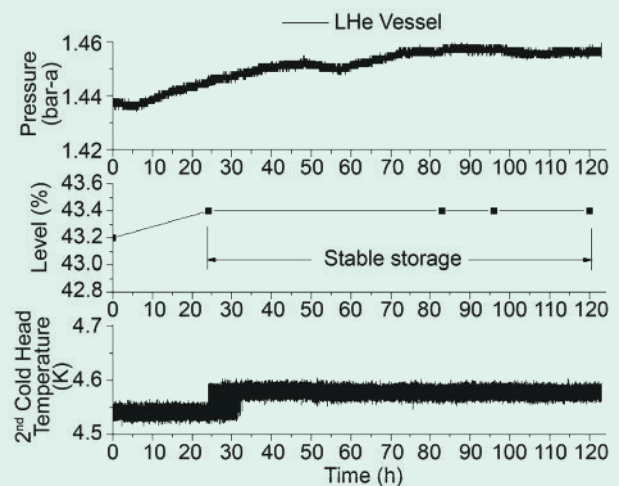


Fig. 4: LHe filling and stable storage. [Reproduced from Ref. 3]

LHe storage: **Figure 4** illustrates the storage process of LHe. The LHe level was maintained at 43.4% by activating the internal heater, which was operated at a power of 0.337 W. The internal pressure was stabilized at 1.46 bar-a ± 0.015 bar throughout the storage period. During this process, the condenser effectively re-condensed the

Table 2: Experimental measurements.

Q _{heat} (W)	LHe vessel pressure (bar-a)	Level (%)	First cold-head temperature (K)	Second cold-head temperature (K)	Q _c (W)	Q _{load} (W)
0.337	1.46	43.4	35.6	4.59	1.892	1.555

vaporized helium, maintaining a steady state. This stability was sustained for a duration of 96 hours, demonstrating that the heat load of the separator was consistently below the cooling capacity of the cryocooler. This ensured efficient and reliable operation of the helium phase separator during the storage phase.

The experimental data are summarized in **Table 2**. The results indicate that the cooling capacity of the second-stage cold head (Q_c) was approximately 1.892 W,⁵ as derived from the cryocooler load map shown in **Fig. 5**. The electrical power of the heater (Q_{heat}) was measured to be 0.337 W. Using the equation $Q_{load} = Q_c - Q_{heat}$, we calculated the heat load of the LHe vessel Q_{load} , to be 1.555 W. This analysis highlights the effectiveness of the cryocooler in maintaining thermal stability while accounting for the heat input from the heater.

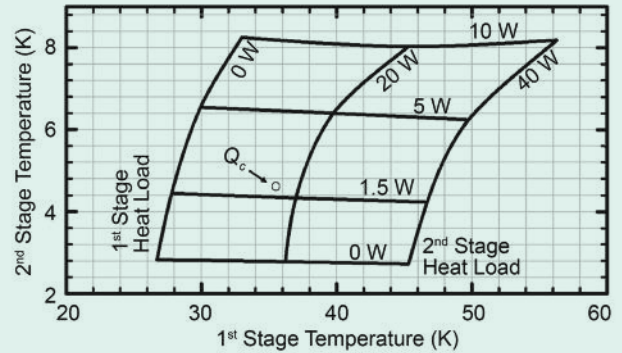


Fig. 5: RDK-415D cold-head load map (60 Hz). [Reproduced from Ref. 5]

During the transmission process, LHe was prone to vaporization due to the heat, leading to the formation of a two-phase fluid within the transmission pipeline. This phenomenon significantly reduced transmission efficiency, hampering the delivery of sufficient LHe and causing instability in user systems.

To address this challenge, the cryogenics group developed a cryogenic freezer re-condensing LHe phase separator. The primary function of this system was to re-condense the two-phase fluid in the pipeline and re-liquefy the separated low-temperature helium gas. The output from the phase separator was high-purity LHe, which effectively minimized the dryness of the LHe in the transmission pipeline, improving the overall stability of the cryogenics system.

The successfully developed re-condensation LHe phase separator could be coupled with a 4 K cryogenic cryocooler to enable zero-boiling operations to condense low-temperature helium. The special design of the radiation isolation baffle further reduced the heat load. In addition to condensing low-temperature helium and storing LHe, the system could convert normal-temperature helium (295 K) into LHe (4 K), functioning as a small-scale liquid-helium production machine.

The system's net cooling capacity was approximately 0.337 W at 4.59 K, with a LHe production rate of approximately 1.8 L per day. The total heat loss was measured at approximately 1.555 W at 4.59 K, showcasing the system's efficient performance. (Reported by Wen-Rong Liao and Chin-Kang Yang)

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Commissioning of Soft X-ray Nanoscopy Beamline at the Taiwan Photon Source

Commissioning of a soft X-ray nanoscopy beamline at the Taiwan Photon Source 27A (TPS 27A) is currently underway. Powered by an elliptically polarized undulator, EPU66, and a newly designed active-mirror plane-grating monochromator (AMPGM), the TPS 27A beamline is capable of delivering a photon beam with high energy-resolving power at a constant beam size.¹ Here, we report the commissioning status of beamline TPS 27A and its two microscopy endstations: the scanning transmission X-ray microscope (STXM) and photoelectron microscope (photoelectron-related imaging and nano-spectroscopy, PRINS). The STXM endstation is partially opened to users as of the end of 2024, and the PRINS endstation is expected to reach the same status by late 2025.

I. Beamline TPS 27A and 27A1 STXM Endstation

The TPS 27A Soft Nanoscopy beamline is designed to deliver a high resolving power and photon flux across a broad photon energy range. To achieve this, a specially developed in-house AMPGM monochromator system with three plane gratings has been implemented. This design supports an energy range from 90 to 3000 eV, with the three gratings covering specific subranges: 90–320 eV, 280–1060 eV, and 1000–3000 eV. Additionally, with the EPU66 undulator system, the photon polarization can be tuned to horizontal, vertical, left-circular, and right-circular states. The beamline is currently in the commissioning stage, which is conducted at the TPS 27A1 STXM endstation. The first X-ray absorption spectroscopy (XAS) of nitrogen gas has been successfully demonstrated, as shown in Fig. 1. The nitrogen K-edge absorption spectroscopy results reveal five distinct vibrational levels in the $N1s \rightarrow 1\pi_g^*$ transition. Fitting analysis indicates that the full width at half maximum (FWHM) for Gaussian and Lorentzian components are 32 and 110 meV, respectively. This corresponds to a resolving power of 12,500 at 400 eV, which aligns well with the expected performance. Currently, the commissioning of the 280–1060 eV energy range is nearly complete. Testing for the 90–320 eV range is planned for the first half of 2025, followed by the testing of the 1000–3000 eV range in the second half of the year.

The TPS 27A1 endstation is designed to perform XAS-related chemical mapping in transmission mode. Its core components include the Zone Plate (ZP) and scanning stages. The STXM endstation is equipped with multiple scanning systems, including stepping motors, piezo walking stages, and piezo actuators with a laser interferometer feedback system. These systems together maintain the relative position between the ZP and the sample, ensuring nanometer-level positioning performance during energy changes and scans, as illustrated in Fig. 2. Due to the fundamental properties of ZPs, where the focal length is proportional to the photon energy, the system is carefully designed and fine-tuned to maintain the imaging area with less than 1 μm lateral movement across the photon energy range of 280–1060 eV.

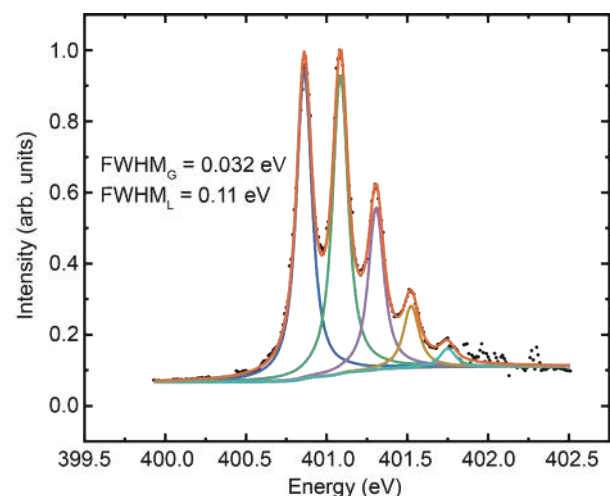


Fig. 1: Nitrogen K-edge absorption spectrum, showing five distinct vibrational levels in the $N1s \rightarrow 1\pi_g^*$ transition. The fitted curve highlights the high-resolution capability of the beamline.

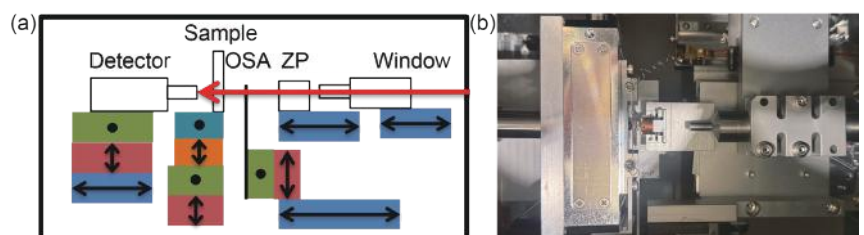


Fig. 2: (a) Schematic representation of the STXM endstation, illustrating the multiple scanning stages, including stepping motors, piezo walking stages, and piezo actuators. (b) Top-view photograph of the STXM endstation, showing the physical layout of the ZP and scanning stages. These components function together to maintain nanometer-level positioning accuracy during energy changes and scans.

Chemical mapping using the spectroscopy capabilities of the STXM endstation is illustrated in **Fig. 3**. The sample consists of Cu nanoparticles (NPs) doped into $g\text{-C}_3\text{N}_4$. The STXM elemental mapping, as presented in **Fig. 3(a)**, was conducted at the Cu L-edge and N K-edge. The green region corresponds to the nitrogen signal, while the red region represents the copper signal. These signals were obtained by calculating the difference between the π^* C-N-C peak and the pre-edge for nitrogen, and the Cu main peak and the pre-edge for copper. The corresponding spectra from the Cu L-edge and N K-edge are shown in **Figs. 3(b) and 3(c)**, respectively. The spectrum shown in **Fig. 3(b)** was acquired from the red regions in the elemental map, which highlight the copper signal. Similarly, the spectrum in **Fig. 3(c)** was obtained from the green regions, which represent the nitrogen signal. These results demonstrate the powerful capabilities of STXM in combining high-resolution microscopy with detailed spectroscopic analysis.

This year, the STXM endstation successfully achieved its first light, marking a significant milestone in its development. Through the current data and commissioning results, we have successfully demonstrated the powerful capabilities of the STXM endstation in delivering high-quality nanoscale imaging and chemical mapping. These achievements validate the endstation's readiness to support the advanced research across diverse scientific fields. This cutting-edge STXM endstation is the result of a collaborative effort between the Department of Physics at Tamkang University and the NSRRC, exemplifying the strength of cross-institutional collaboration in driving scientific innovation. To celebrate this milestone, the Opening Ceremony for the **TPS 27A1** Nanoscopy Beamline and STXM Endstation was held in late December 2024. Moving forward, commissioning efforts will continue in 2025 to further expand the operational energy range and enable more advanced experimental methodologies.

II. TPS 27A2 PRINS Endstation

The **TPS 27A2** endstation aims to perform photoelectron-related imaging and nano-spectroscopy, and its core is

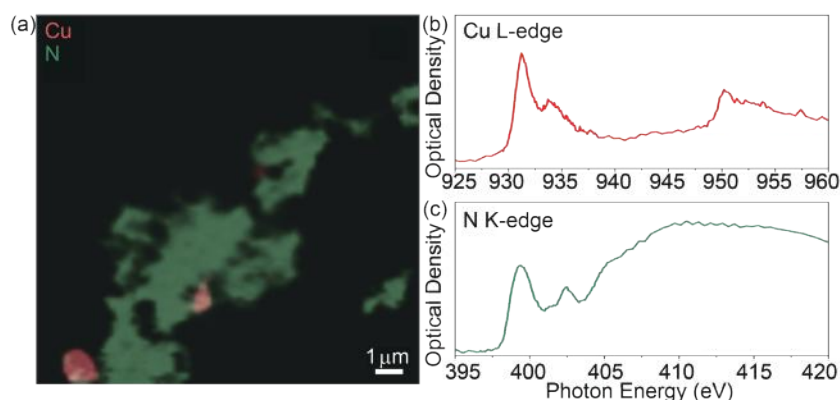


Fig. 3: (a) Elemental mapping of Fe and N for Cu NP/ $g\text{-C}_3\text{N}_4$. (b,c) Corresponding XAS spectra obtained from the red and green regions, respectively.

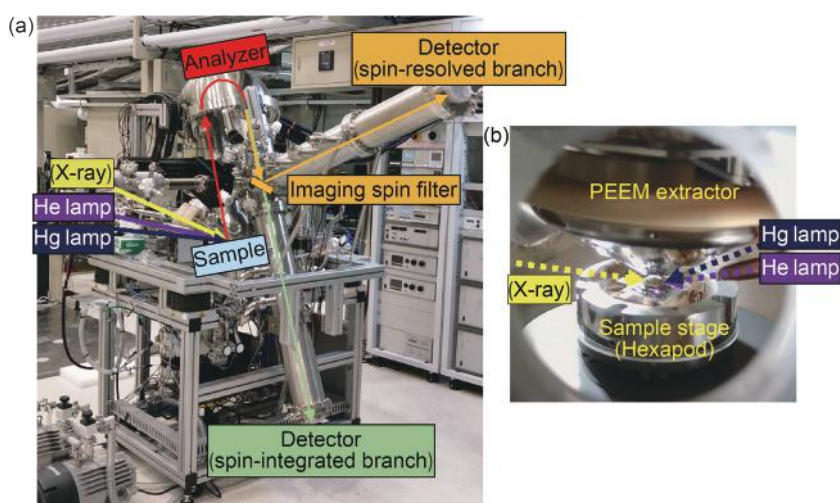


Fig. 4: (a) Configuration of the photoelectron microscope system at the **TPS 27A2**. The beam paths of photoelectrons are indicated by green arrows for the spin-integrated branch or by orange ones for the spin-resolved branch. (b) Image taken close to the hexapod sample stage, extractor lens, and the capillary of the helium discharge lamp. [Reproduced from Ref. 2]

a photoelectron momentum microscope (MM) system, which is capable of obtaining direct-space imaging, momentum-space imaging, and photoelectron spectroscopy with position sensitivity.¹ The off-line commissioning utilizing both ultraviolet (UV) He discharge lamp and Hg arc lamp has been initiated from mid-2022, and the off-line commissioning results have been reported in early 2024.²

The MM system configuration is shown in **Fig. 4**. All light sources, including the soft X-rays and UV lamps, are incident at an angle of 22° relative to the sample surface. The excited photoelectrons are extracted by the extractor lens and projected onto 2D detectors either along the spin-integrated branch or the spin-resolved branch when an imaging spin filter is introduced.

The spatial resolution of direct-space images was tested by analyzing the intensity profile measured on a standard checkerboard-patterned specimen illuminated by the Hg lamp, and the results are shown in **Fig. 5**. The largest field of view (FoV) is approximately $700\ \mu\text{m}$ (**Fig. 5(a)**), and the

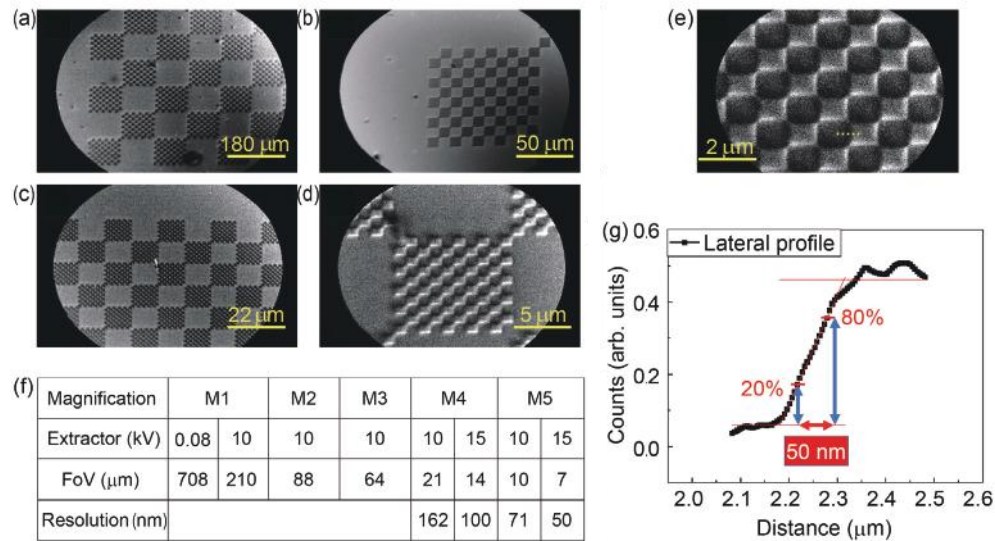


Fig. 5: (a–e) Direct-space photoelectron images of a checkerboard-patterned sample with different FoVs illuminated by the Hg lamp. (f) Table of imaging magnification settings, corresponding FoVs, and resolution. (g) Intensity profile along the edge of the Au patterns as a dashed line in (e). [Reproduced from Ref. 2]

smallest FoV is approximately $7 \mu\text{m}$ (Fig. 5(e)). Various magnification settings and the corresponding FoVs are listed in the table of Fig. 5(f). The spatial resolution was estimated to be 50 nm by analyzing the intensity profile shown in Fig. 5(g) along the edge of the Au patterns, as indicated by a dashed line in Fig. 5(e).

The momentum-space imaging was tested on a Au(111) single-crystal surface by He(I) radiation (21.2 eV), and the results are summarized in Fig. 6. A series of momentum-space images exceeding the first Brillouin zone taken at different binding energies are recorded and shown in Fig. 6(a). After stacking all constant-energy contours together to construct a 3D dataset (k_x, k_y, E_B), which is shown in Fig. 6(b), the electronic band structure along any high-symmetry directions can be obtained simultaneously.

In early 2025, more capabilities can be explored using soft X-rays covered by the TPS 27A beamline, including the imaging based on XAS, X-ray photoelectron spectroscopy, and X-ray magnetic circular/linear dichroism, which can provide additional element-resolved and magnetization-resolved information. (Reported by Hung-Wei Shiu and Tzu-Hung Chuang)

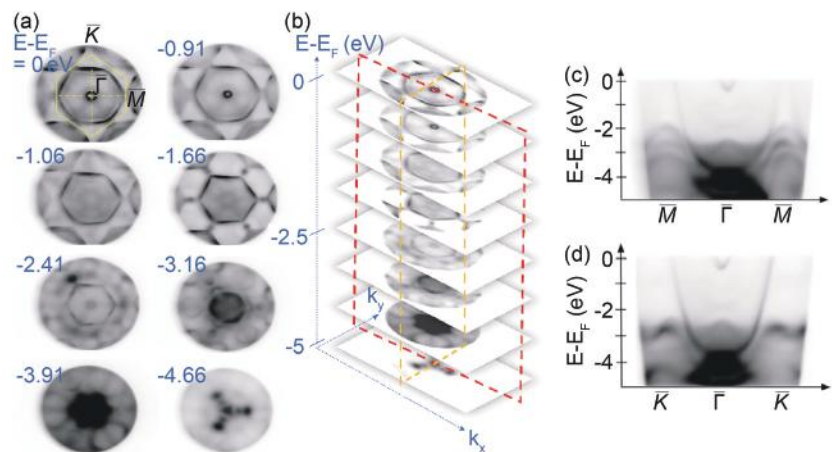


Fig. 6: (a) Momentum-space images recorded at different binding energies obtained from a Au(111) surface at 300 K illuminated by He(I) radiation. (b) Stacking of a series of momentum images with various binding energies, forming a 3D dataset of (k_x, k_y, E_B). (c,d) Slice of the 3D dataset along high-symmetry points of M- Γ -M and K- Γ -K, respectively. [Reproduced from Ref. 2]

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Tender X-ray Spectroscopy Beamline at the Taiwan Photon Source

A tender X-ray beamline, **TPS 32A**, which is part of Phase III of the Taiwan Photon Source (TPS) construction project, has officially opened to users for the 2024-2 cycle. The techniques involve X-ray absorption spectroscopy (XAS) and hard X-ray photoelectron spectroscopy (HAXPES), which are essential tools for investigating material properties at atomic and electronic levels. The **TPS 32A** beamline offers a photon energy range of 1.7–11 keV and delivers a photon flux of 10^{12} photons per second at 6 keV. It has two back-to-back double-crystal monochromators (DCMs) that include both Si (111) DCM and InSb (111) DCM configurations. These DCMs enable switching between configurations to meet user requirements.^{1,2} This design ensures enhanced flexibility, particularly for research teams conducting silicon (Si) element measurements. This beamline can measure elemental XAS spectra, including the K-edges of elements from Si to Zn, as well as the L-edges of second-row and third-row transition metals. Additionally, the use of Kirkpatrick–Baez (K–B) focusing mirrors enables the beam to be reduced to a micron-scale spot size, supporting micro-XAS with spatial resolution. These capabilities address the needs of fundamental research and advanced technological innovation, providing cutting-edge experimental facilities and research resources to academia and industry while opening new scientific frontiers.

The **TPS 32A** beamline integrates HAXPES and XAS techniques, providing users with detailed information on the conduction and valence band electronic structures of materials. As shown in **Fig. 1**, it is divided into four experimental stations: the HAXPES endstation at the focal point, the tender XAS endstation, the hard-XAS endstation, and the micro-XAS endstation.

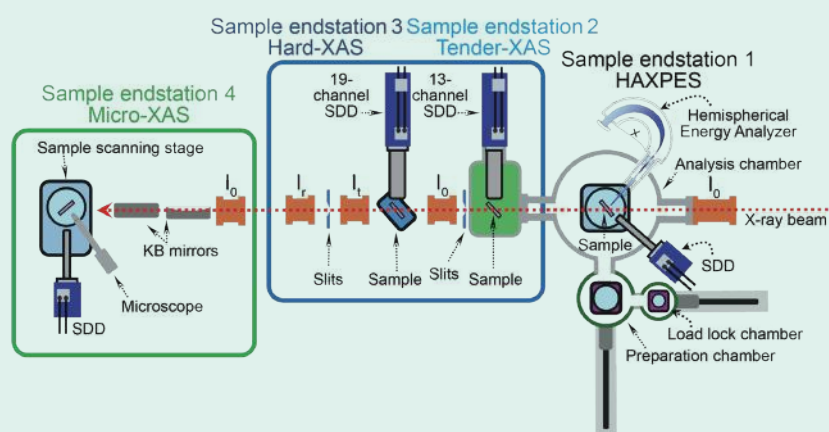


Fig. 1: Layout of the TPS 32A endstations.

The HAXPES endstation, as illustrated in **Fig. 2**, includes a load-lock chamber, a preparation chamber, and an analysis chamber. These chambers maintain ultra-high vacuum conditions exceeding 10^{-10} Torr. The analysis chamber, which is equipped with a high-performance energy analyzer, can detect photoelectron energies up to 6 keV. By adjusting the incident synchrotron tender X-ray with special photon energies, researchers can access deeper core levels and vary the probing depth of the photoelectron spectra. This capability allows for a comprehensive analysis of the valence band composition and electronic structures of surfaces, heterointerfaces, and bulk regions. In addition, a 7-channel silicon drift detector (7ch-SDD) will be installed in this vacuum chamber to facilitate XAS measurements using the partial fluorescence yield (PFY) mode. This setup enables users to investigate the electronic structure of materials both below (occupied states, HAXPES) and above (unoccupied states, XAS) the Fermi level (E_F) at the same location on the material.

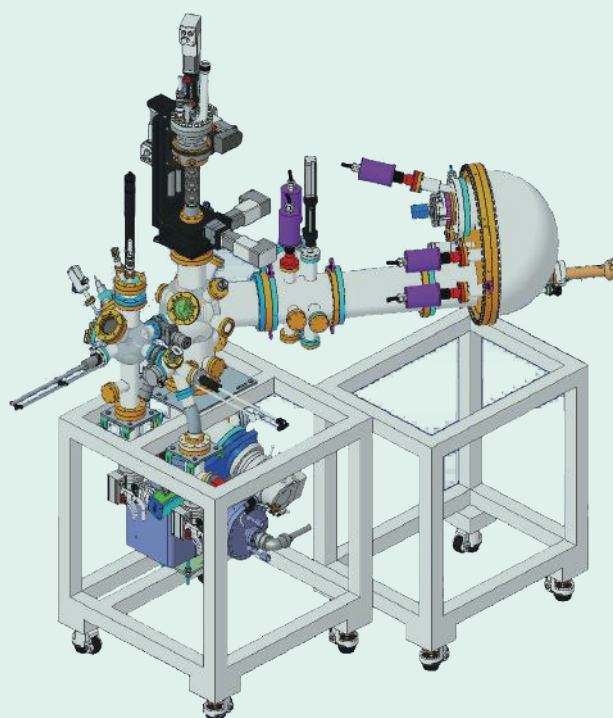


Fig. 2: Schematic drawing of the HAXPES endstation.

Tender XAS endstation is specifically designed for a tender X-ray photon energy range of 1.7–5 keV. The analysis chamber can function in either helium or vacuum mode to minimize photon absorption losses in air at low energies. A load-lock chamber minimizes the time required to stabilize the environment during sample changes. Additionally, a 13-channel silicon drift detector (13ch-SDD) is installed for PFY detection of low-concentration samples, along with simultaneous total electron yield (TEY) measurements. This endstation is designed for measuring the K-edges of silicon, phosphorus, sulfur, and chlorine, along with the L-edges of 4d and 5d transition metals.

The hard-XAS endstation, which covers an energy range of 5–11 keV, is designed for operation under ambient conditions for measuring the L-edges of lanthanides and the K-edges of 3d transition metals. It is equipped with a 19-channel silicon drift detector (19ch-SDD) for PFY mode detection of dilute samples, with sensitivity down to a few ppm. Transmission mode measurements use a grid ion chamber, which offers fast response, a broad linear range, and low background noise. The endstation also supports fly-scan mode, significantly reducing spectral measurement time. For example, Fe K-edge EXAFS scans covering a 1 keV range take approximately 20 seconds in transmission mode and 5 minutes in fluorescence mode.

The endstation is equipped with various *in situ* reaction setups, including electrocatalytic and thermocatalytic reactors and sample cooling systems. Studies of catalytic reactions focus on three main aspects: changes in the catalyst's electronic structure, the formation of intermediates, and the identification of final products. To systematically address these aspects, an advanced electrochemical flow cell integrating XAS, Raman spectroscopy, and gas chromatography–mass spectrometry (GC–MS) has been developed:

XAS: High sensitivity to changes in the electronic structure of the catalyst.

Raman spectroscopy: Effective for detecting reaction intermediates.

GC–MS: Excels in identifying and quantifying final reaction products.

Samples were dispersed on gas diffusion electrodes (GDEs) to facilitate XAS measurements. GDEs feature numerous micro-gas channels to enhance gas diffusion to the catalytic layer. Raman signals are collected through a quartz window, and the final products are analyzed using GC–MS. This setup enables comprehensive studies of catalytic reactions, providing detailed insights into electronic structures, intermediates, and final products to enhance the understanding of reaction mechanisms. This configuration not only enhances the understanding of catalytic processes but also contributes to the development of more efficient catalytic systems.

For thermal catalytic studies, the endstation supports *in situ* measurements at temperatures ranging from room temperature to 1000°C. Samples are placed in capillaries, and reaction gases are introduced through mass flow controllers to allow real-time analysis using XAS, Raman spectroscopy, and GC–MS. This integrated setup, as illustrated in Fig. 3, enables a comprehensive understanding of thermal catalytic processes. It allows researchers to investigate the dynamic changes in catalytic materials under the realistic operating conditions. These capabilities are essential for advancing research in fields such as energy conversion, environmental catalysis, and chemical production.

To enhance the efficiency of routine solid-state sample measurements and to minimize errors caused by user unfamiliarity with the operation, the TPS 32A has

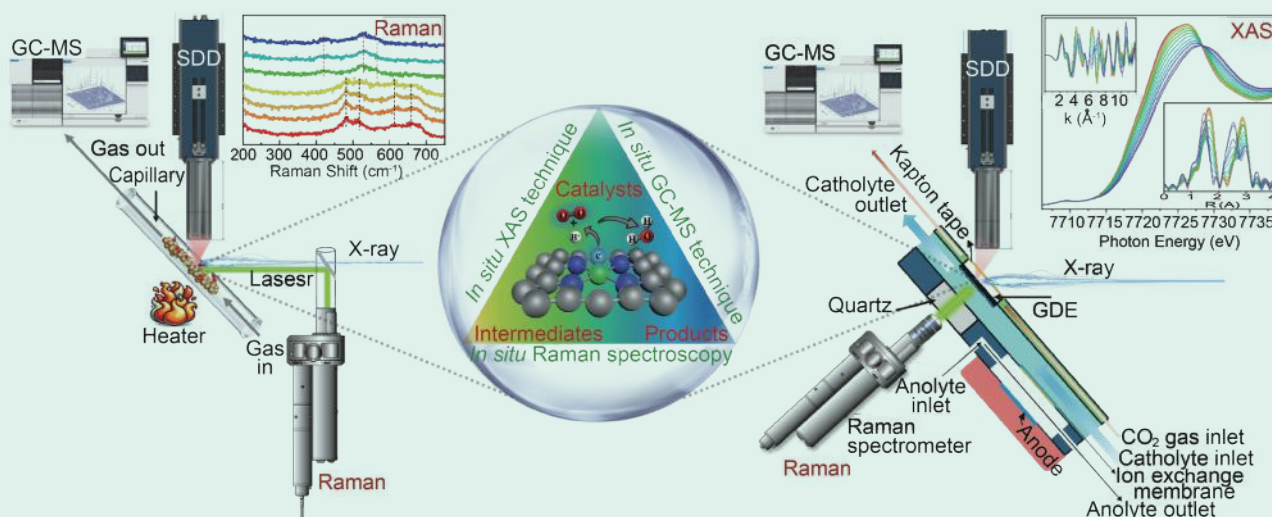


Fig. 3: Schematic drawing of the reaction cell.

developed an automated beamline alignment and sample measurement system. This system improves measurement efficiency and reduces user workload. The automated system comprises the following five subsystems, as shown in Fig. 4.

1. Automatic filter switching system: This system automatically replaces filters for different elements based on measurement requirements, effectively reducing interference from elastic scattering signals. It also helps reduce the occurrence of replacement errors and reduces the frequency of user access to the hutch, thereby improving experimental efficiency.
2. Gas switching system: This system offers two gas-switching options, namely, for high- and low-energy configurations. It automatically selects the appropriate gas settings based on experimental needs, simplifying the operation process.
3. Automatic sample changer with robotic arm: This robotic arm system can automatically load and unload samples, accommodating up to 100 samples in a single operation. Without manual intervention, it completes sample changes and automated measurements, significantly reducing the time lost due to the frequent user access to the hutch.
4. Sample-position-tracking imaging system: This system accurately tracks the position of the sample, ensuring precise alignment of the X-ray beam with the sample during every measurement. The system automatically moves the beam spot to the sample's center and scans the fluorescence signal intensity to ensure that the beam spot is positioned at the optimal signal strength location.
5. Automatic energy calibration system: This system automatically switches calibration foils for different elements, enabling automatic beamline energy calibration without manual adjustments. It simplifies the beamline alignment process and enhances operational efficiency when measuring multiple elements.

These subsystems function in coordination to provide users with an efficient and user-friendly experimental environment, allowing them to focus more on data processing and the analysis of scientific questions.

The micro-XAS endstation, the fourth on the beamline, is positioned at its end. It is equipped with K-B focusing mirrors at its entrance, which focus the X-ray beam to a spot smaller than 10 microns. The endstation is also

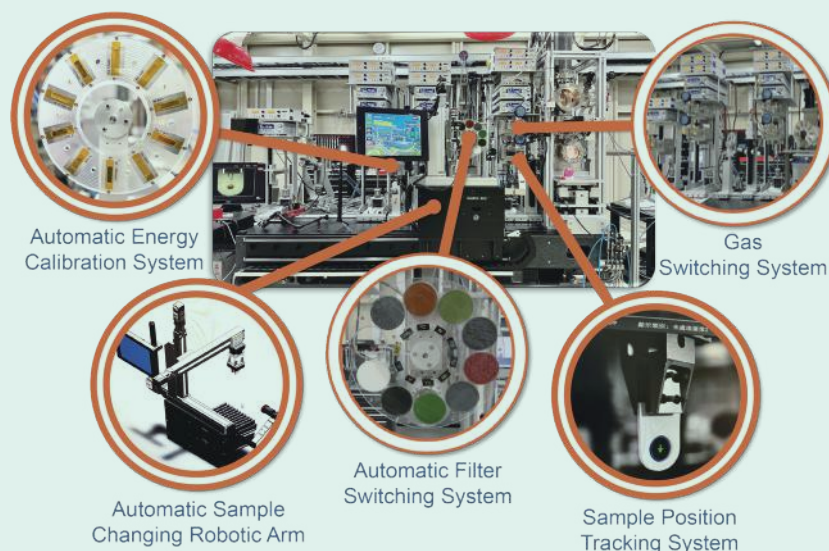


Fig. 4: TPS 32A automation architecture diagram.

equipped with a 7ch-SDD for fluorescence mode detection and includes an optical microscope for sample observation. This setup enables detailed micro-X-ray fluorescence analysis, providing spatially resolved elemental distribution information and facilitating micro-XAS measurements in regions of interest, ultimately offering micron-scale insights into unoccupied electronic states and atomic structures. Micro-XAS offers significant advantages in the fields of energy materials, environmental sciences, and archaeology since it provides high-spatial-resolution analysis of chemical and structural properties.

This report highlights the development of the **TPS 32A** tender X-ray absorption spectroscopy beamline and its endstations. Because of the successful commissioning of the beamline optics and endstations, this beamline has been effectively opened for user access. By incorporating automation and integrating advanced *in situ* analysis techniques, the beamline provides a user-friendly platform for exploring the cutting-edge scientific questions. By the end of 2024, the **TPS 32A** had invited 12 research teams and opened 30% user time for the Proposal Evaluation Committee (PEC) in 2024-2 cycle, resulting in 12 publications. In 2025, **TPS 32A** will open 50% for the PEC cycle and focus on advancing the development and commissioning of the micro-XAS and HAXPES endstations. (Reported by Ying-Rui Lu)

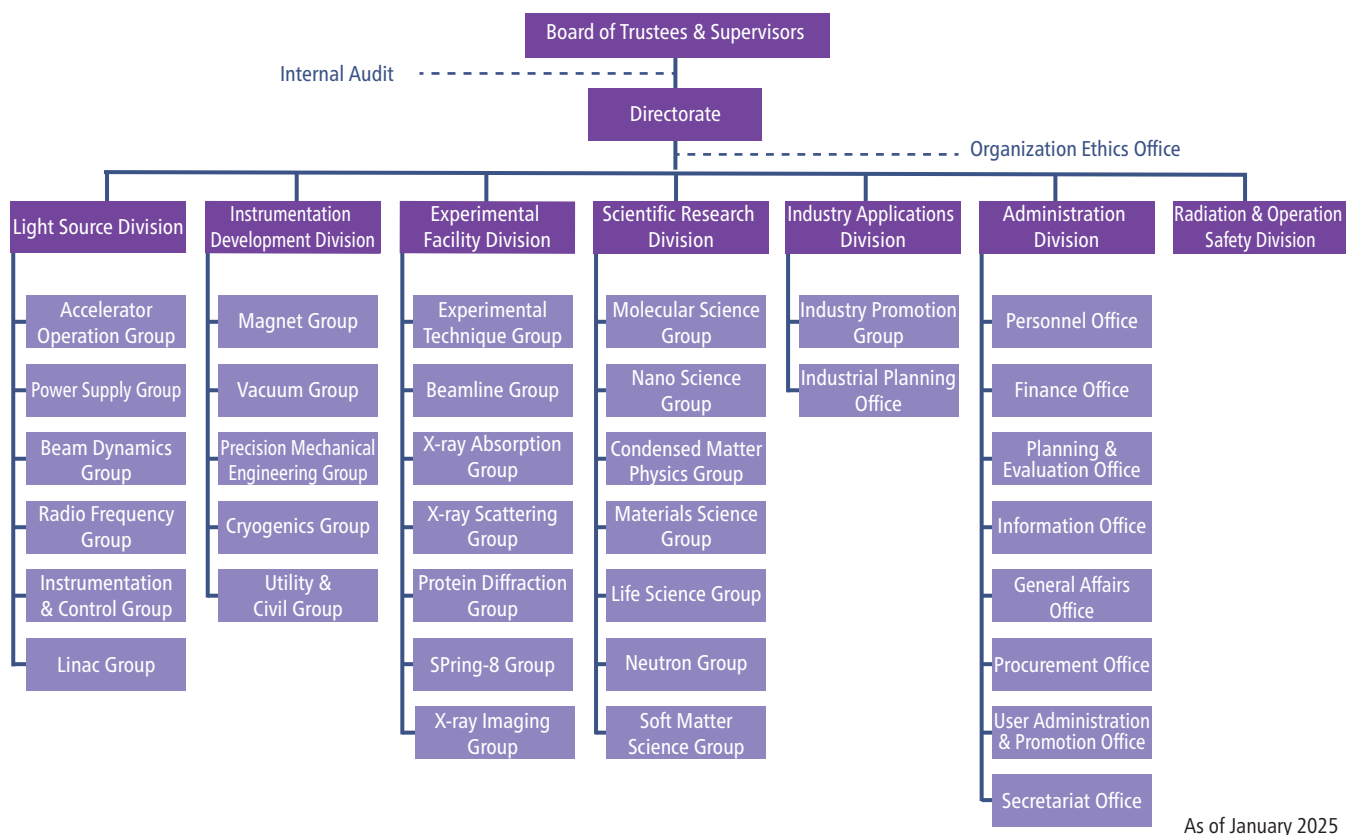
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- Dar-Bin Shieh, National Cheng Kung University
- Wen-Ching Wang, National Tsing Hua University

User Executive Committee 2024

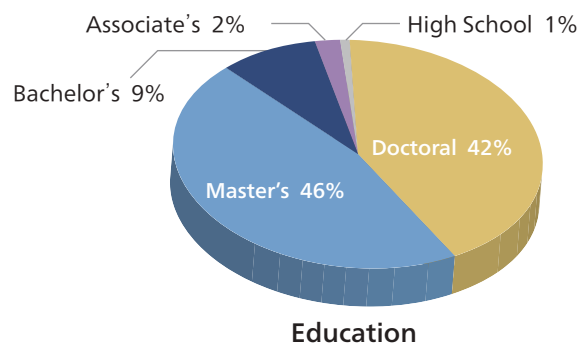
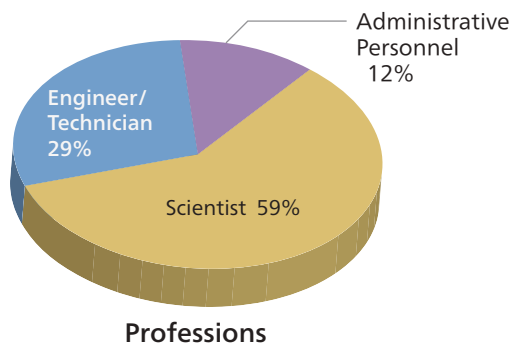
- Hao Ming Chen (Chair), National Taiwan University
- Dun-Yen Kang (Vice Chair), National Taiwan University
- Cheng-Maw Cheng, NSRRC
- Chia-Chen Chung, National Tsing Hua University
- E-Wen Huang, National Yang Ming Chiao Tung University
- Hung-Wei Shiu, NSRRC
- Shan-Li Wang, National Taiwan University
- Yane-Shih Wang, Academia Sinica
- Hsin-Jay Wu, National Yang Ming Chiao Tung University
- Yu-Hao Wu, National Yang Ming Chiao Tung University

Neutron User Executive Committee 2024

- Chao-Hung Du (Chair), Tamkang University
- Wei-Tin Chen (Vice Chair), National Taiwan University
- Yi-Fan Chen, National Central University
- Cheng-Maw Cheng, NSRRC
- Yu-Chun Chuang, NSRRC
- En-Pei Liu, National Taiwan University
- Chieh-Tsung Lo, National Cheng Kung University
- Pai-Chun Wei, National Cheng Kung University
- Chun-Chuen Yang, National Central University

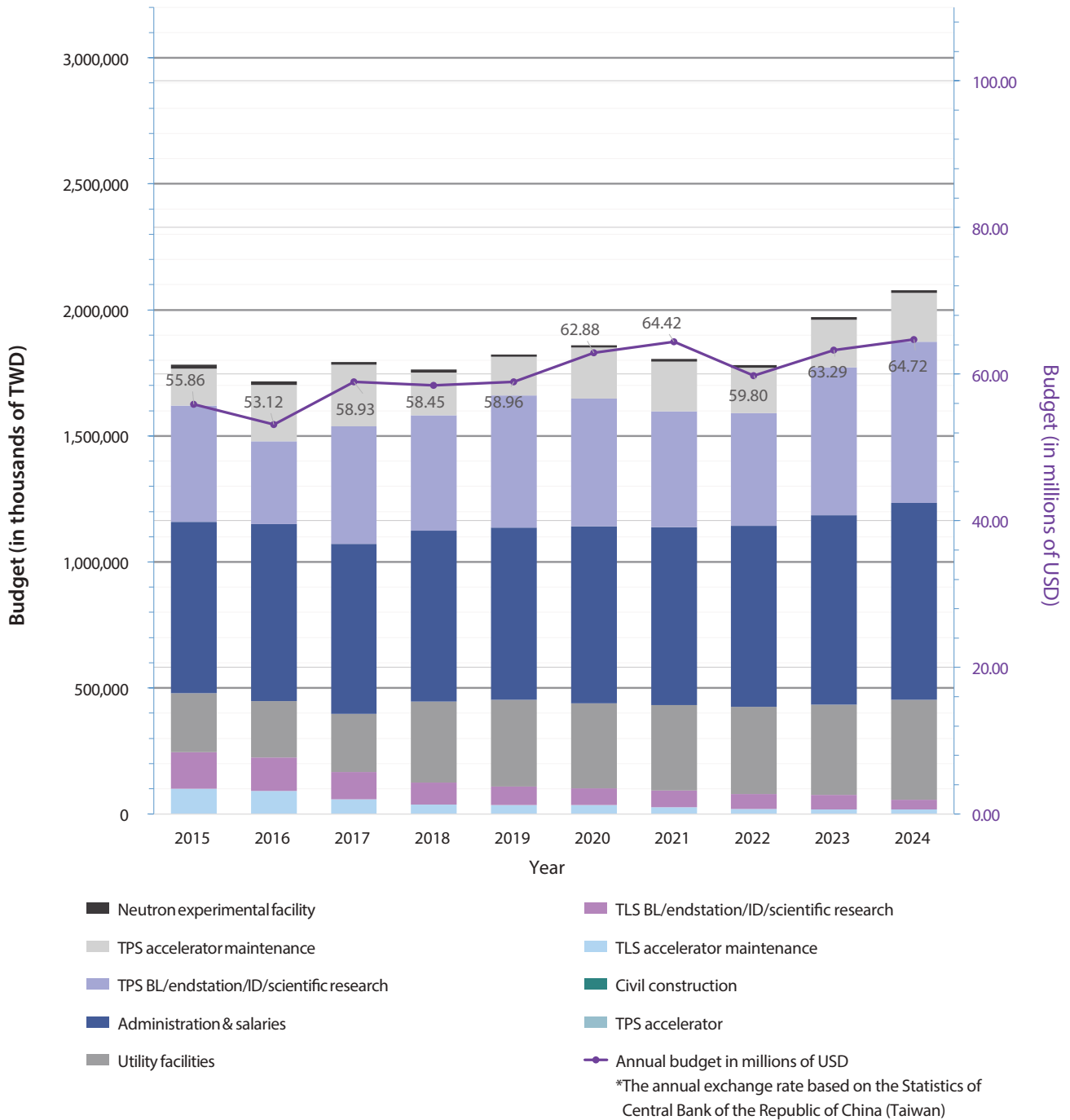
Manpower

As of January 2025, the NSRRC workforce comprises 418 staff members. The following pie charts show the manpower distributions by profession and by educational background.



Budget

The total budget for fiscal year 2024 was USD 64.72 million (based on exchange rate: 1 USD = 32.11 TWD). In terms of the budget in TWD, its original currency, there was an increase of 5.4% over the prior year. Fiscal year 2024 budget covered the operating expenses associated with the following categories: TLS accelerator maintenance, TLS BL/endstation/ID/scientific research, utility facilities, administration & salaries, TPS BL/endstation/ID/scientific research, TPS accelerator maintenance, and neutron experimental facility.



User Statistics

From 1994 to 2024, the total number of beamlines available to general users has increased from 3 to 42, comprising 22 TLS beamlines, 2 Taiwanese beamlines at SPring-8, and 18 newly opened TPS beamlines (see Beamline Status on page 117).

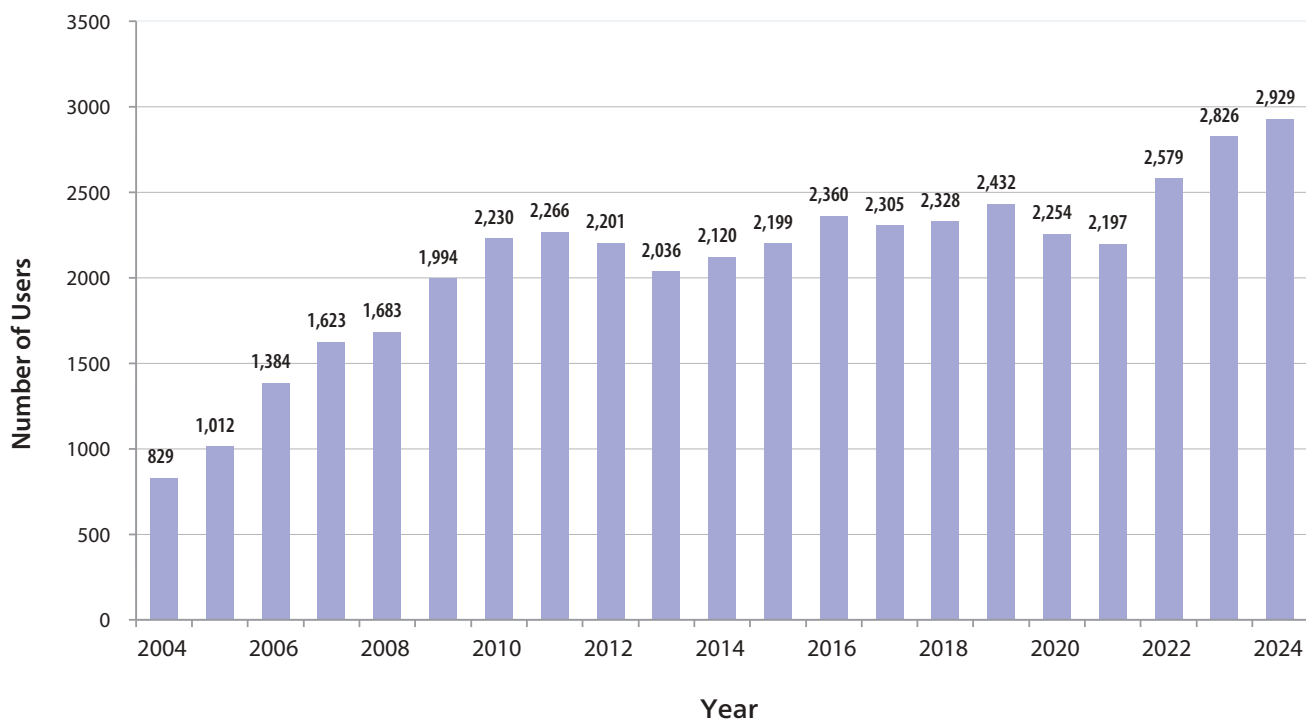
During the year 2024, the User Administration & Promotion Office (UAO) issued 1,054 new user cards and provided services to 2,929 users from 153 affiliations (including 279 users from 89 foreign institutions) to use the above-mentioned 42 NSRRC beamlines. Altogether, 687 proposals led by 413 principal investigators and involving 1,830 experimental runs and 13,861 user runs (visits) were performed.

The overall NSRRC beamtime allocated to users, excluding beamline commissioning, maintenance and training courses, was 6,133.5 shifts for TPS, 10,879 for TLS, and 867 for SPring-8. Each shift consists of eight consecutive hours.

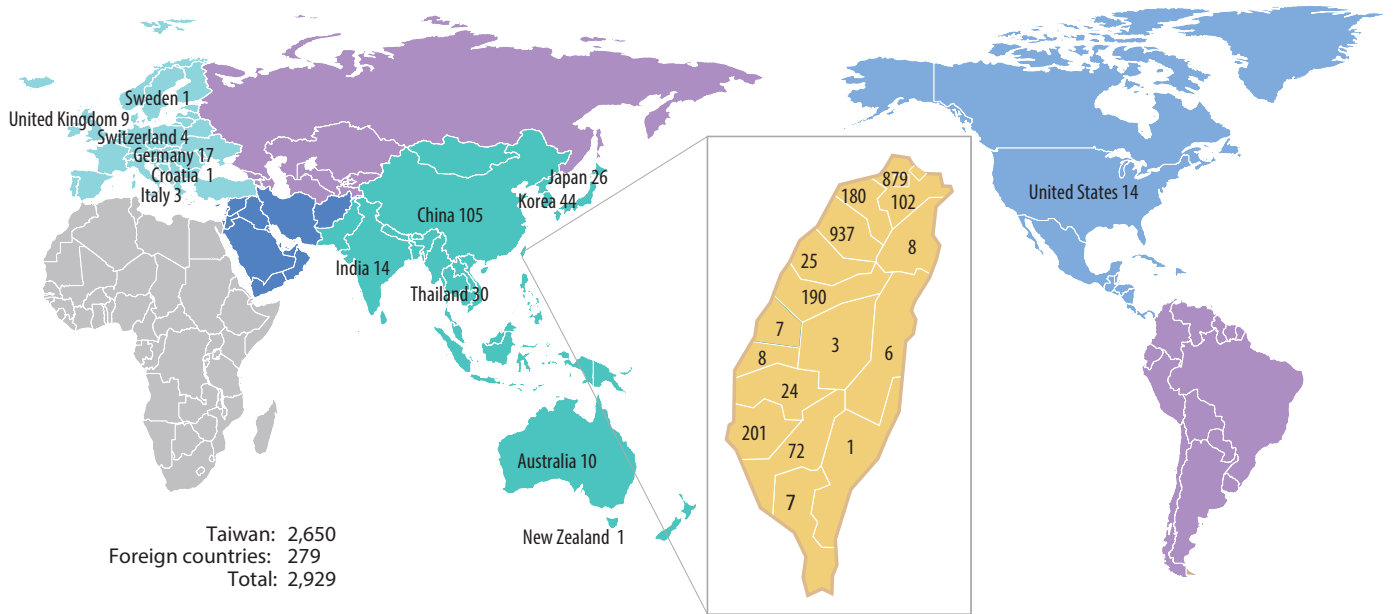
In the year 2024, the UAO processed 220 user subventions for experiments at SPring-8. Another 25 user subventions, which were funded by the National Science and Technology Council, were given to Taiwan neutron users to conduct experiments at neutron facilities worldwide.

In total, 171 master's theses and 31 doctoral dissertations utilizing NSRRC facilities were completed in 2024 (see Appendix). Operational data on beamlines/experimental endstations and users are summarized in the following figures.

Number of users 2004–2024

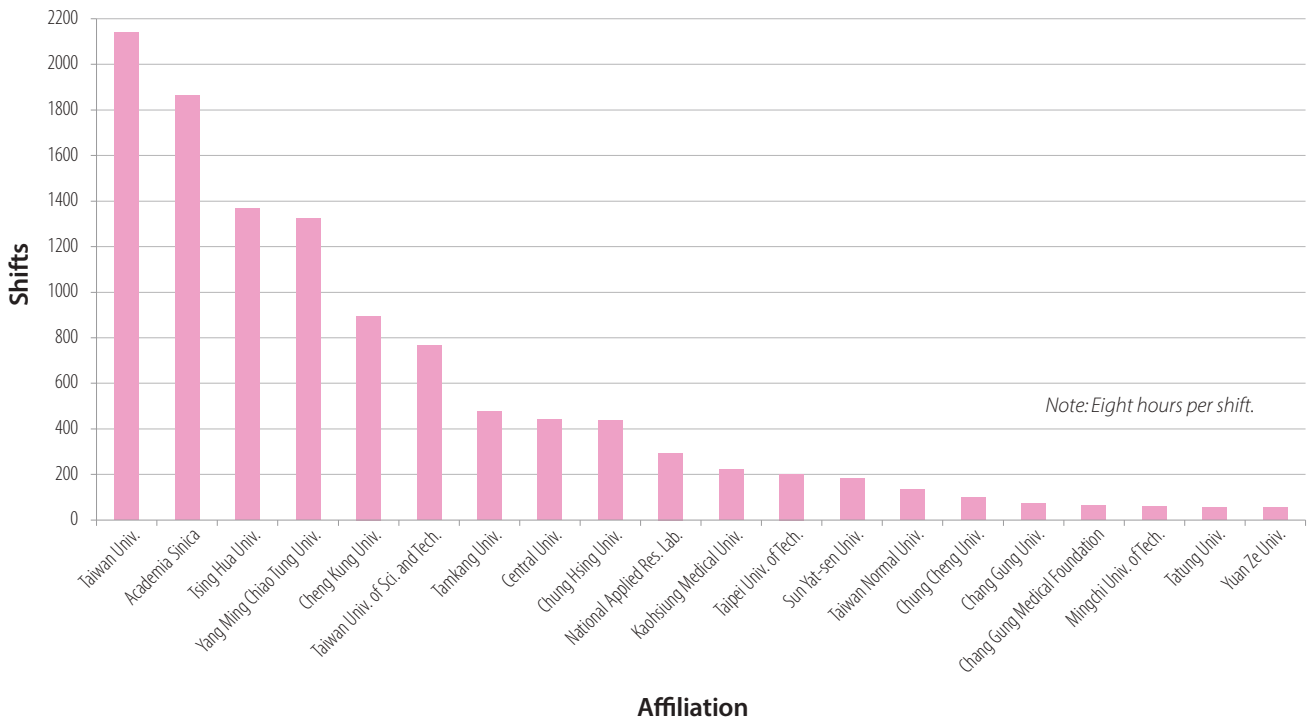


Geographic distribution of users and their numbers

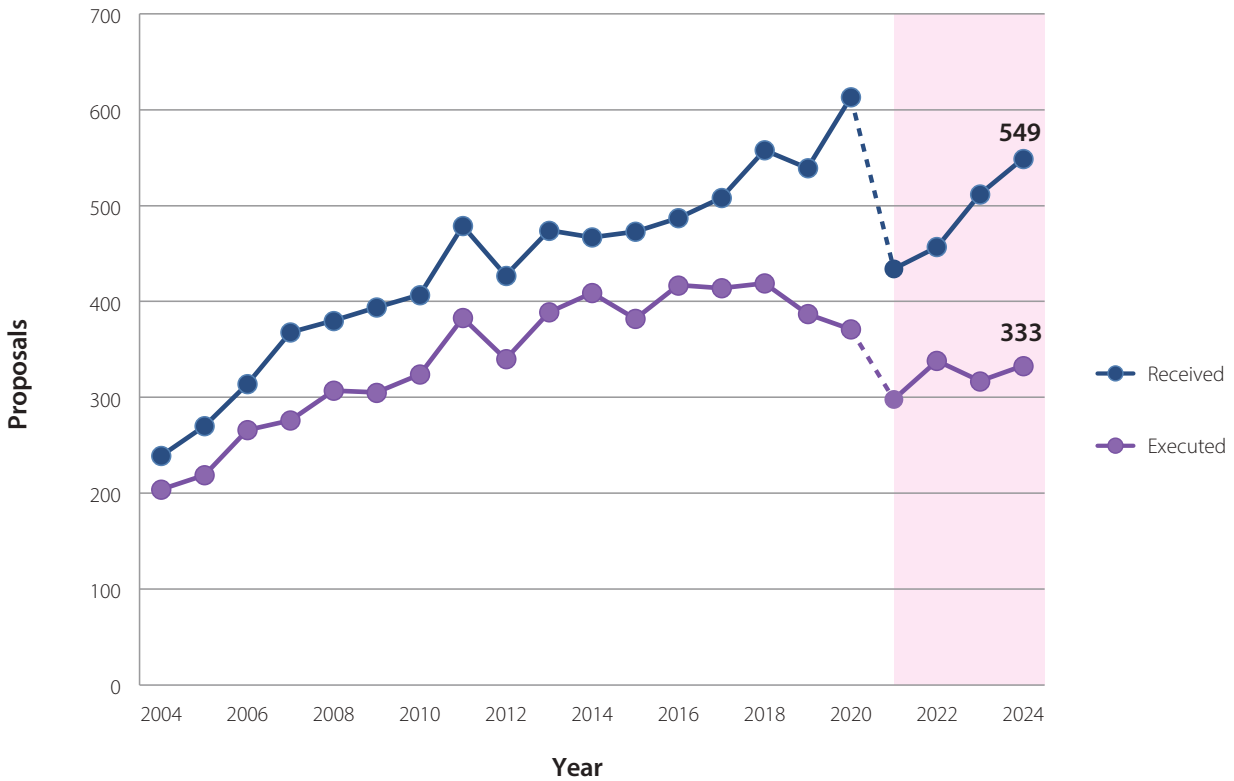


Note: The geographic categories of the users are based on the locations of their affiliations.

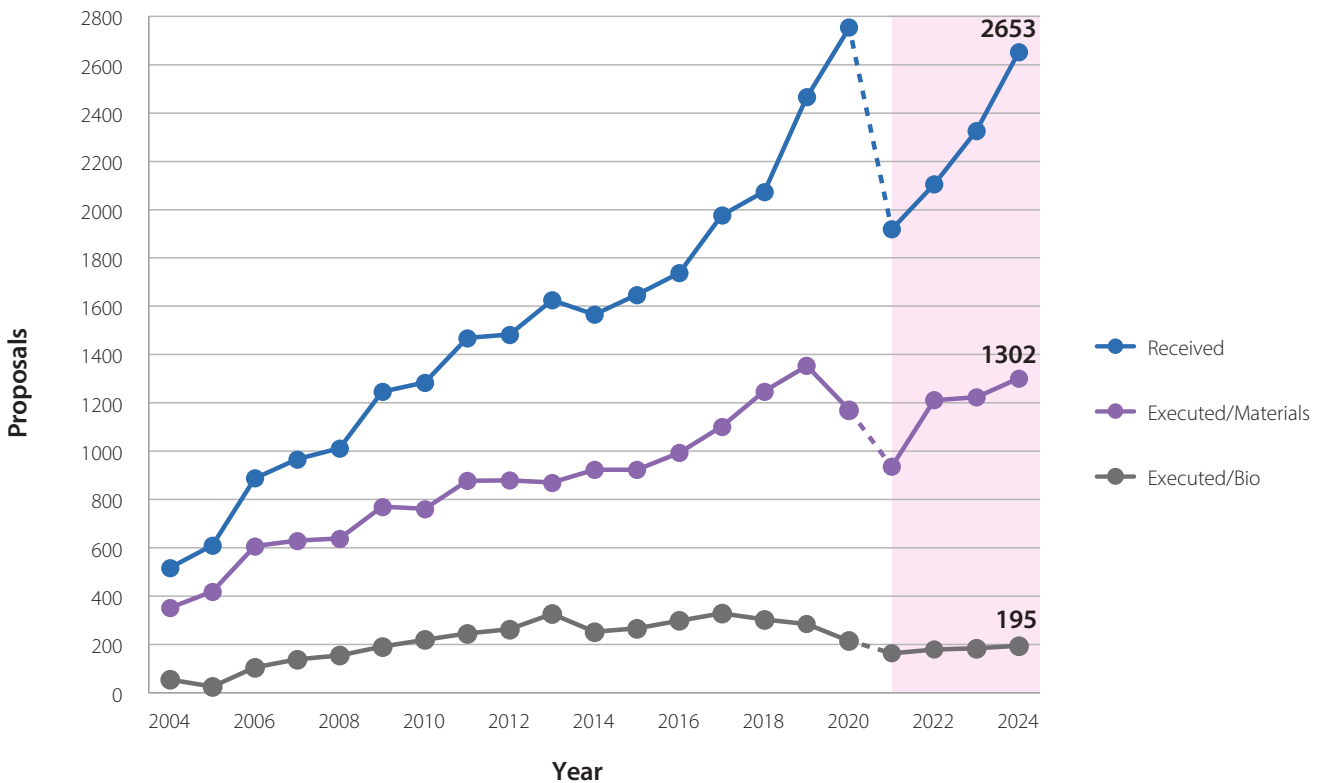
Distribution of beamtime used by domestic affiliations 2024



Number of IR/VUV proposals 2004-2024

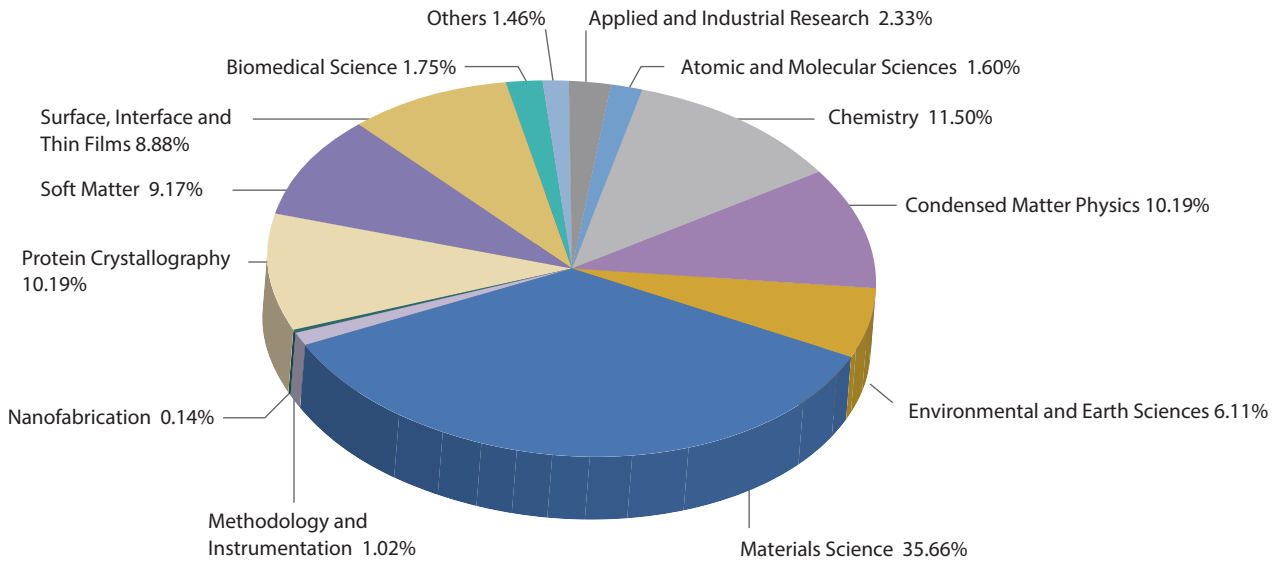


Number of X-ray proposals 2004-2024

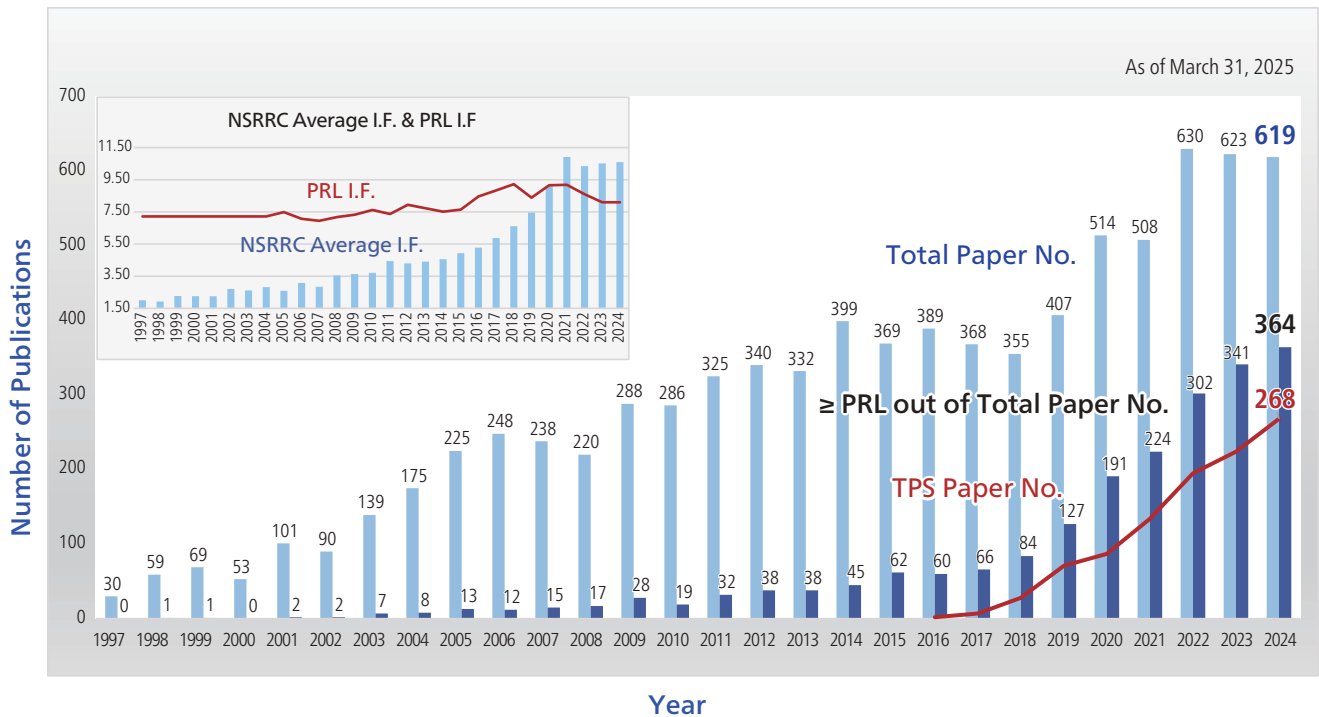


Note: From 2021 onward, NSRRC has changed from 3 calls for proposals per year to 2. This is reflected in a significant decrease in the number of proposals summed up during the year.

Proposal distribution by research fields 2024



Publications



- Notes: 1. The Impact Factor (I.F.) for the latest year's publications is based on the current year's release by Journal Citation Report (JCR). For earlier publications, the I.F. is based on the data of the corresponding JCR year.
- 2. The total number of papers comprises the SCI publications that utilized TLS, TPS, and Taiwan-contract beamlines at SPring-8.
- 3. PRL refers to Physical Review Letters, the world's premier physics journal, and is mentioned as an index in the graph accordingly.

Beamline Status

Beamlines at the Taiwan Photon Source

No.	Beamline	Mono type	Energy range (eV)	Res. power (E/ Δ E)	Endstation / Status	Spokesperson E-mail	Tel. ext.
02A	Brain Imaging	Channel-cut DCM	4k–25k	7,000	in operation	Hwu, Yeu-Kuang phhwu@sinica.edu.tw	3268
05A	Protein Microcrystallography	DCM	5.7k–20k	4,500	in operation	Chao, Frodo frodo@nsrrc.org.tw	2051
07A	Micro-focus Protein Crystallography	DCM	5.7k–20k	4,500	in operation	Chao, Frodo frodo@nsrrc.org.tw	2071
09A	Temporally Coherent X-ray Diffraction	DCM	5.6k–25k	7,000	09A1 in operation	Weng, Shih-Chang weng.sc@nsrrc.org.tw	2091
		HRM	~14.4k	~10 ⁸			
		DCM	8k–10k	7,000	09A2 in operation	Liao, Yen-Fa liao.yenfa@nsrrc.org.tw	
13A	Biological Small-angle X-ray Scattering	DCM	4k–23k	7,000	in operation	Jeng, U-Ser usjeng@nsrrc.org.tw	2131
		DMM	7k–15k	100			
		4BCC	4k–15k	10,000			
15A	Micro-crystal X-ray Diffraction	DCM	9k–35k	7,000	in operation	Wu, Lai-Chin wu.lc@nsrrc.org.tw	2151
		DMM (3%)	9k–25k	333			
		DMM (5%)	9k–25k	200			
19A	High-resolution Powder X-ray Diffraction	DCM	10k–40k	7,000	in operation	Chuang, Yu-Chun chuang.yc@nsrrc.org.tw	2191
21A	X-ray Nanodiffraction	White Beam	5k–30k	7,000	in operation	Chiang, Ching-Yu chiang.cy@nsrrc.org.tw	2211
		4BCM	7k–25k				
23A	X-ray Nanoprobe	HDCM	4k–15k	7,000	in operation	Lin, Bi-Hsuan bihsuan@nsrrc.org.tw	2231
24A	Soft X-ray Tomography	PGM	260–2.6k	2,000	in operation	Lin, Zi-Jing lin.zj@nsrrc.org.tw	2242
25A	Coherent X-ray Scattering	DCM	5.56k–20k	7,000	25A1 in operation	Lin, Jhih-Min lin.jm@nsrrc.org.tw	2251
					25A2 in operation	Huang, Yu-Shan jade@nsrrc.org.tw	
27A	Soft X-ray Nanoscopy	AM-PGM	90–3k	5,000–25,000	27A1 in operation	Hsu, Yao-Jane yjhsu@nsrrc.org.tw	2271
					27A2 commissioning	Wei, Der-Hsin dhw@nsrrc.org.tw	2272
31A	Projection X-ray Microscopy	DCM	5k–30k	7,000	in operation	Yin, Gung-Chian gcyin@nsrrc.org.tw	2312
		DMM	5k–30k	200			
32A	Tender X-ray Absorption Spectroscopy	DCM	1.7k–11k	7,000	in operation	Chen, Chi-Liang chen.cl@nsrrc.org.tw	2321

No.	Beamline	Mono type	Energy range (eV)	Res. power (E/ Δ E)	Endstation / Status	Spokesperson E-mail	Tel. ext.
39A	Nanometer Angle-resolved Photoemission Spectroscopy	AM-PGM	20–650	100,000	39A1 in operation 39A2 construction	Cheng, Cheng-Maw makalu@nsrrc.org.tw	2391
41A	Soft X-ray Scattering	AGM	0.4k–1.2k	26,500	in operation	Huang, Di-Jing djhuang@nsrrc.org.tw	2411
44A	Quick-scanning X-ray Absorption Spectroscopy	Quick-mono	4.5k–34k	7,000	in operation	Pao, Chih-Wen pao.cw@nsrrc.org.tw	2441
45A	Submicron Soft X-ray Spectroscopy	AGM	0.28k–1.5k	21,500 @510 eV 20,000 @860 eV	45A	(BL) Tsai, Huang-Ming tsai.hm@nsrrc.org.tw	7356
					45A1 in operation	(ES) Chang, Chun-Fu chun-fu.chang@cpfs.mpg.de	2451
					45A2 in operation	(ES) Chiou, Jau-Wern jwchiou@nuk.edu.tw	2452

Beamlines at the Taiwan Light Source

No.	Beamline	Mono type	Energy range (eV)	Res. power (E/ Δ E)	Status	Spokesperson E-mail	Tel. ext.
01A1	SWLS – White X-ray (PRT 75%)	none	> 5k	N/A	in operation	Hwu, Yeu-Kuang phhwu@sinica.edu.tw	1011
01B1	SWLS – X-ray Microscopy	DCM	8k	1,000	in operation	Wang, Chun-Chieh wang.jay@nsrrc.org.tw	1012
01C1	SWLS – EXAFS	DCM	6k–33k	7,000	in operation	Chan, Ting-Shan chan.ts@nsrrc.org.tw	1013
01C2	SWLS – X-ray Powder Diffraction				in operation	Chuang, Yu-Chun chuang.yc@nsrrc.org.tw	1013
03A1	BM – (HF-CGM) – Photoabsorption/ Photoluminescence	CGM	4–40	50,000	in operation	Wu, Yu-Jong yjwu@nsrrc.org.tw	1031
05B1	EPU – Soft X-ray Chemistry	SGM	60–1.5k	20,000	in operation	Liu, Chen-Lin Liu.CL@nsrrc.org.tw	1050
07A1	Industrial Application	DCM	5k–23k	7,000	in operation	Tang, Mau-Tsu mautsu@nsrrc.org.tw	1071
08A1	BM – (L-SGM) XPS, UPS	SGM	15–200	20,000	in operation	Lin, Ping-Hui lin.pinghui@nsrrc.org.tw	1081
08B1	BM – AGM	AGM	0.3k–1k	10,000	in operation	Chainani, Ashish Atma chainani.ash@nsrrc.org.tw	1082
09A1	U50 – SPEM	SGM	60–1.5k	15,000	in operation	Chen, Chia-Hao chchen@nsrrc.org.tw	1101
09A2	U50 – Spectroscopy				in operation	Hsu, Yao-Jane yjhsu@nsrrc.org.tw	1102
11A1	BM – (Dragon) MCD, XAS (PRT 75%)	SGM	8–1.5k	15,000	in operation	Chainani, Ashish Atma chainani.ash@nsrrc.org.tw	1111
13A1	SW60 – X-ray Scattering	ACCM	12k–14k	1,000	in operation	Lee, Ming-Tao mtlee@nsrrc.org.tw	1131

No.	Beamline	Mono type	Energy range (eV)	Res. power (E/ΔE)	Status	Spokesperson E-mail	Tel. ext.
13B2	Beamline of Interdisciplinary Energy Researches	DCM	5k–20k	7,000	in operation	Chuang, Wei-Tsung weitsung@nsrrc.org.tw Chan, Ting-Shan chan.ts@nsrrc.org.tw	1132
14A1	BM – IR Microscopy	FTIR	0.05–0.5 (400–4,000 cm ⁻¹)	3,200–32,000	in operation	Lee, Yao-Chang ychee@nsrrc.org.tw	1141
15A1	IASW – Biopharmaceuticals Protein Crystallography	DCM	5k–20k	7,000	in operation	Chao, Frodo frodo@nsrrc.org.tw	1151
16A1	BM – Tender X-ray Absorption, Diffraction	DCM	2k–8k	7,000	in operation	Chan, Ting-Shan chan.ts@nsrrc.org.tw	1161
17B1	W200 – X-ray Scattering	DCM	4k–15k	7,000	in operation	Lin, Yan-Gu lin.yg@nsrrc.org.tw	1172
17C1	W200 – EXAFS	DCM	4k–15k	7,000	in operation	Chan, Ting-Shan chan.ts@nsrrc.org.tw	1173
20A1	BM – (H-SGM) XAS	SGM	70–1.2k	10,000	in operation	Haw, Shu-Chih ho.kelman@nsrrc.org.tw	1201
21A1	U90 – (White Light) Chemical Dynamics (PRT 75%)	none	4–500	50	in operation	Lee, Shih-Huang shlee@nsrrc.org.tw	1211
21A2	U90 – (White Light) Photochemistry				in operation	Wu, Yu-Jong yjwu@nsrrc.org.tw	1210
21B1	U90 – (CGM) Angle-resolved UPS	CGM	4–100	10 ⁵	in operation	Cheng, Cheng-Maw makalu@nsrrc.org.tw	1212
21B2	U90 – Gas Phase				in operation	Cheng, Cheng-Maw makalu@nsrrc.org.tw	1212
23A1	IASW – Small/Wide Angle X-ray Scattering	DCM DMM	5k–23k 6–15k	7,000 100	in operation	Jeng, U-Ser usjeng@nsrrc.org.tw	1231
24A1	BM – (WR-SGM) XPS, UPS, XAS, APXPS	SGM	15–1.5k	15,000	in operation	Wang, Chia-Hsin wang.ch@nsrrc.org.tw	1241

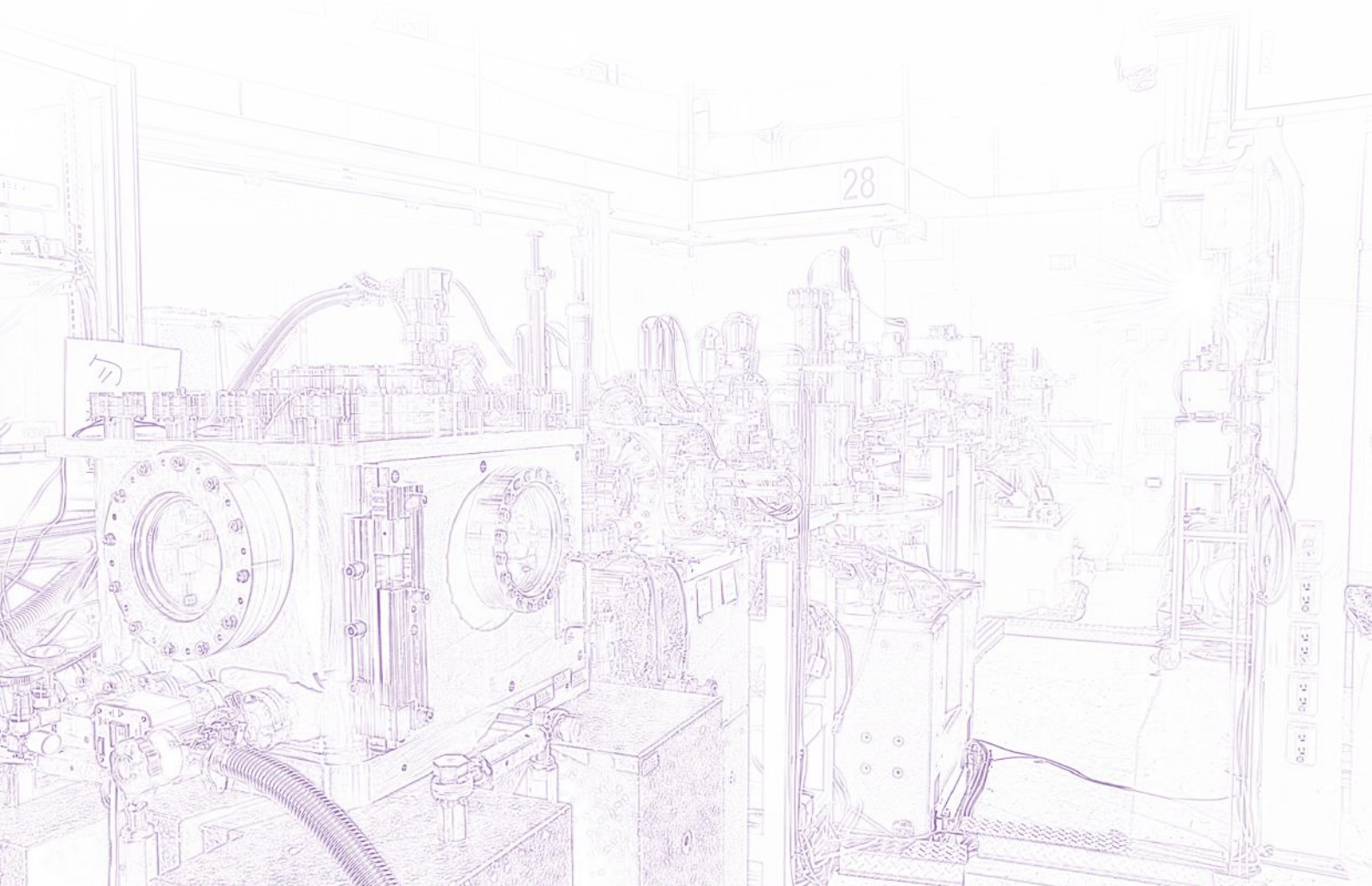
Beamlines at SPring-8, Japan

No.	Beamline	Mono type	Energy range (eV)	Res. power (E/ΔE)	Status	Spokesperson E-mail
SP 12B1	BM – Materials X-ray Study	DCM	5k–100k	7,000	in operation	Ishii, Hirofumi h_ishii@spring8.or.jp
SP 12U1	U32 – Inelastic X-ray Scattering HAXPES/Photoemission	DCM HRM	5k–30k 6k–14.4k	10 ⁴ –10 ⁶ 300,000	in operation	Hiraoka, Nozomu hiraoka@spring8.or.jp

Neutron Instrument at ACNS, ANSTO, Australia

Instrument	Incident energy (meV)	Energy res. (meV)	Status	Contact
SIKA – Cold Neutron Triple-axis Spectrometer	2.6–25	0.026–2	in operation	Yano, Shin-ichiro yano.shin@nsrrc.org.tw

4BCC: 4-Bounce Crystal Collimator
4BCM: 4-Bounce Channel-cut Monochromator
ACCM: Asymmetrically-cut Curved Crystal Monochromator
AGM: Active Grating Monochromator
APXPS: Ambient-Pressure X-ray Photoelectron Spectroscopy
BM: Bending Magnet
CGM: Cylindrical Grating Monochromator
DCM: Double Crystal Monochromator
DM: Diamond Crystal Monochromator
DMM: Double Multilayer Monochromator
EPU: Elliptically Polarized Undulator
EXAFS: Extended X-ray Absorption Fine Structure
FTIR: Fourier Transform Infrared Spectroscopy
HAXPES: Hard X-ray Photoelectron Spectroscopy
H-SGM: High Energy Spherical Grating Monochromator
HDCM: Horizontal Double Crystal Monochromator
HF-CGM: High Flux Cylindrical Grating Monochromator
HRM: High Resolution Crystal Monochromator
IASW: In-Achromatic Superconducting Wiggler
IR: Infrared Radiation
L-SGM: Low Energy Spherical Grating Monochromator
MCD: Magnetic Circular Dichroism
NIM: Normal Incidence Monochromator
PEEM: Photo-Emission Electron Microscope
PGM: Plane Grating Monochromator
PRT: Participating Research Team
QCM: Quadruple Crystal Monochromator
SGM: Spherical Grating Monochromator
SPEM: Scanning Photoemission Electron Microscope
SW60: Superconducting Wiggler-60 mm
SWLS: Superconducting Wavelength Shifter
U32: Undulator-32 mm
U50: Undulator-50 mm
U90: Undulator-90 mm
UPS: Ultraviolet Photoelectron Spectroscopy
W200: Wiggler-200 mm
WR-SGM: Wide-Range Spherical Grating Monochromator
XAS: X-ray Absorption Spectroscopy
XPS: X-ray Photoelectron Spectroscopy
XRD: X-ray Diffraction



Major Events

NSRRC Board Meetings

The NSRRC Board of Trustees (BOT; see page 110 for details) was established in 2003, and each BOT serves a three-year term. The BOT is responsible for ensuring that the NSRRC serves its purpose as an integral member of the scientific community.

The seventh NSRRC BOT, chaired by Prof. Minn-Tsong Lin, held its tenth and last meeting on February 21. In this meeting, the NSRRC management gave status reports on NSRRC operations and recent achievements. The BOT reviewed the 2023 Final Accounts, work performance, and future plan of the TLS.

The eighth NSRRC BOT was approved by the Executive Yuan on January 30, with the term starting on February 29 and Prof. Minn-Tsong Lin serving as Acting Chair. The first Supervisory Board Meeting was held on March 22, and the first BOT Meeting and the second Supervisory Board Meeting was held on April 25. In the meeting, the goal of the operation termination plan of the TLS at the end of 2028 was decided; however, the possibility of its low-load operation was retained.

The National Science and Technology Council (NSTC) appointed Vice Minister Tzong-Chyuan Chen as Acting Chair of the eighth NSRRC BOT, effective from May 20 onward. On August 27, the Executive Yuan approved Vice Minister Chen-Kang Su of the NSTC as the chair of the eighth NSRRC BOT.

The second BOT Meeting and the third Supervisory Board Meeting was held on July 30. NSRRC management briefed participants on the current operation status, and the BOT members discussed the 2025 Budget Estimate as well as revisions to the internal regulations. On September 27, the first Executive BOT Meeting was held, and the revisions to the internal personnel regulations were discussed.

The third meeting of the year took place on December 10, together with the fourth Supervisory Board Meeting. In the meeting, the new internal audit team member list was approved; the sixth Science Advisory Committee was established, of which the tenure runs from January 1, 2025 to December 31, 2028.

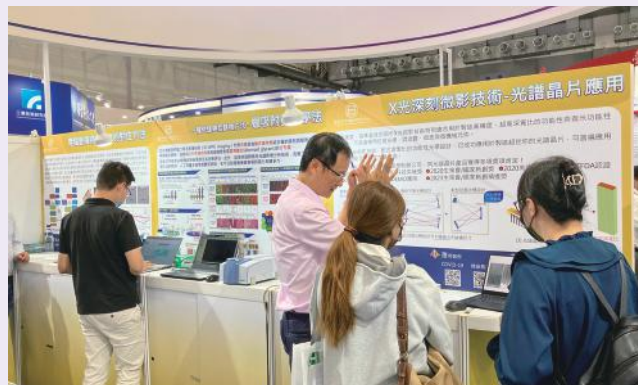


The first BOT Meeting and the second Supervisory Board Meeting was held on April 25. In the photo, from left to right in the front row were BOT members: Ray Hua Horng, Yuan-Tseh Lee, Minn-Tsong Lin (Acting Chair), Mei-Yin Chou, and Bon-Chu Chung. From left to right in the back row were: Jia Shin Lin (Executive Supervisor), Yu-Ju Chen, Huey-Kang Sytwu, Meng-Fan Luo, Mann-Ching Sherry Ku, Yao-Jane Hsu (NSRRC Secretary General), and Chia-Hung Hsu (NSRRC Director).

NSRRC Presented Biomedical Technologies at BIO Asia-Taiwan

NSRRC participated in the BIO Asia-Taiwan International Conference & Exhibition (BIO Asia-Taiwan) at the Taipei Nangang Exhibition Center from July 24 to 27, as part of the Biotechnology-Application Industry-Academia Alliance organized by the College of Life Science at National Tsing Hua University. At the exhibition, NSRRC highlighted the application of synchrotron radiation-based experimental technologies in the biomedical field with main focuses. The first included advanced analysis services, such as core facility support for protein crystallography, structural and compositional analysis of liposomes and exosomes, and the development of drug delivery systems. The second emphasized the practical application of NSRRC's research, featuring innovations like ultra-miniature spectroscopic chips, infrared wax adsorption kinetics for rapid cancer screening, and computed tomography for shadow line alignment. NSRRC staff engaged with numerous enterprises

at the exhibition and received positive feedback on the innovations and contributions of the facility.



NSRRC highlighted the application of synchrotron radiation-based experimental technologies in the biomedical field at the BIO Asia-Taiwan.

The Annual Open House

The annual NSRRC Open House took place on October 6, attracting 350 adults and children for a fun-filled day of science activities. The event featured an exciting chemical magic performance by the Center for Science Education from Tamkang University, along with interesting science stations at the Taiwan Photon Source (TPS) and engaging science game booths.

Ten science stations were set up along the TPS ring, where “Light Scientists” guided participants through the wonders of how TPS light is used to explore everything in the world, from nanostructures to steel buildings. The demonstrations and hands-on activities offered a glimpse into the critical role of “light” in our everyday lives. Additionally, game booths from the Li Hongming Foundation, NTHU's THz Optics & Photonics Center, and the National Taiwan Science Education Center contributed to the lively atmosphere throughout the event.

The NSRRC remains dedicated to advancing scientific research while bringing science closer to the public. Through entertainment and educational activities, the NSRRC continues to foster a broader understanding and spark public interest.



The NSRRC Open House attracted 350 adults and children to visit the science stations at the TPS.

NSRRC Presented Innovative Technologies at FUTEX 2024

The “Future Tech Pavilion (FUTEX)” at the Taiwan Innotech Expo 2024, held from October 17 to 19 at the Taipei World Trade Center Exhibition Hall 1, was co-organized by NSTC, the Ministry of Education, the Ministry of Health and Welfare, and Academia Sinica. FUTEX spotlighted technological trends expected to shape the next three to ten years, featuring six key exhibition areas: “AIoT & Smart Applications,” “Electronics & Optoelectronics,” “Evolutionary Materials & Chemicals,” “Biotech, New Drugs & Medical Devices,” “Net Zero,” and “Humanity & Technology.” In addition, the highly anticipated “AI Theme Area” and “Healthy Taiwan Theme Area” delivered specialized knowledge in an engaging and accessible format.

This year, NSRRC spotlighted a range of cutting-edge technologies at FUTEX, with a focus on innovative technologies and practical applications. The ten featured technologies included:

1. Solid-State RF Power Amplifiers (presented by Fu-Tsai Chung)
2. The Precise Archer in the Accelerator (presented by Chyi-Shyan Fann)
3. From Lab to Application: Magnetron Sputtering Preparation of Non-Evaporable Getter (NEG) Films (presented by Chin Shueh)
4. The Large Helium Gas Cryogenic Purification System (presented by Huang-Hsiu Tsai)
5. The Liquid Helium Phase Separator with Condenser (presented by Huang-Hsiu Tsai)
6. The Multi-Channel Cryogen Fluid Transfer Line (presented by Huang-Hsiu Tsai)
7. Fast Cancer Screening (presented by Yao-Chang Lee)
8. High-Efficiency Micron-Scale X-ray Computed Tomography (presented by Gung-Chian Yin)
9. Refining at 1600°C: Advanced Synchrotron X-ray Insights into Blast Furnace Ironmaking and the Path to Net-Zero Carbon Emissions (presented by Shi-Wei Chen)
10. Tango Partner: Green Ammonia Plus, Carbon Emissions Minus (presented by Yan-Gu Lin)

Through exhibitions, NSRRC demonstrated its leadership in scientific research and industrial innovation, underscoring its role in driving technological breakthroughs and future advancements.



NSRRC scientists introduced synchrotron radiation technologies to the visitors at FUTEX 2024.

Prof. Ru-Shi Liu of National Taiwan University Received the 2024 NSRRC Outstanding Paper Award

This year, Prof. Ru-Shi Liu from the Department of Chemistry at National Taiwan University was honored with the fourth “Outstanding Paper Award” established by the NSRRC. This esteemed accolade, highly regarded within Taiwan’s research community, includes a trophy and a prize of TWD\$300,000. Prof. Liu’s research team was recognized for their groundbreaking work on “luminescent energy transfer pathways of near-infrared light-emitting diodes” using synchrotron-based light sources, impressing the award’s review committee. After a rigorous evaluation process, Prof. Liu’s publication was chosen as the standout entry.

Dr. Der-Hsin Wei, Deputy Director of the NSRRC, chaired the review meetings for the award, inviting a panel of distinguished experts and researchers to assess impactful publications from the past eight years that employed NSRRC’s facilities. Prof. Liu’s research revealed significant applications in various fields, including optoelectronic technology, security and anti-counterfeiting, agricultural technology, and biomedical imaging. Since its publication in 2018, the team’s work has been cited more than 300 times, establishing it as an influential paper in its field. The innovative technology developed has been successfully transferred to Everlight Electronics Co., Ltd., a prominent light-emitting diode (LED) manufacturer in Taiwan, making this a well-deserved recognition for Prof. Liu.

With long-term support from the NSTC, Prof. Liu has dedicated his research to the formulation, synthesis, analysis, and applications of core technologies in materials chemistry, including “photon-to-photon conversion,” “photon-to-electron conversion,” and “photon-to-heat conversion.” His inventions have found applications across various industries of optoelectronics, green energy, biomedicine, and nano-materials, leading to several patents and technology transfer. Notably, Prof. Liu was the pioneer in developing Taiwan’s first red LED, laying the groundwork for the country’s LED industry.

Throughout his career, Prof. Liu has garnered numerous awards for his exceptional contributions to academic and industrial advancements. His honors include the Excellent Young Person Prize (1989), National Invention Award – Silver Medal from Intellectual Property Office (1995), Young Chemists Award (1998), Z. Hsu Scientific Paper Award (2011) and Z. Hsu Chair Professor Award (2019) from the Far Eastern Y. Z. Hsu Science and Technology Memorial Foundation, Outstanding Research Award from National Science Council (2012, 2017), Hou Chin-Tui Award (2018), TECO Award in Chemical Engineering/Materials (2019), Academic Award of the Ministry of Education (2020), FutureTech Awards (2021, 2024), Outstanding Paper Award from Chemical Society (2021), and recognition as a Highly Cited Researcher by Clarivate



The 2024 NSRRC Outstanding Paper Award was presented to Prof. Ru-Shi Liu.

Analytics (2018 to 2021, 2023). The NSRRC’s “Outstanding Paper Award” is a testament to Liu’s significant impact on both academia and industry.

The award ceremony, originally scheduled to take place during the 30th Users’ Meeting & Workshops, was canceled due to government announcements regarding the suspension of work and classes in Hsinchu City on the event date. Nevertheless, the award winner of the year was still announced, and the trophy will be presented at the next Users’ Meeting.

Background of the Outstanding Paper Award

Academician Chien-Te Chen was honored with the prestigious Presidential Science Prize in 2017. To encourage more scientists to engage in technological developments in synchrotron-based accelerators, as well as scientific research, Chen donated both the trophy and the monetary award to the NSRRC. His goal is to support Taiwan’s synchrotron radiation facilities and inspire scientists to excel in research and make magnificent contributions to the field of science. To uphold Chen’s vision, NSRRC used the donated monetary award to install rooftop solar panels. The gross profit generated by solar electricity is used to fund the Outstanding Paper Award.

The objective of the NSRRC Outstanding Paper Award is to advance scientific research in synchrotron-based light sources in Taiwan and encourage researchers in the academic community to develop the technology in accelerators, beamline engineering fields, as well as to publish papers in high-impact international journals by utilizing NSRRC light source facilities. The award is given for papers published in three specific fields: applied science, life science, and natural science. Applications and reviews for each of the three fields are processed every three years in sequential, rotating order.

NSRRC's Industry Applications Division Received the Technology Management Award from CSMOT

The Industry Applications Division of the NSRRC was honored with the 2024 Technology Management Award (Academic and Research Team Category) by the Chinese Society for Management of Technology (CSMOT) on December 6 at Yuan Ze University. This recognition is for the division's exceptional planning, promotion, and execution of the "Industrial Application Service Platforms for the Synchrotron Radiation Facility."

A key mission of the NSRRC is conducting cutting-edge basic and applied research in synchrotron radiation. The Industrial Application Group has been part of its structure since its early founding, and in 2021, the Industry Applications Division was established to meet the growing industrial demand for synchrotron light sources. The division includes the Industrial Planning Office and the Industry Promotion Group, which focus on developing strategies and executing industrial application projects, respectively.

The division integrates NSRRC's research and development capabilities and is developing platforms in such sectors as semiconductors, energy, and accelerator technologies. Its goal is to connect academic research with industry applications, using large-scale accelerator light source technology to address industrial bottlenecks, develop innovative technologies, and boost Taiwan's global competitiveness.

Founded in 1990, CSMOT brings together experts in technology management to foster collaboration between industry, government, and academia. The Technology Management Award, the society's highest honor, annually recognizes individuals and teams for their outstanding contributions to the field and encourages the practical application of academic research in Taiwan's technology management.



NSRRC Industry Applications Division received the Technology Management Award from CSMOT.

Opening Ceremony of the TPS 27A1 Soft X-ray Nanoscopy Beamline & STXM Endstation



The group photo was taken after the plaque was unveiled at the TPS 27A1 Soft X-ray Nanoscopy Beamline and STXM Endstation.

The Taiwan Photon Source (TPS) 27A1 Soft X-ray Nanoscopy Beamline and Scanning Transmission X-ray Microscopy (STXM) Endstation, developed through years of dedicated effort, is now ready to provide high-quality soft X-ray sources and nanoscale chemical imaging capabilities. The opening ceremony of the TPS 27A1 Nanoscopy Beamline and STXM Endstation, organized by the Department of Physics at Tamkang University (TKU) and the NSRRC, took place on December 20 at the NSRRC.

The ceremony began with welcome remarks by Director Chia-Hung Hsu of the NSRRC. The guests of honor from TKU, led by the Chairperson of its Board of Trustees, along with the NSRRC executive team, delivered speeches and unveiled the endstation plaque. The beamline staff then demonstrated the features and core techniques of the endstation to the participants. After the ceremony, attendees proceeded to the lobby for light refreshments and continued their discussions.

This state-of-the-art STXM endstation, collaboratively designed and constructed by the Department of Physics at TKU and the NSRRC, with support from the National Science and Technology Council, highlights the successful outcomes of cross-institutional collaboration. This cutting-edge facility is expected to empower researchers both domestically and internationally in multidisciplinary fields, such as energy, semiconductors, magnetism, and environmental science, aiming to advance scientific research in Taiwan.

MOUs and Collaboration

In 2024, NSRRC focused on the further expansion and extension of our scientific research and development activities. The NSRRC is committed to making the power of synchrotron light sources available to more cross-field applications, building new experimental equipment, developing new capabilities, and nurturing scientific talents. With the continuous sharing of new technologies and by supporting scientific research activities, the NSRRC has, from its foundation, sought opportunities for local and international collaboration to build on previous successes and to encourage new partnerships that contribute to scientific progress. The NSRRC has formalized relationships with a number of institutions and universities by establishing bilateral collaboration and signing the Memoranda of Understanding (MOU). These initiatives are important for accelerating the pace of growth in research and development that is beneficial to all scientists and people around the world.

The NSRRC has forged alliances with a range of partners who offer complementary skills, research experiences, and activities. In 2024, the NSRRC had MOUs and collaboration agreements with the following international synchrotron facilities and research institutes in four aspects.

1. Contract beamlines/endstations

- **TPS 02A** Brain Imaging Beamline with Academia Sinica and National Tsing Hua University
- **TPS 13A** Bio-SAXS Beamline with Academia Sinica
- **TPS 27A** STXM/Ptychography Endstation with Tamkang University
- **TPS 39A** nanoARPES Endstation with Taiwan Consortium of Emergent Crystalline Materials
- **TPS 45A1** NSRRC-MPI Beamline with Max-Planck-Institute for Chemical Physics of Solids
- **TPS 45A2** TKU Endstation with Tamkang University

2. International collaboration on beamline/endstation operation and maintenance

- Two Taiwan contract beamlines—**SP 12B** and **SP 12U** at SPring-8, Japan



NSRRC renewed the letter of intent with National Experimental High School at Hsinchu Science Park.

- Cold neutron triple-axis spectrometer—**SIKA** at ANSTO, Australia

3. Collaborations with research institutes and universities

- Taiwan: Academia Sinica, National Atomic Research Institute, National Center for High-performance Computing, National Central University, National Cheng Kung University, National Chung Cheng University, National Chung Hsing University, National Experimental High School at Hsinchu Science Park, National Health Research Institute, National Museum of Natural Science, National Pingtung University of Science & Technology, National Sun Yat-sen University, National Taiwan University, National Taiwan University of Science and Technology, National Tsing Hua University, National Yang Ming Chiao Tung University, Tamkang University, Tunghai University
- Asia and Oceania: IVPP/CAS, IAS/Wuhan University, Soochow University, NFPSS, IPR/Osaka University,

ISSP/University of Tokyo, JASRI, RIKEN SPring-8, KEK, Okayama University, Tohoku University, PAL, POSTECH, SLRI, ThEP, SSSL, Alagappa University, ANSTO, AS

- Europe: ALBA, Arinax, DESY, JINR, DLS, MPG, ESRF, SOLEIL, CELLS, DESY, MAX IV, HZB, Global Phasing Ltd, ELI Beamlines, EMBL, Elettra, ESS, CSIC, SOLARIS
- America: ALS, ANL, APS, CHESS, NSLS-II, SLAC, Western University, CNPEM, LSU
- User Agreement: APS, BESSY II, BNL, LBNL, NSLS, ORNL

4. Joint graduate programs with Taiwanese universities

- Structural Biology PhD Program with National Tsing Hua University, Academia Sinica, National Health Research Institutes
- International PhD Program for Science—Synchrotron Radiation & Neutron Beam Application with National Sun Yat-sen University
- Science and Technology of Synchrotron Light Source with Graduate Institute of Applied Science and Technology, National Taiwan University of Science and Technology

Conferences, Forums, and Symposia

Synchrotron Radiation Forum at the 2024 PS Annual Meeting

The 2024 Annual Meeting of the Physical Society (PS) of Taiwan took place at National Central University from January 24 to 26. Dr. Tzu-Hung Chuang of the NSRRC organized the satellite meeting entitled “The emergent opportunity for scientific research on sustainable energy materials by high-brilliance synchrotron light sources” as the NSRRC forum on the first day. Sustainable energy has become one of the most prominent fields of research and development in recent years. After the launch of the new high-brightness synchrotron light source, with the support of the National Science and Technology Council and the joint efforts of NSRRC colleagues and users, TPS has achieved fruitful research results using various energy materials. During the PS Annual Meeting, which gathered almost all Taiwanese physicists, the NSRRC seized the opportunity to present and discuss the new scientific research opportunities that synchrotron radiation can bring to the energy materials field. The forum was conducted in the form of invited talks. Focusing on different energy

materials, six experts from related fields were invited, including four NSRRC users and two scientists from the NSRRC, to share and discuss the recent research findings and measurement techniques in each subfield. The forum attracted nearly 100 participants.



The Synchrotron Radiation Forum was held at the 2024 PS Annual Meeting in Taoyuan.

Synchrotron Radiation and Neutron Research Forum at the 2024 MCPST Annual Meeting

The 2024 International Conference on Modern Challenges in Polymer Science and Technology (MCPST) was held on January 24 and 25 at National Cheng Kung University. On the second day of the conference, Dr. Wei-Tsung Chuang of the NSRRC held the Synchrotron Radiation and Neutron Research Forum. As energy-related studies have become one of the most popular themes among interdisciplinary research, the forum’s topic was set as “New opportunities for metal-organic polymer catalysts” this year. Through this topic, the organizers aimed to attract users from the polymer field who are involved in the synthesis of organic systems, conjugated polymers, or macromolecular frameworks. The forum was presented by the following NSRRC scientists: Drs. U-Ser Jeng, Chun-Ming Wu, Chih-Wen Pao, and Yu-Chun Chuang, each providing explanations for useful experimental techniques. Several users were invited to share recent research findings, including Prof. Lai Ying-Huang from Tunghai University, Prof. Chen Yi-Hong from Wuhan University, and Prof. Guo Jun-Hong from Yang

Ming Chiao Tung University. Through exchanges and discussion in the forum, NSRRC sought to attract potential users to become experimental practitioners in the future.



The Synchrotron Radiation and Neutron Research Forum was held at the 2024 MCPST Annual Meeting.

Synchrotron Light Source Forum at the 2024 Chemistry National Meeting

The 2024 Chemistry National Meeting was hosted by Tamkang University from March 29 to 31. On the second afternoon of the meeting, NSRRC held the NSRRC forum entitled “Applications of Synchrotron Light Source in Chemistry.” Four NSRRC scientists, Drs. Jeng-Lung Chen, Yu-Chun Chuang, Lee-Jene Lai and Shu-Chih Haw, made presentations on the advantages of and opportunities for synchrotron-radiation techniques in chemical research, including X-ray absorption spectroscopy, synchrotron powder diffraction, and X-ray tomography. Further, four users shared their experiences of using synchrotron-based techniques in their studies regarding the symmetry-mode analysis of Hg-containing double perovskites, energy storage applications, bioinspired catalysis and bioconjugated protein structural dynamics, and electrochemical oxygen evolution reaction. This forum provided an opportunity for discussions on potential future research and collaboration directions.



The NSRRC forum of Applications of Synchrotron Light Source in Chemistry was held at the 2024 Chemistry National Meeting.

Asian Forum for Accelerators and Detectors 2024 (AFAD2024)

The Asian Forum for Accelerators and Detectors (AFAD) in 2024 was hosted by the NSRRC from April 17 to 19 in Hsinchu. AFADs are held annually under the guidance of the Asian Committee for Future Accelerators to strengthen collaboration between accelerator laboratories across Asia and Oceania in the development of accelerator-related technologies and their applications. The forum organizers invited leading experts and scholars in the fields of particle accelerators and detectors, both domestically and internationally, to share their recent research and development in their facilities. The event facilitated extensive face-to-face exchanges between domestic researchers, young scholars, and international experts on key technologies for them to stay updated on the latest developments in particle accelerators and detectors.

The major topics discussed in the forum included accelerators and their related technologies for photon science; detector technology development; accelerator technologies for industrial and medical applications; innovative accelerator techniques; accelerator and its related technologies for hadron (neutron) science; network and computing; and cryogenics, cryomodule, and superconducting technology for accelerators. The forum was held in a hybrid format, both in person and online, with a total of 174 participants.



AFAD2024 was held at the NSRRC in April.



Hard X-ray Emission Spectroscopy Technical Forum

The Hard X-ray Emission Spectroscopy Technical Forum was held to give information on hard X-ray emission spectroscopy techniques at the TLS 13B2, TPS 38A, and TPS 47A beamlines. NSRRC scientists gave presentations on the construction and progress of the TPS 38A hard X-ray emission spectroscopy optics and shared recent experiment results using X-ray emission spectroscopy at the Taiwan contract beamlines at SPring-8 conducted under the Interdisciplinary Program in energy. In the forum, distinguished speakers were invited to share their insights on future promising research directions, including Prof. Way-Faung Pong from Tamkang University, Prof. Hao Ming Chen from National Taiwan University, Prof. Chun-Chuen Yang from National Central University, and Prof. Jin-Ming Chen from National Yang Ming Chiao Tung University. The organizers invited several young and promising users to participate in the forum, with the hope of gathering users and research topics during the beamline

construction phase, fostering collaboration and discussions for future research.



The Hard X-ray Emission Spectroscopy Technical Forum presented the latest hard X-ray emission spectroscopy techniques at the NSRRC.

Third Joint International Symposium of the NSRRC and IPR Osaka University



The 3rd Joint International Symposium of the NSRRC and IPR Osaka University was held in May 2024.

The 3rd Joint International Symposium of the NSRRC and Institute for Protein Research (IPR) of Osaka University—Establishment of the Structural Biology Network in Asia and Oceania was held on May 21 and 22 at the NSRRC. This was the third symposium in the series, with the first and second held in 2015 and 2017, respectively. The third symposium, originally scheduled for 2020, was delayed due to the pandemic and thus took place in 2024. The symposium featured seven international speakers (five from Japan, one from Korea, and

one from Thailand) and six domestic speakers, who shared the updates on their protein crystallography facilities and research findings. A site tour to the latest experimental stations at the TPS was also arranged. This symposium contributed to the establishment of future collaborations and regional connections. Both former and current directors of the Institute for Protein Research at Osaka University participated in the event, and a discussion was held with the director and deputy director of the NSRRC, further strengthening mutual understanding and support for collaboration.

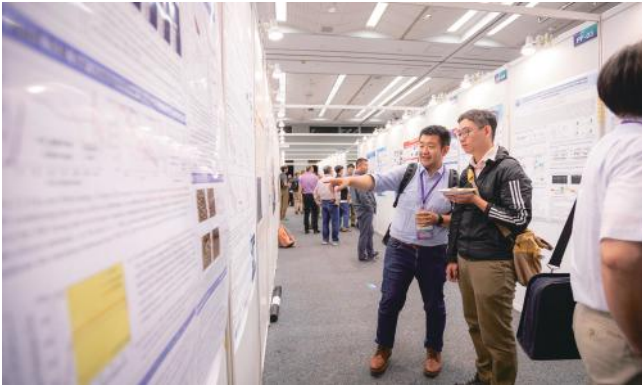
The XIX International Small-Angle Scattering Conference (SAS2024)

The XIX International Small-Angle Scattering Conference (SAS2024), organized by the NSRRC, was held from November 3 to 8 at the Taipei International Convention Center (TICC) in Taiwan. Held every three years since 1965, the SAS conference has become one of the most significant large-scale events for interdisciplinary researchers in the global small-angle scattering community.

SAS2024 brought together nearly 500 scientists and students from around the world, with around 200 talks and more than 200 poster presentations. The conference showcased recent advances and highlighted the broad impact of small-angle scattering techniques on established and emerging scientific fields. In conjunction with SAS2024, a special issue of the *Journal of Applied Crystallography* will be published as the SAS2024 Conference Proceedings, featuring the latest research contributions from registered conference participants.

The conference also included a site tour to the TPS and TLS, where participants witnessed the high-quality synchrotron light capabilities and recently developed small-angle scattering techniques that serve specialized research needs in structural biology, biomedical science, materials science, and the semiconductor industry.

Notably, the program featured the Sow-Hsin Chen Distinguished Lectureship to honor Academician Chen's pioneering contribution to nuclear science and technology in Taiwan. Additionally, the 2024 Taiwan Neutron Science Society Annual Meeting and Neutron Scattering Workshop, held concurrently at the same venue, aimed to advance neutron scattering techniques and enhance Taiwan's international visibility in the scattering community. At the event's conclusion, the conference baton was passed to MAX IV and ESS, the joint hosts of SAS2027 in Lund, Sweden.





SAS2024 brought together nearly 500 scientists and students from around the world.

Synchrotron X-ray and Neutron Science and Applications Forum at the 2024 MRS-T International Meeting

The 2024 Materials Research Society of Taiwan (MRS-T) Annual Meeting took place at National Chung Hsing University on November 15 and 16. The NSRRC, as a co-organizer, held the “Synchrotron Radiation and Materials Forum” on the afternoon of November 16. Dr. Yan-Gu Lin of the NSRRC invited three distinguished domestic scholars to share their research results using synchrotron light sources. Additionally, four NSRRC scientists introduced the applications of advanced technologies at TPS beamlines in materials science, presenting the latest research trends and potential applications to the attendees.

Furthermore, Dr. Lin also promoted the scientific applications of TPS experimental facilities at the seminar organized for principal investigators in materials science during the MRS-T Annual Meeting. He presented cutting-edge experimental techniques and synchrotron radiation analytical methods, and he cordially invited the researchers

and scholars in the fields to conduct experiments at the NSRRC.



Researchers and students attended the Synchrotron X-ray and Neutron Science and Applications Forum at the 2024 MRS-T International Meeting.

Training Courses

NSYSU-NSRRC Joint Summer School on Small-Angle Scattering

The NSYSU-NSRRC Joint Summer School on Small-Angle Scattering was held from July 17 to 28, with focuses on small-angle X-ray scattering (SAXS) and small-angle neutron scattering (SANS). The Summer School provided a platform for junior scientists, engineers of industrial applications, and senior undergraduate and graduate students to exchange scientific ideas, share knowledge, and collaborate. A total of 90 participants gathered at the NSRRC for the program.

The Summer School included not only an introduction to basic concepts, technologies, and applications of SAXS but also hands-on experiments at the NSRRC. Through two weeks of instruction and interaction, participants gained a solid understanding of basic quantum scattering principles, fundamental calculations of two-point spatial correlation functions, and the use of related software for data analysis.

The Summer School attracted participants from various fields, including physics, chemistry, chemical engineering, materials science, and biotechnology, making it a diverse and wide-ranging program. The program was well-balanced, emphasizing both lecturer instruction and participant interaction, fostering a deeper understanding of small-angle scattering across different disciplines and its application in relevant research fields. Through discussions, hands-on practice, group presentations, and outdoor activities, the program strengthened the connections and knowledge exchange between researchers and participants from different fields. The discussions explored the applications of SAXS and its complementary role in other areas of research, such as the analysis of microstructure and molecular forces in such fields as material physics, soft matter science, polymer physics, and biomedical materials.

During the hands-on training sessions, participants operated SAXS experimental stations in groups, collecting and analyzing SAXS data, thereby achieving immediate practical benefits from their learning. At the conclusion of the course, a group-based problem-solving competition was held, allowing participants to present their learning outcomes. Finally, participants were encouraged to continue their research by using NSRRC experimental facilities, paving the way for long-term connection to the synchrotron radiation research and development.

Through the Summer School, it was observed that these domestic participants were on par with their international counterparts, highlighting the importance of continuing to develop and cultivate SAXS techniques in Taiwan. This would ensure Taiwan's academic research competitiveness in relevant fields and provide a strong talent pool for the construction, operation, and maintenance of related facilities in the future.



The NSYSU-NSRRC Joint Summer School emphasized SAXS and SANS.

Summer Internship

The summer internship program has been one of NSRRC's cornerstones for educational outreach, targeting undergraduate students, primarily those in their second to fourth years, since 2013. The program provides systematic instruction on synchrotron-related scientific knowledge and hands-on training at TLS and TPS beamlines. A total of 32 students were accepted into the 2024 Summer Internship program held from July 1 to 31.



The objective of the summer internship was to promote synchrotron-related sciences at the undergraduate level.

This year, the internship program specifically encouraged students to engage in discussions and to share their learning experiences. We were pleased to see that students not only worked diligently in their studies but also actively exchanged knowledge and insights gained from their hands-on experiences. They even assisted participants at other experimental stations, shared their findings, and, at the conclusion of the program, exchanged contact information.

This internship program was a resounding success. It trained over 30 undergraduate students in synchrotron technologies and experimental techniques while also fostering valuable connections between NSRRC and universities. It played a key role in NSRRC's future talent development and in strengthening ties with leading researchers.

Focal-Plane Array Infrared Microspectroscopy Training Course

The Focal-Plane Array Infrared Microspectroscopy Training Course was held on July 11 and 12 at the NSRRC. There were 31 resident physicians, industry professionals, university laboratory assistants, graduate students, and undergraduate students in the training course. The participants were users and potential users from diverse fields, ranging from biomedical sciences, earth and environmental sciences, space science, electrochemistry, and biomaterials and nanomaterials to archaeology.

The training course aimed to provide synchrotron radiation

knowledge on Fourier transform infrared spectroscopy, safety operating procedures at the experimental stations, various measurement techniques in infrared spectroscopy, infrared imaging construction techniques, spectroscopic data processing and analysis, and the use of image processing application programs. Participants gained hands-on experience at the infrared microspectroscopy endstation and its experimental facilities, enabling them to understand the spectral information provided by infrared microscopy and the design of experiments to address the scientific issues that they might encounter in the future.



Users and potential users of NSRRC infrared beamlines participated in the Focal-Plane Array Infrared Microspectroscopy Training Course and practiced analyzing data at TLS 14A1.

Summer School on Free Electron Lasers

The Summer School on Free Electron Lasers (FEL) 2024 was held at the NSRRC from July 15 to 19. A total of 26 students participated in the 5-day program. The FEL are widely recognized by the international accelerator community as one of the most important light sources for the future. To train scientists dedicated to FEL science and technology, the school offered extensive knowledge of this emerging technology and its applications in electronics, biomedical science, physics, chemistry, and materials science.

During the summer school, renowned international FEL experts and domestic scientists who conduct research in this field or have conducted experiments using FEL were invited to give lectures in English. This course was designed to help students gain a deeper understanding of FEL; spark their interest in FEL, accelerator technologies, scientific experiments and applications; and make them aware of the demand for this crucial fourth-generation light source. This course trained domestic students, fostering the development of accelerator technology research and applications and addressing the need for talent in industry, medicine, and research.



Summer School on FEL aimed to cultivate scientists in FEL accelerator technologies.

Protein Crystallography Training Courses I and II

The Protein Crystallography Training Courses were held from July 22 to 26 and from July 29 to August 2. Each course consisted of 18 first-year master or PhD students, newly hired postdoctoral fellows, and research assistants, who were from 28 different laboratories. The goal of the training courses was to disseminate experimental techniques of macromolecular crystallography to researchers and students with an interest in using this specific method to further the scope of their research. The courses provided lecture programs and hands-on training, which covered a broad spectrum of topics on synchrotron-based protein crystallography, ranging from the crystallization of proteins, data collection strategy, phasing techniques, and radiation damage on protein crystals to the structure determination of proteins. The knowledge and skills that the students acquired from the courses broadened their perspectives on their research and were essential for experiments in biotechnology and pharmaceuticals.



Protein Crystallography Training Courses offered hands-on sessions to practice growing crystals, collecting data, and analyzing diffraction data.

Training Course on the Data Analysis of X-ray Absorption Spectroscopy

The Training Course on the Data Analysis of X-ray Absorption Spectroscopy was designed to deepen participants' understanding of data acquisition and analysis. The course was held at the NSRRC on August 6 and had 74 attendees. NSRRC scientists gave lectures on the experimental principle and the setup and application of X-ray absorption fine-structure (XANES) data collection. Two common software packages used for data acquisition and the analysis of X-ray absorption spectra, Athena and Artemis, were explained, and the wavelet analysis of XANES simulation using the FDMNES code was also demonstrated.



There were 74 users and potential users participating in the Training Course on the Data Analysis of X-ray Absorption Spectroscopy.

NAPXPS/XPS Training Course

In response to the enthusiastic demand for near ambient pressure X-ray photoelectron spectroscopy (NAPXPS), NSRRC organized the NAPXPS/XPS Training Course on August 9. The course included lectures as well as hands-on training in spectrum analysis, and due to time constraints, the number of participants was limited. A total of 33 students and principal investigators attended, consisting of 28 registered participants and five auditors.

During the course, participants raised numerous questions and were eager to apply the techniques to address their research topics. Some principal investigator participants expressed strong interest in conducting experiments at the NSRRC in the future. As a result, this training not only promoted XPS technology but also offered an opportunity to broaden the user base for NSRRC's scientific services.



The NAPXPS/XPS Training Course was attended by 33 students and researchers.

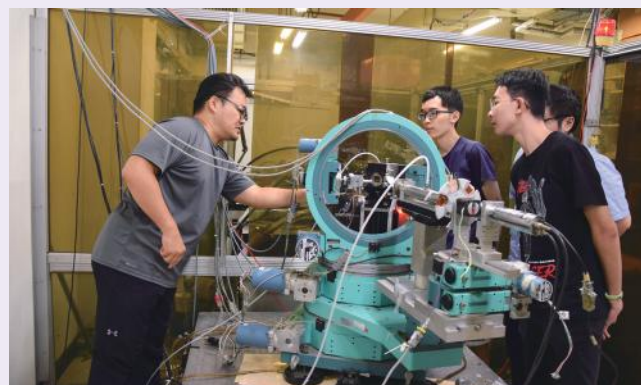
Training Course on X-ray Thin-Film Scattering

The Training Course on X-ray Thin-Film Scattering was held from August 13 to 16 with the aim of providing background knowledge on X-ray thin-film scattering and hands-on training on experimental operations at TPS 09A. The course was designed for new members in the research laboratories to facilitate the application of X-ray scattering in thin-film system research.

This training course consisted of theoretical instruction on the first two days and hands-on practice on the last two days. A total of 40 participants were admitted for the

theoretical sessions, and 20 slots were available for the in-house practice for X-ray diffraction experiments. In total, 53 participants (including auditors) from 10 universities and research institutions in Taiwan attended the course.

In previous years, the course focused mainly on TPS 09A. However, this year, a special presentation given by Dr. Jun-Yu Chen was included to introduce the application and research of TPS 25A in thin-film systems, offering participants an additional research perspective.



Participants attended the lectures, followed by hands-on training at TPS 09A as part of the Training Course on X-ray Thin-Film Scattering.

2024 Autumn School for Micro-Sized Single-Crystal Diffraction and High Pressure Techniques

The Autumn School for Micro-Sized Single-Crystal Diffraction and High Pressure Techniques was held for the first time in 2024. The training course introduced crystallography theory, the operation of single-crystal and high-pressure experimental equipment, and hands-on training for single-crystal data analysis software at the TPS 15A beamline. A total of 67 participants attended the course, including 14 instructors and 53 students, from diverse fields, such as physics, chemistry, materials science, condensed matter physics, and earth sciences.

Most participants had limited knowledge of crystallography, and some had no practical experience in data collection or crystal structure analysis. After attending the autumn school, students expressed a strong interest in becoming users and conducting experiments at the NSRRC.



The Autumn School for Micro-Sized Single-Crystal Diffraction and High Pressure Techniques was held for the first time in 2024.

Learning Opportunities for NEHS Students

NSRRC began to provide learning opportunities for the National Experimental High School (NEHS) at Hsinchu Science Park in 2019. In 2020, NEHS selected 23 second-year high school students to participate in experiments with NSRRC scientists. The students first took thematic classes on radiation safety and research and science related to synchrotron radiation, and then they began thematic studies in a group with NSRRC scientists for an academic year.

This year, eight research teams at the NSRRC provided students with nine scientific research topics, including enhancing photocatalytic effects by modifying TiO_2 nanoparticles with two-dimensional material quantum dots; application of projection X-ray microscopy and

transmission X-ray microscopy in paleontological fossils, and the internal three-dimensional structure of materials; preparation of protein crystals and exploration of crystal structures; versatile membrane proteins in genes and applications in drug deliveries; lipid micelle in genes and applications in drug deliveries; single-crystal analysis of wave-particle duality and the photoelectric effect; developing materials for single-photon sources; crystals growth and structural analysis by using X-ray diffraction; and studies of structures and properties of biomolecules in aqueous solutions.

The learning opportunities helped to sow the seeds of knowledge in brilliant high school students, providing them with opportunities to explore a national research center.

Appendix



List of Publications

Publications (including conference proceedings) listed here are based on the results obtained from facilities associated with the NSRRC

Notes:

1. Asterisks (*) indicate corresponding authors. Their Chinese names are shown in parentheses if applicable.
2. Publications are sorted by endstation and listed in alphabetic order according to the last name of the first author.
3. Publications using more than one beamline are listed in corresponding beamline sections.
4. Publications marked © are selected to report in the sections of Research Highlights.

Experiments Performed at NSRRC Beamlines

TPS 05A Protein Microcrystallography

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- C.-C. Chen, H. Li, J.-W. Huang, and R.-T. Guo*(郭瑞庭), "Structural and Molecular Insights of Two Unique Enzymes Involved in the Biosynthesis of a Natural Halogenated Nitrile", *FEBS J.* **291**, 5123 (2024).
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TPS 13A Biological Small-angle X-ray Scattering

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TLS 01A1 SWLS – White X-ray (PRT 75%)

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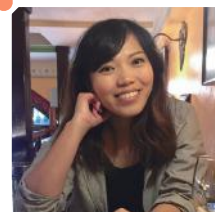
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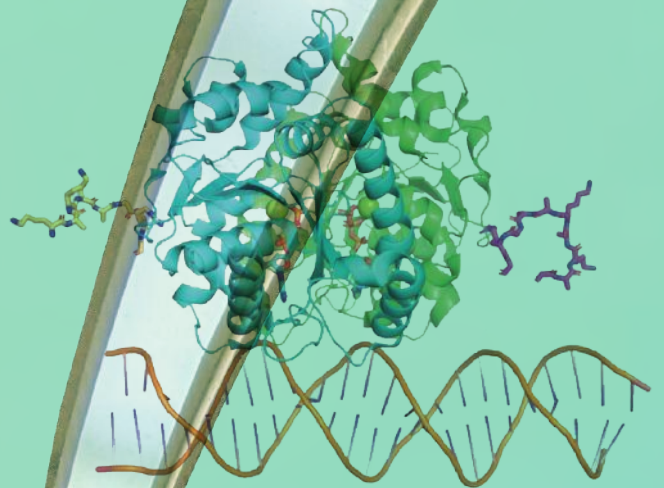
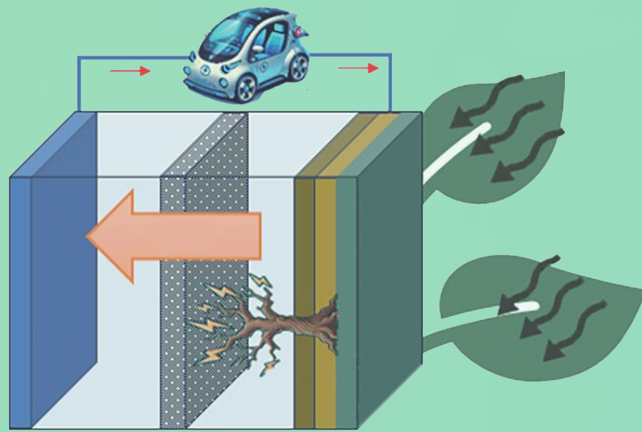
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