ISSP

ACTIVITY REPORT OF SYNCHROTRON RADIATION LABORATORY

2024

Activity Report 2024
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Preface

The Synchrotron Radiation Laboratory (SRL) of the Institute for Solid State Physics (ISSP) continues to promote advanced materials science through the development of cutting-edge soft X-ray spectroscopic techniques at our three research sites in Sendai, Harima, and Kashiwa. Building upon the foundation established over the past years, FY2024 has been a milestone year marked by the long-awaited start of user operations at the new 3 GeV synchrotron facility NanoTerasu and by the rapid progress of next-generation X-ray and laser spectroscopy platforms.

The official launch of NanoTerasu on April 9, 2024 marked a major turning point for our soft X-ray research program. During the first half of the year, more than 30 user proposals were accepted, and the number exceeded 40 in the second half, reflecting strong national expectations for this new facility. The storage ring, operated by the NanoTerasu team, has reached a routine current of 200 mA, delivering exceptionally bright soft X-ray radiation. With the beamlines BL07U and BL08U achieving a spectral resolving power of $E/\Delta E > 15{,}000$, we were able to conduct cutting-edge spectroscopic experiments that were previously unfeasible. At the HORNET-II station, RIXS measurements reached energy resolutions on the order of $E/\Delta E \approx 10{,}000$ in the 400–710 eV range, enabling detailed studies of low-energy elementary excitations. The APXPS station achieved stable operation up to 1 atm, allowing true ambient-pressure in-situ chemical reaction studies, an important step forward in surface science. In 2024, research using BL07U and BL08U resulted in three refereed publications, accompanied by one press release and one news release. Continuous upgrades to beam stability, polarization control, and energy-range extension are underway to further strengthen the capabilities of NanoTerasu.

At the Harima office, the soft X-ray beamline BL07LSU of SPring-8, now reconfigured by RIKEN, has been used as an R&D beamline for next-generation X-ray imaging and optics technologies. A spatial resolution of ~50 nm has been achieved through the development of advanced optical components such as mirrors, gratings, and by further refining ptychographic imaging techniques. Work is underway to extend this system into a four-dimensional spectromicroscopy platform integrating spatial, temporal, and energy domains. At the X-ray free electron laser SACLA, experiments now routinely probe femtosecond-scale dynamics of electrons, spins, and lattice vibrations, and new exploratory studies targeting nonlinear optical responses and X-ray-induced phase transitions have been initiated.

The Kashiwa E-building continues to serve as a hub for high-resolution laser spectroscopy. The high-resolution spin- and angle-resolved photoemission spectroscopy (SARPES) system, which has achieved an energy resolution of 1.7 meV, underwent major stabilization improvements in 2024. Introduction of the fundamental wave into the optical path successfully compensated for long-term degradation of the laser amplifier, resulting in significant improvements in beam focus, intensity stability, and reproducibility of band-dispersion measurements. Planned upgrades include a new high-power optical parametric amplifier (OPA) for tunable pump-probe experiments, enabling studies of nonlinear optical responses and photoinduced phase transitions under strong excitation. Enhancements of real-time laser diagnostics using FROG and related tools are also in progress. The time-resolved soft X-ray spectroscopy (TR-SX) station with the 2D ARTOF analyzer has also continued to evolve,

supporting both 6 eV photoemission and sub-picosecond time-resolved studies. Work is ongoing to introduce an ultra-high-speed readout and visualization system to improve measurement efficiency.

Through the combined use of high-brilliant NanoTerasu, advanced R&D at Harima and SACLA, and state-of-the-art laser spectroscopy in Kashiwa, SRL is steadily advancing toward a fully integrated research platform that covers spatial, temporal, and spin-resolved spectroscopies from the vacuum ultraviolet to the soft X-ray regions.

November 15, 2025
Yoshihisa Harada
Director of SRL-ISSP

1. Status of spin-and angle-resolved photoelectron spectroscopy with laser light at LASOR

Spin and angle-resolved photoemission spectroscopy (SARPES) stands as a powerful experimental technique that provides detailed information about the occupied electronic states in solids, including their energy, momentum, and spin. Recetnly, the growing interest in Rashba surface states and topological materials with spin-polarized electronic structures due to strong spin-orbit interactions has highlighted the importance of spin-resolved measurements. These spin-resolved experiments require high energy resolution and sufficient photoelectron yield rates to effectively detect small energy scales on the order of several meV. In response to these criteria, we successfully developed a high-energy resolution SARPES setup at the Laser and Synchrotron Research Center (LASOR) in the Institute for Solid State Physics (ISSP), using a vacuum-ultraviolet (6.994-eV) laser and spin detectors with the very-low-energy-electron-diffraction (VLEED) [1]. Since our initiative of developing the laser-SARPES in FY2014 and the commencement oft he facility for collaborative research in FY2015, our SARPES station has been instrumental in obtaining precise spin-resolved electronic structures near the Fermi level in solids.

Our laser-SARPES setup consists of an analysis chamber, a carousel sample-bank chamber connected to a load-lock chamber, and a molecular beam epitaxy (MBE) chamber, all connected via ultra high vacuum (UHV) gate valves. The hemispherical electron analyzer, a custom-made ScientaOmicron DA30-L, is designed to incorporate a VLEED-type spin detector. The available photon sources for electron excitation include the 6.994-eV laser, generated as the 6th harmonic of a high-power Nd:YVO4 quasi-continuous wave laser, and a helium discharge lamp (VG Scienta, VUV5000). In the MBE chamber, various instruments for surface evaluation and preparation, such as evaporators, low-energy electron diffraction, a sputter gun, and a quartz microbalance, can be installed, with samples heated by direct current heating or electron bombardment. The carousel chamber offers UHV storage for up to 16 samples. Spin-polarized states have been studied in both the bulk and surface of various topological materials, including magnetic and superconducting ones, atomic layers, and ferromagnetic compounds.

In FY2018, we began upgrading our laser-SAPRES system by integrating a pulsed laser to establish a pump-probe measurement setup. The newly installed pulsed 10.7-eV laser system [2,3], based on ytterbium fiber, achieves a 270-fs pulse duration, 1-MHz repetition rate, and high power through chirped pulse amplification, developed by the Kobayashi group at LASOR in ISSP[4]. This advanced system, as shown in Fig. 1, allows us to measure optically excited electron populations in unoccupied bands across the energy and momentum space and to track pump-induced ultrafast dynamics of both charge and spin. In addition to offering significant capabilities for the time-resolved measurements, the 10.7-eV laser system provides wider momentum information than lower photon energy sources such as 6.994-eV laser and offers better momentum resolution and polarization controls than the

helium discharge lamp. The polarization of the 10.7-eV probe laser, generated through third harmonic generation in Xe gas, can be selectively controlled using an MgF_2 half-wave plate, while the pump photon energy is selectable between 1.19-eV and 2.38-eV to accommodate a variety of band-gap materials. The original 6.994-eV light source remains available by adjusting mirrors and lenses in the vacuum beamline.

In FY2023, nine research proposals from external researchers were accepted and conducted using our SARPES setup, resulting in the publication of two research papers [4,5]. These proposed studies explored spin-polarized states across various platforms, including half-metallic ferromagnets, Dirac semimetals, topological insulators, two-dimensional materials, ferromagnetic oxides, and organic compounds. To support these advanced studies and further improve our experimental capabilities, we have also been focusing on upgrading the 10.7-eV laser system to enhance the stability of the pulsed light. This includes the development of laser evaluation systems, such as an autocorrelation measurement setup and Frequency-Resolved Optical Gating (FROG), as well as the integration of a new rod fiber amplifier into the system, which allows for higher output light.

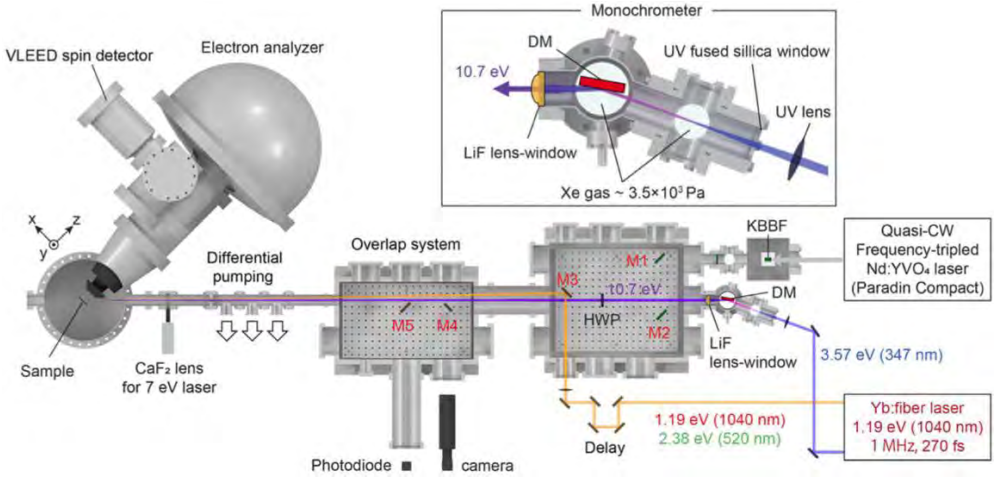

Fig. 1 Scaled layout of the 10.7-eV pulse laser beamline for tr-SARPES[4].

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2. Activities High Harmonic Laser Experiments (E-Labo)

1) IVESTIGATION OF SPIN POLARIZATION IN CVD-GROWN GRAPHENE ON NI(111) SUBSTRATE

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Graphene is a single atomic layer of carbon atoms arranged in a honeycomb structure. Since its first isolation in 2004, many of graphene's extraordinary properties have been explored, such as being a gapless semiconductor in which the valence and conduction bands meet exactly at the Fermi level (E_F) [1,2], exhibiting massless Dirac fermions, high electron mobility, and unique electrical transport properties [3-5]. Graphene also displays fascinating mechanical properties [6], outstanding electrical conductivity, and significant quantum phenomena such as the quantum Hall effect [7] and the quantum spin Hall effect [8]. Notably, pure graphene is an intrinsically nonmagnetic semiconductor material, and lacks localized magnetic moments, which hinders its use in spintronic devices. To overcome this challenge and enhance the potential of graphene for practical use, several techniques have been developed to tailor its electronic and magnetic properties, such as doping graphene with magnetic impurities [9,10] or placing it in close proximity to a magnetic material [11,12]. The former approach involves substituting carbon atoms with magnetic atoms, which can induce ferromagnetic behavior and open a gap in the band structure, thereby modifying the material's electronic and magnetic characteristics. However, such dopants can also introduce vacancies and reduce conductivity, which are detrimental to graphene's exceptional electronic properties. Meanwhile, the latter technique induces spin polarization in graphene via the proximity effect, in which the magnetic exchange field from the adjacent ferromagnetic material is transferred to the graphene layer, thereby generating a magnetic moment within it. The close lattice match between graphene and ferromagnetic substrates such as Ni(111) leads to strong hybridization between the graphene π states and the Ni 3d states [13], resulting in a pronounced shift of the π-band toward higher binding energies (approximately 2.8 eV). To avoid this large shifting, we have studied the electronic structure and spin polarization of chemical vapor deposition (CVD)-grown graphene on Ni(111) (G/Ni(111)).

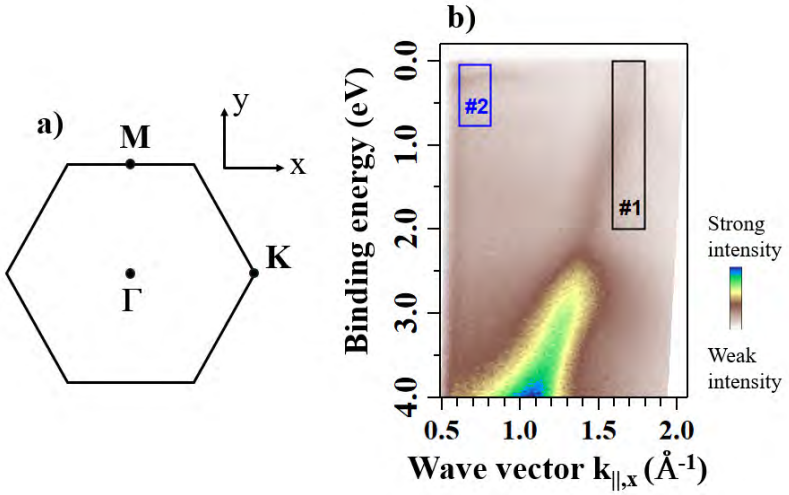

Fig. 1 (a) The first Brillouin zone of graphene. (b) The ARPES mapping of the π band of CVD-grown graphene on Ni(111). The black and blue rectangles represent the areas of the spin-resolved photoemission observation.

Here, we examined the reproducibility of spin polarization in the π-band of G/Ni(111) and Ni 3*d* states by spin- and angle-resolved photoemission spectroscopy (SARPES) measurements conducted at the Institute for Solid State Physics (ISSP). The *x*-(*y*-)axis of the spin detector coincides with the **ΓK** (**ΓM**) direction of the first Brillouin zone of graphene, as shown in Fig. 1(a). The G/Ni(111) system was magnetized parallel to the *x*-axis using an external magnetic field, and the *x*-component of the spin polarization was measured in the SARPES experiment.

Figure 1(b) shows the ARPES intensity map of the π band of G/Ni(111) along the **ΓK** direction, obtained using a photon energy of 21.2 eV from the He Iα radiation. The spin signal was acquired from the black rectangular region (#1), corresponding to the graphene π-band, and from the blue rectangular region (#2), corresponding to the Ni 3*d* states. We confirmed that the Dirac point is located close to the Fermi level (E_F). We also observed spin polarization near the Dirac point in the π-band of graphene. Spin polarization in the Ni 3*d* state region was not confirmed. However, we believe that the observed spin signal still originates from proximity-effect–induced spin polarization in the π-band of G/Ni(111).

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2) SHOCKLEY SURFACE STATES MODIFIED BY ADSORPTION OF ORGANIC ACCEPTOR MOLECULES

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The ability to control and modify the electronic properties of material surfaces is essential for the realization of next-generation devices involving electron and spin transfer at the interface between material surfaces and electrodes. In the field of surface science, the properties of many surface states and techniques for the modification of these surface states have been pursued. In particular, the Shockley state (SS), which is a pseudo-two-dimensional free electronic surface state on the (111) surface of an fcc-metal, is sensitive to various molecular adsorptions and has been the subject of many studies. However, SS is easily and quickly dissipated by irregularly adsorbed molecules. Meanwhile, for regularly arranged molecules on the surface, SS survives, and in some cases, its properties are significantly modified[1]. Specifically, this study focuses on the effect of organic molecule adsorption on the Rashba effect, a phenomenon where the energy bands of the SS undergo spin splitting.

In quasi-two-dimensional electron systems with broken spatial inversion symmetry, such as metal surfaces and semiconductor interfaces, Rashba-type spin-orbit interactions (Rashba effect) are known to occur through atomic spin-orbit interactions[2-5]. The Rashba effect has been demonstrated in quantum wells and thin films based on III-V semiconductors, as well as in two-dimensional electron gases on the metal surface, and is expected to play an important role as an elemental technology in spintronics. Many studies have revealed that Rashba coupling can be modified by the adsorption of atoms and molecules on metal surfaces and by distortions at the semiconductor interface. In recent years, it has become possible to intentionally control the strength of Rashba coupling and design systems for various applications. For example, the adsorption of noble gases or organic molecules on a noble metal (111) changes the strength of Rashba coupling. This is interesting given that the Rashba coupling is determined by the asymmetry of the wave function of the surface electrons in a very narrow region no less than 1 Å from the nucleus of the outermost layer of the metal surface, where molecules adsorbed at least a few Å away from the nucleus can affect the Rashba coupling. In fact, we have found that even highly inert organic molecules, such as normal alkane, can enhance the Rashba splitting observed on the SS of Au(111) by adopting ordered adsorption structures[6].

In this study, we directly observed the changes in the SS when tetracyanobenzene (TCNB: Fig.1) adsorbed in a well-ordered manner on an Au(111) using angle-resolved photoemission spectroscopy. TCNB possesses a cyano-groups at its four molecular terminals, which is a strong electron-withdrawing group, making it a representative acceptor organic molecule. The interaction between TCNB and Au(111) is expected to involve electron transfer to TCNB, and it is of interest to see how this affects the SS.

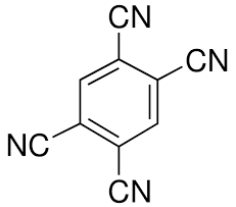

Fig. 1 Molecular structure of TCNB

The Au(111) surface was prepared via repeated cycles of rare gas ion (Ar^+) sputtering at voltages of approximately 1 keV and subsequent annealing at approximately 400–600 °C for 5–10 min. The surface quality was verified using LEED measurements. The TCNB molecular layer on clean Au(111) was prepared using a K-cell in a vacuum chamber. The coverage of the molecular layer was estimated using a quartz crystal microbalance. Angle-resolved photoemission spectroscopy (ARPES) and spin-resolved ARPES (SARPES) measurements were performed at the Laser and Synchrotron Research Center of The ISSP in The University

of Tokyo (Japan) using the sixth harmonic ($h\nu$ = 6.994 eV) of a basic wave of a Nd:YVO_4 quasi-CW laser with a nonlinear optical crystal KBe_2BOF_2 and a hemispherical analyzer (DA30-L, Scienta Omicron).

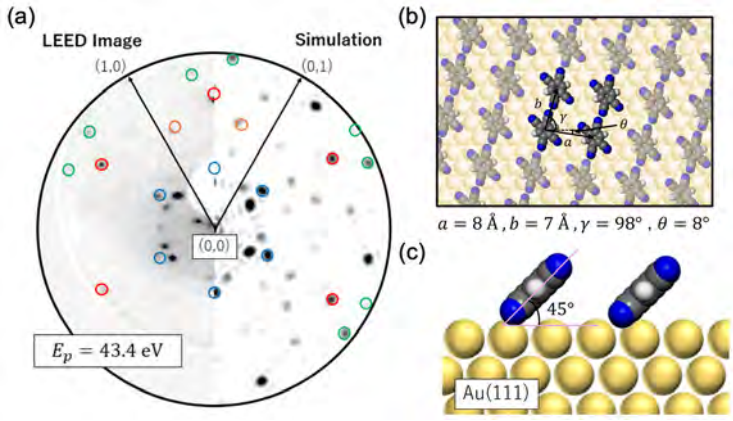

Fig. 2 (a) Low energy electron diffraction (LEED) results for TCNB/Au(111). The left semicircle shows observed LEED pattern, while the right semicircle shows simulated one. (b) TCNB/Au(111) structure used for the LEED simulation. (c) Schematic illustration of the TCNB molecules adsorbed on Au(111).

Fig. 2 shows the LEED results for TCNB/Au(111) and the structure deduced from its analysis. The observation of a distinct diffraction pattern for TCNB/Au(111) indicates that TCNB forms a well-defined adsorbed film on Au(111). Comparing the observed LEED pattern shown in Fig. 2(a) with the simulated LEED pattern based on the TCNB/Au(111) structure shown in Fig. 2(b) reveals a generally good agreement. Furthermore, simulation results indicate that TCNB is oriented on Au(111) with a molecular plane tilted at approximately 45°, as shown in Fig. 2(c).

Figs. 3(a) and (b) show the observed ARPES and SARPES spectra of TCNB/Au(111), respectively. The spin-polarized SS bands were clearly observed around the Γ point. The SARPES measurements revealed that the spin polarization remains high on TCNB/Au(111). Furthermore, the Rashba splitting observed for the SS on TCNB/Au(111) was found to be slightly larger than that on bare Au(111). These results indicate that adsorption of TCNB modifies the surface charge density of Au(111) without altering its translational symmetry parallel to the surface plane. We are currently analyzing the mechanism behind this increased Rashba splitting.

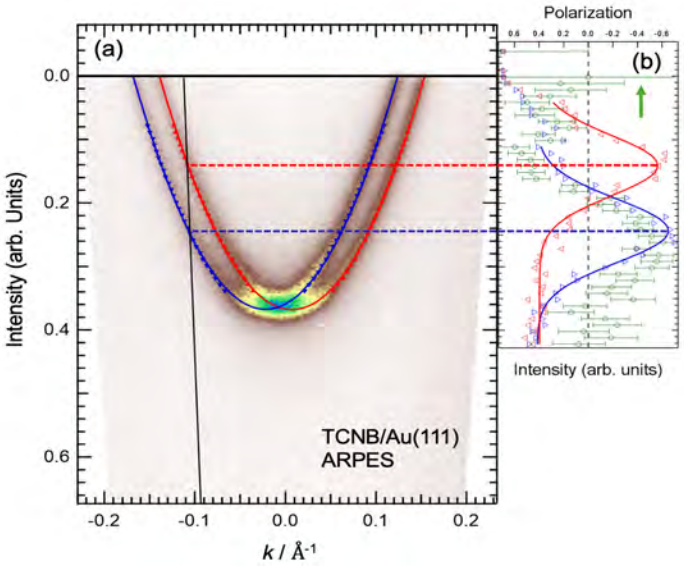

Fig. 3 (a) ARPES and (b) SARPES results of TCNB/Au(111).

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3) SPIN-ARPES MACHINE TIME ACTIVITY REPORT February 2025

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(A)Overview

The first objective of our experiment was to study the spin polarization of photoelectrons from the charge-density-wave (CDW) material $TiSe_2$ and the non-CDW $TiTe_2$ at low photon energy with high resolution. In our previous experiment done in 2023 with laser light source of h**ν**=6.994eV, we observed an unexpected spin texture of $TiSe_2$, which showed sharp contrast to that observed at high photon energies. As clearly seen in Fig.1(a), the spin texture probed with h**ν**=67eV shows an antisymmetric structure, while at h**ν**=6.994eV it is symmetric, resulting in a non-zero net spin polarization after angle integration. Since $TiSe_2$ hosts a CDW, it was curious to find out whether this could be the cause of this effect in spin polarization. For this purpose, we repeated the measurement on $TiSe_2$, but at an elevated temperature and out of the CDW phase. Additionally, a structurally similar compound with no CDW order, namely $TiTe_2$, has been measured for cross comparison.

The second objective of our experiment was to investigate SnTe above its ferroelectric transition temperature, T_c~110K. As shown in ref.[1], spin-integrated ARPES suggests that SnTe retains its bulk Rashba band splitting above this temperature, indicating persistent structural distortion or polar fluctuations that break inversion symmetry. In this light, it would be of great interest to also study the spin polarization at an elevated temperature, and to directly observe whether the spin signal follows the band splitting.

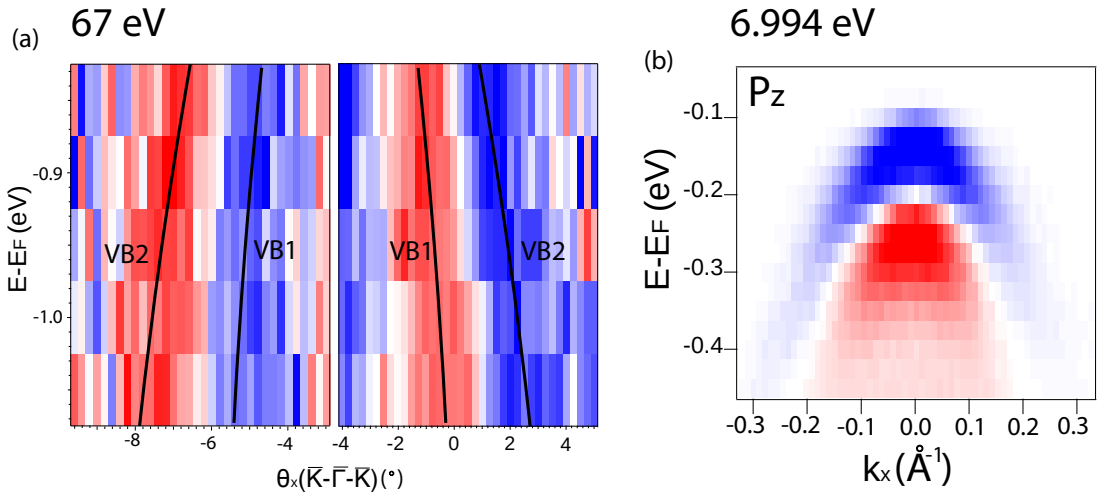

Figure 1: (a) Spin-resolved band map of $TiSe_2$ probed by (a) $h\nu$=67eV and (b) $h\nu$=6.994eV, experiment performed in 2023.

(B)Quality of measurement/data

Thanks to the well-maintained spin-ARPES end station and high-efficiency VLEED spin detectors, spin-resolved bandmaps could be taken with high statistics in all 3 spatial spin directions. The efficient end station, stable light source and excellent support from the lab members allowed us to measure more, and higher-statistics spin-resolved data than expected, and many of them are of significant interest, and are currently being analysed in-depth. The only thing that was missing, and which would have helped us obtain even better results, was the possibility to change the sample azimuthal rotation.

(C)Status and progress of evaluation

The high-quality spin-resolved data allows us to obtain insights into the spin texture with high efficiency, and further to interpret the key influence of spin texture probed by SARPES. The observed symmetric spin texture of $TiTe_2$ and for $TiSe_2$ above the transition temperature, rules out the CDW phase as the cause of this effect, and elucidates a different scheme of photoemission final states. The comparison of SARPES data obtained in 2023 and 2025 also demonstrated the sensitivity of measured spin polarization to the angular orientation of the sample. The measured spin texture of SnTe at elevated temperatures is unprecedented to our knowledge, and it confirms our postulate about the persistence of Rashba splitting above the ferroelectric transition temperature.

(D)Results

For $TiSe_2$, we observed a persistent symmetric spin polarization across the CDW transition temperature. As shown by an EDC of spin polarization at normal emission as a function of temperature in Fig.2(a), except for an upward shifting of the bands, temperature increase induces no observable effect. However, the spin polarization in the z direction shows an opposite structure from data obtained in 2023, as contrasted in Figs. 1(b) and 2(b). If we compare the experimental parameters in the two measurements, the only difference is an azimuthal rotation of the sample by 26 degrees. This sensitivity to experimental geometry may be an indication of the composite basis functions in the initial state, which deserves more investigation.

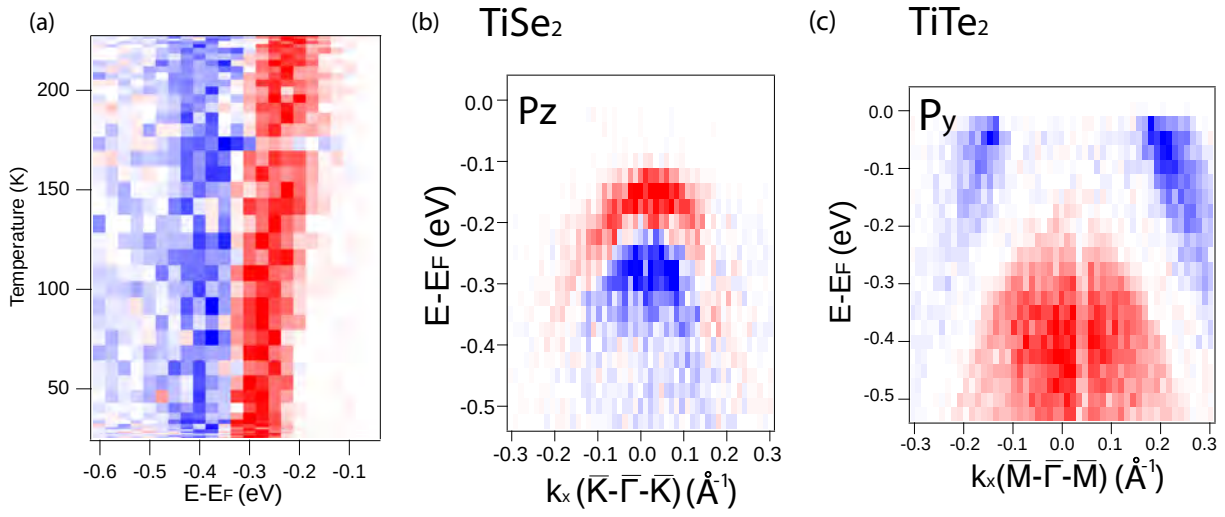

Figure 2: (a) Spin EDC of $TiSe_2$ at normal emission, as a function of temperature (b) spin-polarized band map of $TiSe_2$ in quantisation direction z taken at photon energy $h\nu$=6.994eV (c) spin-polarized band map of $TiTe_2$ in quantisation direction y.

For $TiTe_2$, a similar symmetric spin texture has been observed, which is most pronounced in the y quantisation direction, as shown in Fig.2(c). This result further excludes that CDW order as a factor, and suggests a generally different photoemission final state regime.

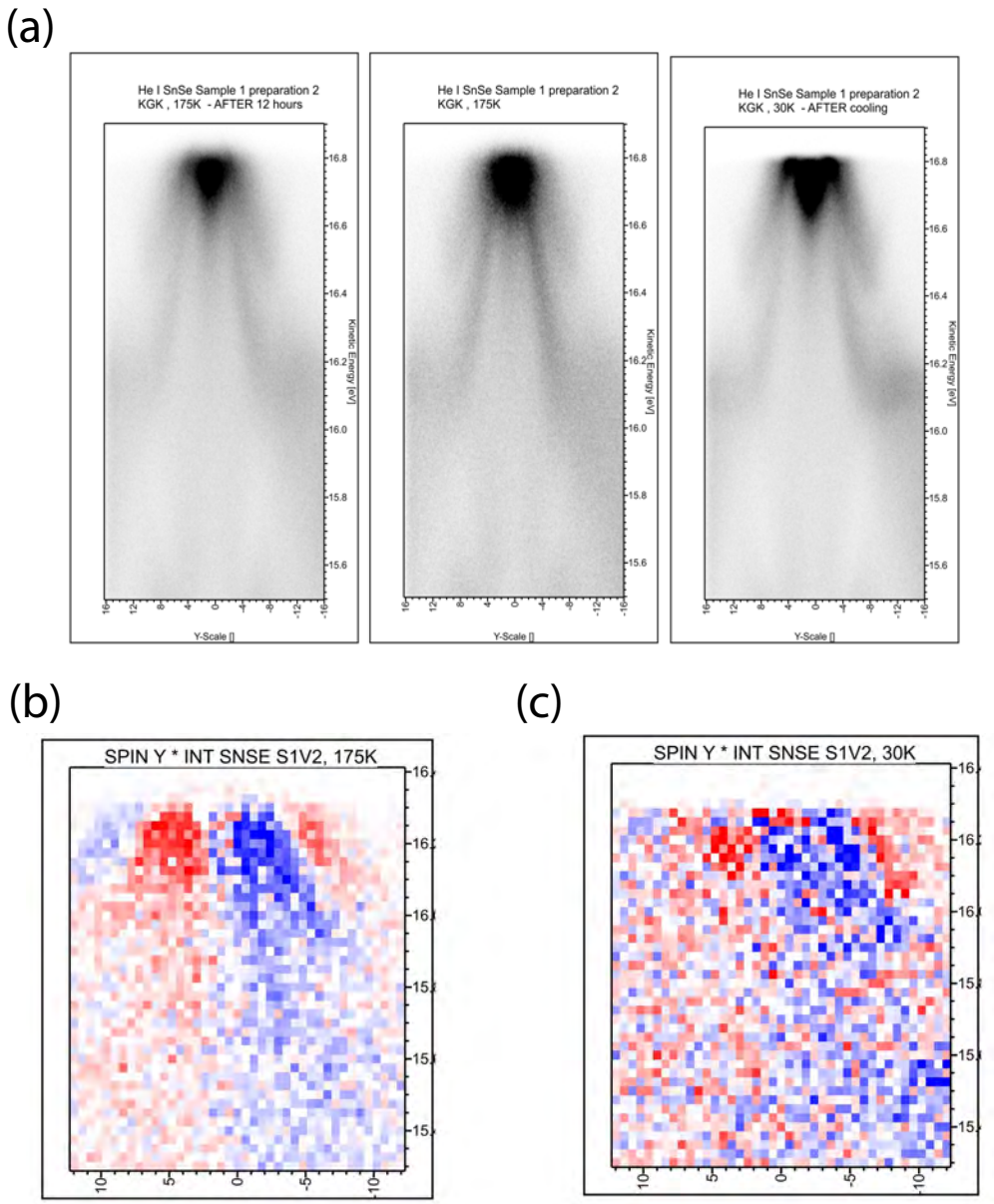

Figure 3: (a-c) Spin-polarized bandmap of $TiSe_2$ taken in **Γ**-K and **Γ**-M orientations, with s- and p-polarized light, (d) spin polarization of $TiSe_2$ in the z-direction, taken in the **Γ**-K orientation and with s-polarized light, at binding energy 0.8eV, (e) spin polarization EDC at the **Γ** point as a function of wave plate angle.

The experiments yielded the, to our best knowledge, first SARPES result at low-h**ν** for SnTe. High-resolution band maps taken with Helium lamp shown in Fig.3(a) confirms the successful decapping of the sample and a good surface quality. We then measured spin-resolved band maps at T=175K and T=30K respectively, as shown in Figs.3(b) and (c). In accordance with our spin integrated results, the Rashba splitting persists above the

ferroelectric transition temperature, indicating an effective polar field, despite the expected rocksalt symmetry in the bulk.

For the publication of our results we are now awaiting one step photoemission calculations with spin resolution by the group of Prof. Jan Minar in Pilsen.

References:

[1] Persistence of Structural Distortion and Bulk Band Rashba Splitting in SnTe above Its Ferroelectric Critical Temperature**.** Frédéric Chassot, Aki Pulkkinen, Geoffroy Kremer, Tetiana Zakusylo, Gauthier Krizman, Mahdi Hajlaoui, J. Hugo Dil, Juraj Krempaský, Ján Minár, Gunther Springholz, and Claude Monney
Nano Letters **2024** *24* (1), 82-88

3. Seminar

(SOR Seminar)

Date: April 22 ,2024

Title: Nanoscale 3D imaging through raster scan X-ray microscopy

Speaker: Zirui Gao (Brookheaven National Laboratory, New York, USA)

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ZHANG Ziheng(2024.10~)

5. Publication List

E-labo. Spin Paper 2024

No.	Title	Authors	Journal	Vol.	Page	Year
1	Visualization of spin-polarized surface resonances in Pb-based ternary topological insulators	Koichiro Yaji, Yuya Hattori, Shunsuke Yoshizawa, Shunsuke Tsuda, Fumio Komori, Youhei Yamaji, Yuto Fukushima, Kaishu Kawaguchi, Takeshi Kondo, Yuki Tokumoto, Keiichi Edagawa & Taichi Terashima	Scientific reports	14	25868	2024
2	Robust Weak Topological Insulator in the Bismuth Halide Bi_4□Br_2I_2.	Ryo Noguchi, Masaru Kobayashi, Kaishu Kawaguchi, Wataru Yamamori, Kohei Aido, Chun Lin, Hiroaki Tanaka, Kenta Kuroda, Ayumi Harasawa, Viktor Kandyba, Mattia Cattelan, Alexei Barinov, Makoto Hashimoto, Donghui Lu, Masayuki Ochi, Takao Sasagawa, and Takeshi Kondo	Physical Review Letter	133	86602	2024
3	Spin-polarized saddle points in the topological surface states of elemental bismuth revealed by pump-probe spin- and angle-resolved photoemission spectroscopy	Yuto Fukushima, Kaishu Kawaguchi, Kenta Kuroda, Masayuki Ochi, Motoaki Hirayama, Ryo Mori, Hiroaki Tanaka, Ayumi Harasawa, Takushi Iimori, Zhigang Zhao, Shuntaro Tani, Koichiro Yaji, Shik Shin, Fumio Komori, Yohei Kobayashi, and Takeshi Kondo	Physical Review B	110	L041401	2024
4	Photoemission angular distribution beyond the single wavevector description of photoelectron final states	Hiroaki Tanaka, Shota Okazaki, Yuto Fukushima, Kaishu Kawaguchi, Ayumi Harasawa, Takushi Iimori, Fumio Komori, Masashi Arita, Ryo Mori, Kenta Kuroda, Takao Sasagawa, and Takeshi Kondo	Physical Review B	109	L241114	2024