BL2A

Success of the World's First Solar Flare X-ray Focusing Imaging Spectroscopic Observation by the Sounding Rocket Experiment FOXSI-4 and Post-Flight Calibration of Its CMOS Sensors

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The solar corona is full of dynamic phenomena such as solar flares. The understanding of these phenomena has progressed step-by-step with the evolution of observation technology in EUV and X-rays from space. But fundamental questions remain unanswered or have not been addressed so far. Our scientific objective is to understand the underlying physics of the dynamic phenomena in the solar corona, covering some of the long-standing questions in solar physics, such as particle acceleration in flares and coronal heating. To achieve this objective, we identify imaging spectroscopy (the observations with spatial, temporal, and energy resolutions) in the soft X-ray range (from ~0.5 keV to ~10 keV) as a powerful approach for the detection and analysis of energetic events [1]. This energy range contains many lines emitted below 1 MK to beyond 10 MK plasmas and a continuum component reflecting the electron temperature.

The soft X-ray imaging spectroscopy is realized with the following method. We take images with a short enough exposure to detect only single X-ray photon in an isolated pixel area with a fine pixel Silicon sensor. So, we can measure the energy of the X-ray photons one by one with spatial and temporal resolutions. When we use a high-speed soft X-ray camera that can perform the continuous exposure with a rate of more than several hundred times per second, we can count the photon energy with a rate of several 10 photons/pixel/second. This high-speed exposure is enough to track the time evolution of spectra generated by dynamic phenomena in the solar corona including solar flares, whose lifetimes are about from several ten seconds to several ten minutes.

For the world's first focusing imaging-spectroscopic observation of a solar flare in X-ray range, we launched a NASA's sounding rocket as the fourth flight of FOXSI sounding rocket series (called "FOXSI-4") on April 17th, 2024 (Fig. 1) and successfully obtained the unprecedented solar flare data using a combination of high-precision X-ray mirrors and high-speed X-ray cameras (Fig. 2). For the soft X-ray cameras, we use fully depleted CMOS sensors with a silicon thickness of 25 μm [2,3], which provide high sensitivity to high-energy X-rays.

FOXSI-4 detected more than 10^7 X-ray photons during an observation period of about 5 minutes. Using these photons, we can make spatially and temporally resolved X-ray spectra from a solar flare (see Fig. 3) and then investigate the physics of the high-energy plasmas generated by the solar flare.

For the precise scientific analysis of FOXSI-4 data, post-flight calibration of the CMOS sensors is indispensable. In FY2024, this was done for the energy range from 0.85 keV to 4.5 keV at UVSOR BL2A.

Fig. 1. Photo of FOXSI-4 launch at Poker Flat Research Range in Alaska.

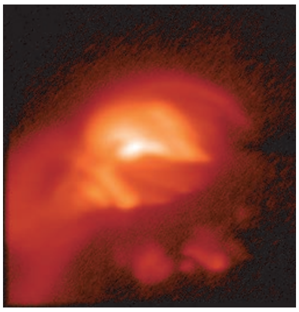

Fig. 2. A solar flare image in soft X-rays observed by the CMOS camera aboard FOXSI-4 sounding rocket.

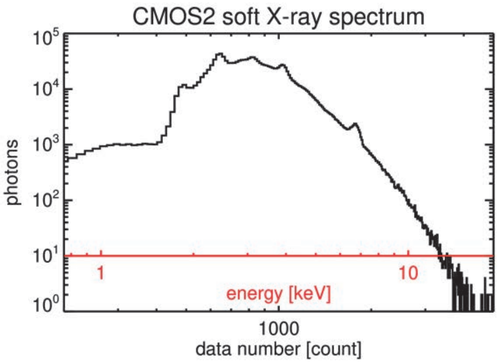

Fig. 3. A soft X-ray spectrum of a solar flare obtained with the CMOS camera aboard FOXSI-4.

[1] N. Narukage *et al.*, White paper of the "soft X-ray imaging spectroscopy", arXiv:1706.04536 (2017).
[2] N. Narukage *et al.*, UVSOR Activity Report **51** (2023) 48.
[3] R. Shimizu *et al.*, Proc. SPIE Int. Soc. Opt. Eng. **13103** (2024) 1310308.

BL2A

Operando Analysis of Battery Materials Using NEXAFS Spectroscopy with a Transportable Potential-Applying Manipulator

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Research on battery materials is actively being conducted from the perspective of resource and energy issues, as well as global environmental concerns. In the development of new battery materials, information on changes in the valence of each element before and after the application of potential to the fabricated battery is essential for analyzing the reaction mechanism. Moreover, operando measurements during analysis enable more accurate reaction analysis. Additionally, in the analysis of battery materials, non-exposure measurements to atmospheric components are indispensable due to the materials' reactivity to atmospheric substances. From this perspective, we developed a manipulator for soft X-ray absorption spectroscopy that allows samples to be transported without exposure to the atmosphere [1].

The elements contained in battery materials exhibit a wide range of absorption edge energies, from lithium to sulfur, chlorine, and even metals. Consequently, depending on the synchrotron radiation facility, it may not be possible to measure all elements on a same beamline, or the facility itself might not be able to accommodate the measurements. Furthermore, due to differences in sample holders across facilities and beamlines, challenges arise when handling rare samples, such as difficulty in making adjustments and reproducing surface conditions. To enable analysis of the surface state of the same sample, we developed a transportable manipulator [1]. This device allows the sample to be directly attached to the sample-fixing part and transported under vacuum or gas atmospheres. The sample-fixing part is connected to a coaxial feedthrough Bayonet Neill-Concelman (BNC) at the top of the device, enabling the measurement of sample current. Furthermore, since the device can be installed on the conflat flange with an outer diameter of 70 mm (ICF70) port, it facilitates cross-beamline or cross-facility measurements. Additionally, to more accurately evaluate the reactions occurring in batteries, we developed a device capable of applying voltage to the battery, allowing for measurements of the actual reaction state under operational conditions [2].

Previously, the soft X-ray absorption spectra of electrodes and solid electrolytes were measured after disassembling batteries that had undergone charge and discharge cycles without exposure to the atmosphere. However, since the samples used were different, the changes due to charge and discharge were not fully reflected. On the other hand, operando observation allows for the monitoring of changes at the same location, which is expected to enable a more accurate evaluation of electrochemical changes. In this study, we analyze battery materials using a manipulator capable of applying voltage.

The sample was attached to the transfer vessel in a glove box under an argon atmosphere and transported to the analyzer. Soft X-ray absorption spectrum of the sample was measured at the BL2A of the UVSOR in the Institute for Molecular Science. The XAFS spectra was obtained using the partial fluorescence yield (PFY) mode at room temperature. Fluorescence detector used was a silicon drift detector (SDD).

Figure 1 shows the time dependence results of the Mg K-edge NEXAFS of MgI_2 under applied voltage. The peak near 1305 eV, which corresponds to metallic Mg, was not observed. This is because the battery had high resistance and not enough electricity flowed, so the electrode reaction did not progress to a detectable range. However, as discharge progressed, the spectrum shifted slightly to the lower energy side. This indicates that the slight changes associated with the reduction reaction at the electrode could be observed using this device.

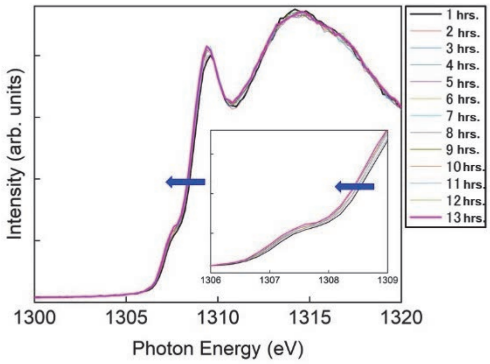

Fig. 1. Mg K-edge NEXAFS of MgI_2 under applied voltage.

[1] E. Kobayashi and A. Inoishi, UVSOR Activity Report **50** (2022) 44.

[2] E. Kobayashi and A. Inoishi, UVSOR Activity Report **51** (2023) 49.

BL2A

Mo L_{III}-edge XANES Study of Formation of Catalytically Active Mo_2C Species on H-MFI Zeolites for Methane Aromatization

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Molybdenum-modified H-MFI zeolite (Mo/H-MFI) is a typical GTL (Gas To Liquid) catalyst for natural gas transformation. High catalytic activity over the Mo/H-MFI is shown for methane conversion to benzene although definite deactivation due to coke deposition has been a serious problem for durable MTB reactivity. It has been revealed that the reactions at high pressure (0.15 - 0.20 MPa) and the addition of H_2 to the CH_4 reactant were each effective in suppressing catalyst deactivation. On these reaction conditions, deactivation process over the catalysts is one of the important points. A cause of the deactivation is based on coke deposition on strong acid sites over H-MFI and structural deactivation on carbonized MoC_x species. Thus, clarification of Mo sites with high and durable activity for methane dehydrogenation is one of the most important subjects. In the present study, Mo L_{III}-edge XANES is applied to characterize the active Mo carbide species on Mo/H-MFI. In the present study, time course of the formation of active Mo_2C species on H-MFI has been investigated.

Mo(5wt%)/H-MFI (Si/Al_2 ratios in H-MFI supports are 23 and 50) catalysts were prepared by impregnation of H-MFI in $MoO_2(acac)_2$-$CHCl_3$ solution, and followed by drying overnight and calcination at 773 K. The methane aromatization reactivity was evaluated at 1023 K in a steady-state reaction by using CH_4(20%)-H_2(2%)-Ar(base) reactant as described in a previous report[1]. Mo L_{III}-edge XANES spectra were obtained in BL2A of UVSOR-IMS in a total-electron yield mode using InSb double-crystal monochromator. REX-2000 (Rigaku) software was used for normalization of each XANES spectra.

It has been summarized that the catalytic MTB activity over Mo/H-MFI with Si/Al_2=50 shows high activity at high pressure (0.15-0.20 MPa), and deactivation rate over reaction time is extremely small. In contract, Mo/H-MFI with Si/Al_2=23 exhibited high activity with significant deactivation over time[2]. For these catalysts, time course of the formation of Mo species during the reaction was investigated as follows. The L_{III}-edge XANES spectra over the Mo/H-MFI catalysts on time course of the MTB reaction in 0.15 MPa are shown in Fig. 1. In both catalysts before reaction, the spectra are almost similar to that of MoO_2, indicating the formation of Mo^{4+} by pre-reduction treatment with CO(2%)-Ar. For Mo/H-MFI in Si/Al_2=23, the XANES spectrum after the reaction for 30 minutes was very similar to that of α-type Mo carbide (α-MoC_{1-x}) with cubic structure, indicating that the Mo species were carbonized reductively at the early stage of the reaction. The XANES edge energy of the active Mo species shifted to the lower energy side with the progression of reaction time, suggesting that reduction by carbonization had further progressed. For Mo/H-MFI in Si/Al_2=50, the XANES spectrum after the reaction for 30 minutes was very similar to that of β-type Mo_2C with orthorhombic structure. It indicates the formation of easily reduced Mo sites to form more deeply carbonized species than that of Mo/H-MFI in Si/Al_2=23. Since the β-Mo_2C active species hardly changed with the progression of reaction time, it is suggested that they exist as stable carbide species. The result is likely to relate to a suppression effect of deactivation.

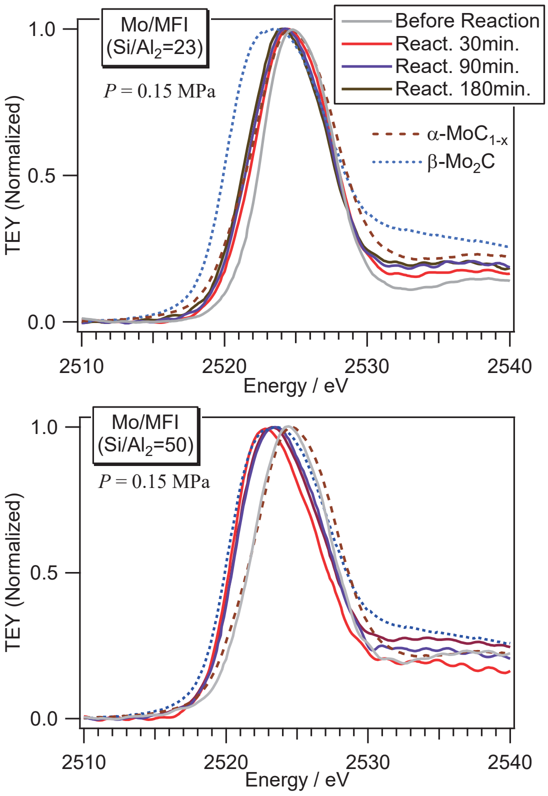

Fig. 1. Mo L_{III}-edge XANES spectra of Mo/H-MFI catalysts in Si/Al2=23 (top) and 50 (bottom) after the MTB reaction at 1023 K.

[1] H. Aritani *et al.*, J. Environm. Sci. **21** (2009) 736.
[2] K. Kuramochi *et al.*, UVSOR Activity Report **51** (2023) 69.

BL2A

Local Structure Investigation of Zn Dopant in Ga_2O_3

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Gallium oxide (Ga_2O_3) is a promising ultrawide bandgap semiconductor for high-voltage and high-power applications. Monoclinic β-phase of Ga_2O_3, which has tetrahedral and octahedral gallium sites, is unintentionally *n*-type doped, and a good control of electron carrier concentrations in the range of 10^{16}–10^{19} cm^{-3} or even higher can be achieved using intentional doping by Si, Sn, Ge and Nb. However, the electrical conductivity of *p*-type in β-Ga_2O_3 is difficult to be realized. First-principles calculations showed that Zn can be a suitable acceptor forming two shallow levels at 0.05 eV and 0.07 eV above the valence band in β-Ga_2O_3 when Zn substitutes Ga in tetrahedral and octahedral coordination, respectively. We utilize halide vapor phase epitaxy growth to synthesize epitaxial layers of β-phase Ga_2O_3 doped with Zn, which can serve as a suitable acceptor. To determine the location of Zn ions within the β-Ga_2O_3 lattice, we employed X-ray absorption near edge structure (XANES) in conjunction with first principles density functional theory (DFT) calculations.

The Zn concentration of thin films, named as ZGO-L and ZGO-H, were 1.7×10^{19} and 2.5×10^{20} ions/cm^3, respectively. XANES measurements of the Zn L_3-edge and Ga L_3-edge were performed on the BL2A beamline of UVSOR Okazaki, Japan, using the partial fluorescence yield method (PFY). A $Be_3Al_2Si_6O_{18}$ (beryl) double-crystal monochromator gives the Zn L_3-edge and the Ga L_3-edge in the energy regions 1000 – 1070 eV and 1100 – 1170 eV, respectively. The samples were set with their surface perpendicular to the incident X-ray beam. Fluorescence X-rays of Zn L_α and Ga L_α were collected using an energy dispersible silicon drift detector (SDD).

The XRD pattern of the Zn doped samples appear to be similar to that of the undoped Ga_2O_3 samples. However, the information obtained from the XRD measurements was insufficient to conclude the exact position of the Zn atoms in the monoclinic Ga_2O_3 lattice. Moreover, it was also not clear whether Zn can build clusters or if it can include small numbers of ZnO or $ZnGa_2O_4$.

XANES spectra with the Zn L_3-edge (1020 eV) were shown in Fig. 1 for the Zn-doped Ga_2O_3 thin film sample and a reference ZnO powder sample. The intensity of each spectrum was normalized to a value of 1 at 1050 eV after the removal of the background intensity. The spectral signal-to-noise ratios of the thin films were slightly low because of the dilute concentration of zinc, however, fine structures such as peaks *A* to *G* were detected. In terms of such identical peaks, the spectrum shape of ZGO is clearly different from that of standard sample ZnO with a wurtzite type structure. The theoretical investigation using first principles calculation indicated that Zn ions occupied at cationic positions at the tetrahedral coordinated sites [1].

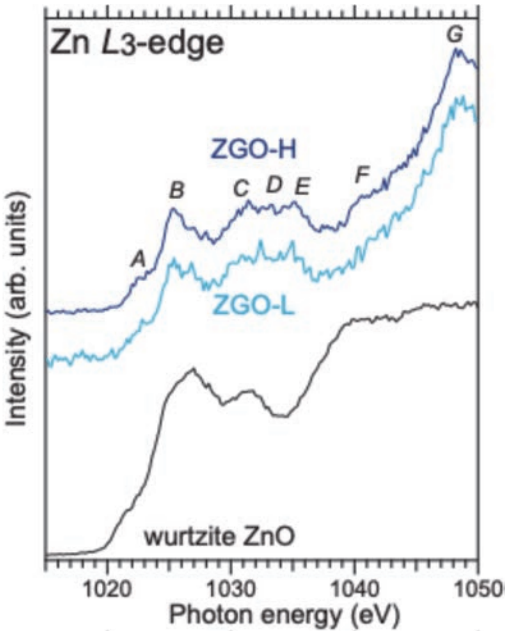

Fig. 1. Zn L_3-edge XANES spectra of the Zn-doped Ga_2O_3 thin films, together with the spectrum of wurtzite ZnO powder as a reference.

[1] S. Yoshioka *et al.*, J. Phys. Chem. C. **128** (2024) 18879.

BL2A

sXAS Study of the Fe-NiS OER Catalyst

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Iron-doped nickel sulfide (Fe–NiS) catalysts are widely studied for the oxygen evolution reaction (OER) because adding Fe into NiS significantly enhances both activity and stability compared to pure NiS. Nickel sulfides possess high electrical conductivity and accessible redox chemistry but tend to reconstruct under anodic potentials: Ni^{2+} centers are oxidized to higher-valence states, and sulfide ligands transform into oxy- or sulfate species. Introducing Fe further adjusts the Ni–S electronic environment, accelerates redox kinetics, and helps suppress corrosion by promoting in situ formation of protective iron–oxygen clusters.

Soft X-ray absorption spectroscopy (sXAS) at the Ni L_3 and S K edges is particularly well suited to tracking these reconstruction events. At the Ni L_3 edge, 2p→3d transitions reveal changes in nickel oxidation state and local coordination—indicators of active Ni^{3+} (and Ni^{4+}) species forming under OER bias. At the S K edge, 1s→3p transitions distinguish sulfide (S^{2-}), intermediate oxy-sulfur species (e.g., sulfite), and final sulfate (SO_4^{2-}) states. Comparing spectra before and after OER clearly shows how the Ni–S framework evolves: the sulfide network converts into an oxyhydroxide-like shell enriched in Ni^{3+}/Ni^{4+} and surface sulfates, which directly influence catalytic kinetics and durability.

Understanding these electronic and chemical transformations is crucial for rational catalyst design. Ni^{3+} species on the surface are recognized as the true active sites for OER, while conversion of sulfur ligands to sulfate creates a passivating layer that repels corrosive ions (e.g., Cl^- in seawater), thereby improving long-term performance. Operando sXAS thus provides a microscopic picture of how Fe–NiS catalysts dynamically self-organize into their most active and stable configurations during water oxidation. These linsights guide strategies such as optimizing Fe/Ni ratios, controlling sulfide crystallinity, and adjusting electrolyte composition to develop highly efficient and durable OER catalyst

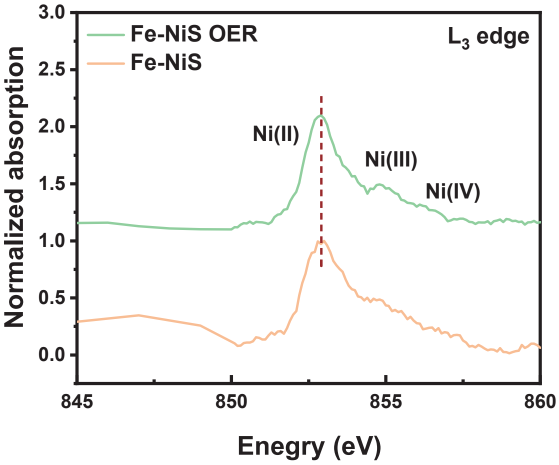

Fig. 1. Ni L-edge sXAS of Fe-NiS catalyst.

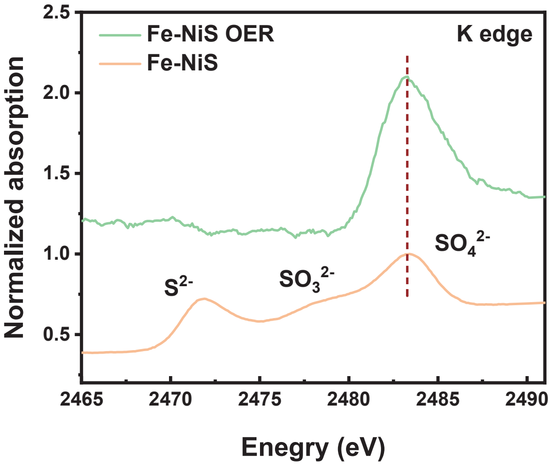

Fig. 2. S K-edge sXAS of Fe-NiS catalyst.

[1] Q. Dai *et al*., Nano Res. **17** (2024) 6820.
[2] X. Luo *et al*., Nat. Commun. **15** (2024) 8293.
[3] F. Jalilehvand, Chem. Soc. Rev. **35** (2006) 1256.

BL2A

Analysis of Electronic Structure of P3HT/ MoO_x Interface by Soft X-ray Absorption Spectroscopy

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Organic thin-film solar cells are advantageous due to their low manufacturing costs; however, their low power conversion efficiency remains a challenge. One approach to improve efficiency is the introduction of metal oxides like MoO_3 as hole extraction layers or electron extraction layers between the organic layer and the two electrodes[1,2]. Furthermore, doping metal oxides to control their valence band structures and achieve appropriate energy alignment with the HOMO and LUMO of organic semiconductor layers is expected to lead to high-efficiency organic thin-film solar cells. However, when doped metal oxides are stacked with organic semiconductors, the formation of new interfacial electronic states is considered.

This study investigates the electronic states at the interface between molybdenum oxide, known as a hole extraction layer, and the organic film using soft X-ray absorption spectroscopy. To obtain insights into the buried interface, measurements utilizing the partial fluorescence yield method in soft X-ray absorption spectroscopy are effective. However, there are very few examples of incorporating such techniques into the development of organic thin-film solar cells

The sample are thin molybdenum oxide film fabricated on silicon using the RF magnetron sputtering method. One of the films was cleaned with neutral detergent, distilled water, acetone and UV ozone. P3HT was dissolved in chlorobenzene and deposited onto the thin film by spin coating. NEXAFS spectra of the MoO_x film using both total electron yield (TEY) and partial fluorescence yield (PFY) modes were measured at the beamline 2A of the UVSOR in the Institute of Molecular Science. For TEY, the drain current from the sample was measured. For PFY, fluorescence X-rays were collected using an energy dispersible silicon drift detector (SDD). All experiments were performed at room temperature.

Figure 1 shows the NEXAFS spectrum of P3HT/ MoO_x/Si thin film in PFY mode. The observed spectrum is considered to consist of two components, Peak A and Peak B[3]. Adsorbing P3HT onto the MoOx thin film resulted in Peak A becoming more intense than Peak B. This suggests that electronic states were generated at the interface between the organic film and the oxide film. Furthermore, when P3HT was adsorbed onto a cleaned thin film, the intensity ratio changed, indicating that surface treatment of the oxide film alters the interfacial electronic states. This finding implies the possibility of controlling interfacial electronic states.

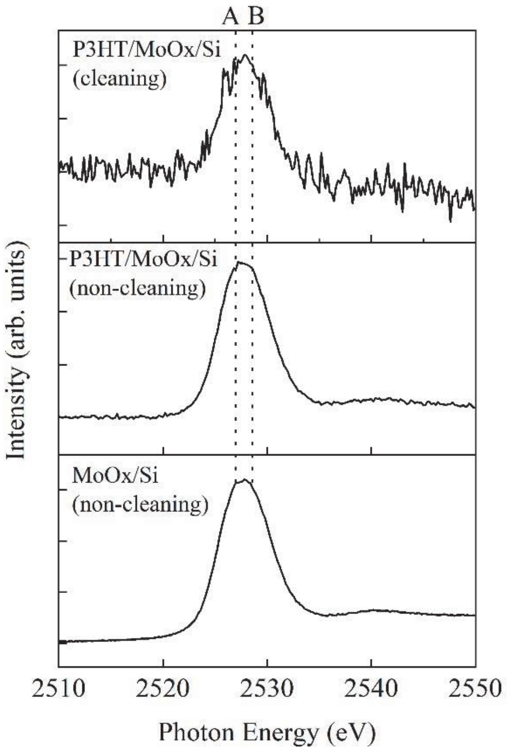

Fig. 1. Mo L-edge NEXAFS spectra of P3HT/MoO_x/Si thin film in PFY mode.

[1] D. W. Zhao *et al.*, Appl. Phys. Lett. **95** (2009) 153304.
[2] X. Hu, *et al.*, J. Phys. Chem. C **118** (2014) 9930.
[3] A. Svyazhin *et al.*, Inorg. Chem. **61** (2022) 869.