Commissioning of a Scanning Transmission X-ray Microscopy Beamline

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Construction of a scanning transmission X-ray microscopy (STXM) beamline, BL4U, was started in April 2012 as part of the UVSOR-III upgrade project [1]. This beamline covers the X-ray energy range from 100 to 700 eV. The STXM end station is used to measure near edge X-ray absorption fine structure (NEXAFS) spectra to analyze chemical states of specific elements, especially light elements, with high spatial resolution. To achieve satisfactory performance of the STXM, we need high intensity, high photon energy resolution, and high stability of the beam position as well as fine coordination of the light source, the monochromator and the STXM. We have been developing interface software and a control system for the complete beamline.

BL4U is equipped with an in-vacuum undulator and a variable included angle Monk-Gillieson mounting monochromator using a varied-line-spacing plane grating. During monochromator commissioning, the X-ray energy range from 270 to 640 eV was calibrated. Then, the 9th harmonic peak of the undulator was synchronized with the X-ray energy of the monochromator by changing the gap width from 13 to 40 mm. The energy resolution ($E/\Delta E$) of the monochromator was evaluated from the absorption spectrum of the K absorption edge of nitrogen gas. The nitrogen gas was introduced into the STXM chamber (~1 mbar) and the transmitted x-rays were measured with a photo multiplier tube. The absorption spectrum measured with exit slits of 30 x 30 µm is shown in Fig. 1. From this spectrum, the resolving power was estimated to be ~6,000. On the other hand, the intensity of X-rays around the carbon K absorption edge from 283 to 300 eV, was very low because of carbon contamination on the mirrors. We expect this problem will be solved by cleaning the mirror chambers and recoating the mirrors.

As a test sample, printer toner particles were studied at the oxygen K edge. Toner particles were embedded in resin and a thin specimen was prepared with an ultra-microtome. A stack of 78 X-ray transmission images was acquired from 522 to 564 eV. The exit slit was set to 30 x 30 µm and the dwell time was 3 msec for each pixel. A spectrum of each component of the specimen, matrix and resin, is extracted from the each specific part of the image stack. By fitting these spectral data of the matrix, the resin and the wax, to the image stack, chemical distributions of the components are clearly distinguished as shown in Fig. 2. These analyses are performed by using aXis2000 [2].

The commissioning of the beamline is almost finished. The STXM system is now ready for user operation. Currently, different kinds of measurement methods and sample cells for in-situ observation are under development for various upcoming applications.

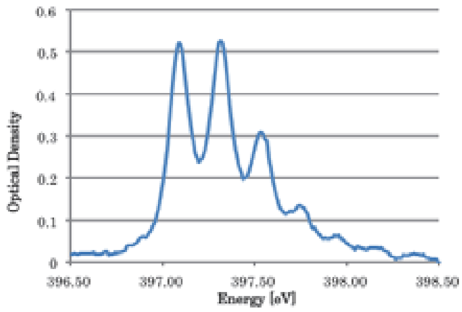

Fig. 1. 1s-π* absorption spectrum of nitrogen gas.

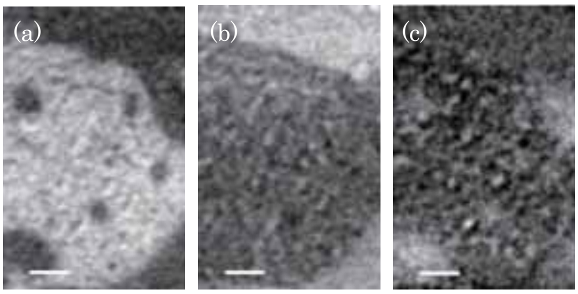

Fig. 2. Chemical maps of three components in a printer tonner sample, (a) matrix, (b) resin and (c) wax. Higher concentrations of each component are brighter. Scale bars are 1 µm.

[1] T. Ohigashi, T. Araki, E. Nakamura, N. Kondo, E. Shigemasa, N. Kosugi and M. Kato, UVSOR Activity Report **39** (2012) 42.
[2] http://unicorn.mcmaster.ca/aXis2000.html

Reflectance Measurements of Black Coatings for Stray Light Rejection Used in Bepi Colombo Mission

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We study the tenuous atmosphere of Mercury through the spectroscopic observations. To date, some species (e.g., H, He, O, Na, Mg, K and Ca) have been identified in the Mercury's atmosphere. As these species have many resonance scattering lines in the extreme ultraviolet (EUV) region, spectroscopic observations will enable us deduce the source process, current environment, and evolution of the Mercury's atmosphere.

We are now developing an ultraviolet spectrometer; PHEBUS (Probing of Hermean Exosphere by Ultraviolet Spectroscopy) which is a part of the BepiColombo mission. From the orbit around Mercury, PHEBUS will observe the atmosphere in the EUV and FUV (far ultraviolet) region. In this mission, the signals from the targets are so faint that the stray light must be kept as low as possible. The black coatings techniques are commonly used in various space telescopes and optical instruments in order to reject the stray light in the instrument.

In this experiment, we measure the reflectivities of two types of black coatings in EUV range. As these black coatings have low reflectivities (less than 1 %), the intense light is needed for the accurate measurement.

We install Indium filters on the entrance of the beam line in order to eliminate the multi-order lines. At first, we investigate the purity of the 83.4 nm line through indium filters. We judge the purity from consistency between the wavelength characteristics of indium filters for continuous lines at UVSOR and those for the particular lines at the EUV facilities of Institute of Space and Astronautical Science (ISAS). The latter is measured for the emission lines of the oxygen gas with the discharged light source. Comparing the transmittances of indium filter measured at UVSOR and at ISAS, we confirm that the pure 83.4 nm light is achieved at UVSOR with the entrance filter.

With the pure 83.4 nm line, we measure the reflectivities of two black coating samples at various incident angles (30, 40, 50, 60, 70 and 80 degrees). One is fabricated by EBINA Co. (hereafter referred to as sample #1) and another is by ACTAR (hereafter referred to as sample #2). These black coatings will be used inside the spectrometer. Sample #1 is also used in the EXCEED spectrometer on board the JAXA's SPRINT-A mission.

The results are shown in Fig. 2 and both samples show low reflectivity. Especially, Sample #1 has lower reflectivity than sample #2 at every incident angles.

As the next step, we plan to measure the reflectivities of these black coatings at various wavelengths.

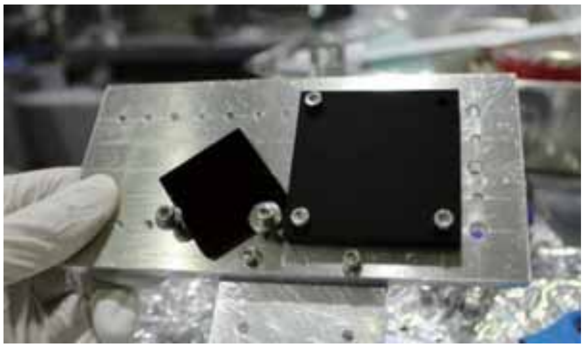

Fig. 1. Photo of the black coating samples. Left is fabricated by EBINA and right is by ACTOR.

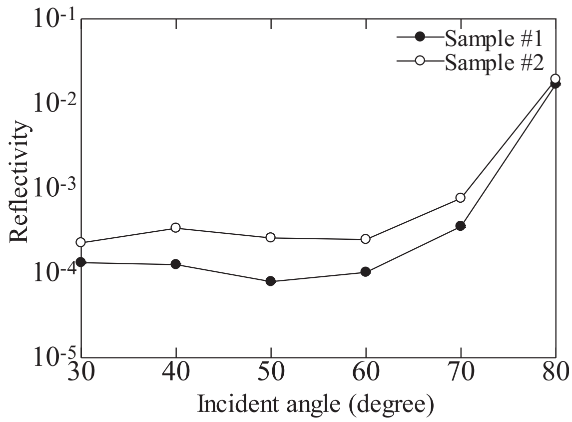

Fig. 2. The reflectivities of the black coating samples at 83.4 nm. Both samples show low reflectivities.

Performance Test of Infrared Microspectroscopy Using Synchrotron Radiation at UVSOR-III

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Infrared (IR) synchrotron radiation (SR) has the advantage of higher brilliance and intensity than those of thermal light sources such as the globar (a SiC rod heated to ~1500 K) and the mercury lamp (a discharge lamp with mercury vapor), which utilize the black-body radiation from a heated object [1]. IR microspectroscopy (IR-MS) is currently one of the most common analysis methods for various materials in many fields of science and industry. Commercial IR microspectroscopy instruments, designed for mid-IR spectral range with a thermal IR source, are generally available. However, because of the low brilliance of the thermal light sources, the spatial resolution cannot be the same as the diffraction limit (~ wavelength). With the use of IR-SR, one can further improve the spatial resolution of IR microspectroscopy. Then, we tested that a commercial IR microspectroscopy instrument can be equipped to the IR-SR beamline, BL6B [2] and obtained the performance combined with IR-SR.

A photograph of the IR-MS instrument (FTIR6100+IRT7000, JASCO Corp.) installed at BL6B is shown in Fig. 1. IR-SR was introduced to IR-MS by two plane mirrors through the atmosphere from the free port at the downstream of a CVD-diamond window, which separates a low vacuum area of the Fourier-transform IR interferometer (FTIR) from an ultra-high vacuum area of the front-end part of BL6B.

Fig. 1. Layout of the IR microspectroscopy (IR-MS) apparatus installed at BL6B. The infrared synchrotron radiation (IR-SR) is introduced to the IR-MS by two plane mirrors through the atmosphere.

To demonstrate the performance of IR-SR, IR images of a U. S. Air Force (USAF) test target measured with IR-SR and an internal thermal (globar) source are shown in Fig. 2. The test target, on which a metal pattern is formed on a quartz substrate, was used for the evaluation of spatial resolution. The test target was measured on the focal plane of the IR microscope. By using IR-SR, a clear spatial image was obtained with the instrumental spatial resolution of 5×5 μm^2 in contrast to that of a thermal source, even though the optics in the IR-MS is aligned to the internal source. This implies that the IR-SR is suitable to IR-MS.

The IR-MS instrument will be installed permanently in FY2013.

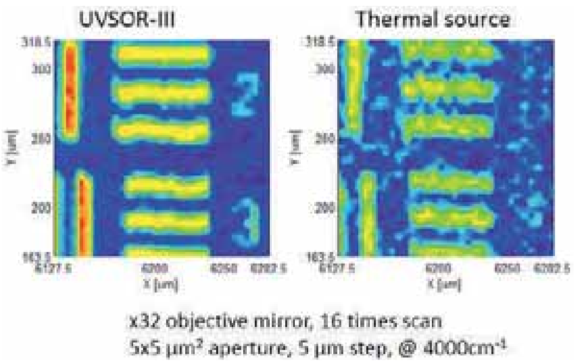

Fig. 2. Obtained reflectance image of USAF test target at the wavenumber of 4000 cm^{-1} using IR-SRfrom UVSOR-III compared with an internal globarlamp (thermal light source). The objective of the IRmicroscope had a magnification of 32×. The instrumental spatial resolution was set as 5×5 μm^2.

[1] S. Kimura and H. Okamura, J. Phys. Soc. Jpn. **82** (2013) 021004.
[2] S. Kimura, E. Nakamura, T. Nishi, Y. Sakurai, K. Hayashi, J. Yamazaki and M. Katoh, Infrared Phys. Tech. **49** (2006) 147.

BL7B

Development of the Cold Mirror Coating (Reflective Narrow Band Filter) across 300 mm Diameter for Lyman-Alpha Line (121.6 nm)

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Our team consisting of Japan, US, Spain, France, and Norway is developing a Chromospheric Lyman-Alpha SpectroPolarimeter (CLASP), which is proposed to fly with a NASA sounding rocket in 2015 [1]. CLASP will explore the magnetic fields in the solar atmosphere (the upper chromosphere and transition region) via the Hanle effect in the Lyman-alpha (Lyα) line (121.6 nm) for the first time. This experiment requires precise spectropolarimetric observations with a polarimetric sensitivity of < 0.1% and a wavelength resolution of 0.01 nm.

In order to achieve the polarimetric sensitivity of < 0.1%, the rejection of visible light (VL) from the instrument is indispensable, since the total intensity in the solar VL wavelength range is about 2×10^5 times larger than solar Lyα line. For the VL rejection, we plan to perform the multi-layer "cold mirror coating" on the 300 mm diameter primary mirror of the CLASP. This coating is a reflective narrow band filter with the reflectivity of more than 50 % in the Lyα line, and less than 5 % for VL in average.

Though this coating itself is difficult to adjust the peak wavelength and to enhance the reflectivity, the uniformity of the coating performance across the 300 mm diameter area is also required for the CLASP. If there is a large amount of non-uniformity in the reflectivity, this causes the spurious polarization. So, we performed the development of the uniform coating with Acton Optics and Coatings.

Figure 1 shows the location of the 12 witness samples deployed in the 300 mm diameter area during the coating process. Figure 2 is their reflectivity as a function of wavelength near the Lyα line at AOI = 2 degree measured with UVSOR BL7B. The spatial distributions of the reflectivity at the Lyα line and the peak wavelength are summarized in the left and right panels of Fig. 1, respectively. As a result, the reflectivity of the developed coating is distributed among 60%±2%.

Based on these results, we estimate how much this coating generates the spurious polarization. Figure 3 shows the differences in the pupil maps of the Stokes signals for +Q input between the ideal (uniform) and real coating cases. With these maps, we confirmed that the spurious polarization by this coating is ~ 0.001 %, which is 2 orders smaller than the required polarimetric sensitivity of < 0.1%. Hence, we conclude that the development of the uniform enough coating for the CLASP is successfully completed.

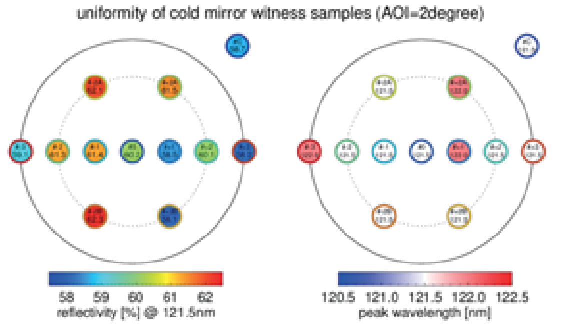

Fig. 1. Location of witness samples during the coating process and the uniformity of the coating performance.

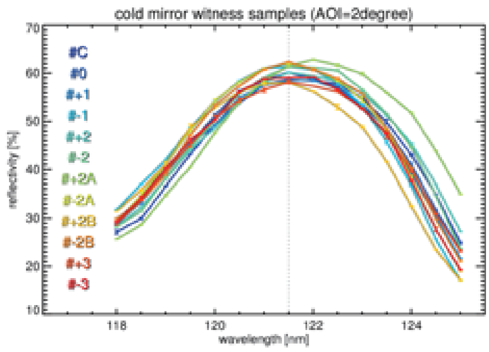

Fig. 2. Measured reflectivity of 12 witness samples as a function of wavelength near the Lyα line.

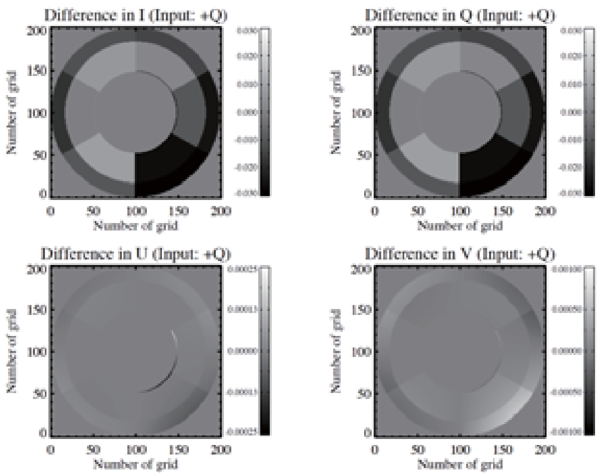

Fig. 3. Difference in Stokes signal pupil map for +Q input between the case for the uniform coating and that for the non-uniform coating.

[1] R. Kano *et al.*, SPIE **8443** (2012) 84434F.

BL7B

Development of the Multi-Layer Coating for the High-Efficiency Reflective Polarizer in Lyman-Alpha Line (121.6 nm)

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Our team consisting of Japan, US, Spain, France, and Norway is developing a Chromospheric Lyman-Alpha SpectroPolarimeter (CLASP), which is proposed to fly with a NASA sounding rocket in 2015 [1]. CLASP will explore the magnetic fields in the solar atmosphere (the upper chromosphere and transition region) via the Hanle effect in the Lyman-alpha (Lyα) line (121.6 nm) for the first time. This experiment requires precise spectropolarimetric observations with a polarimetric sensitivity of < 0.1% and a wavelength resolution of 0.01 nm.

In order to achieve the polarimetric sensitivity of < 0.1%, CLASP requires the high throughput as much as possible for suppressing the photon noise. For this purpose, we plan to apply the high-efficiency reflective polarizer consisting of the Fused Silica substrate and the multi-layer coating of SiO_2 and MgF_2 proposed by Bridou *et al.* [2] as the polarization analyzer of CLASP. This high-efficiency reflective polarizer is theoretically predicted to have a reflectivity of 58.4 % for s-polarized beam, which is about 2.65 times higher than MgF_2 plate of 22 % (This MgF_2 reflectivity was measured at UVSOR BL7B in 2009 [3].).

For the development of the high-efficiency reflective polarizer, we fabricated three polarizers and six witness samples with the multi-layer coating of SiO_2 and MgF_2 performed by Acton Optics and Coatings, and we evaluated their performance with the witness samples at UVSOR BL7B using our measurement system shown in Fig. 1. Our system consists of the beam splitter, beam cleaner and goniometer. The beam splitter made of MgF_2 splits the synchrotron beam into two beams for the measurements and for monitoring the intensity variation of BL7B. This BL7B monitor makes the effect of the intensity fluctuation of the synchrotron beam on the measurements significantly small. The beam cleaner mounted at the Brewster's angle eliminates the p-polarized beam from the beam of BL7B. Then, 100% linear polarized beam can be used for the measurements with the goniometer.

The top and bottom panels in Fig. 2 are the measured reflectivity of 6 witness samples of the high-efficiency reflective polarizer for s- and p-polarized beam, respectively. We note that when we measure R_p and R_s, the goniometer is mounted at the configurations shown in Fig. 1 and rotated 90 degrees from Fig. 1, respectively. Based on our measurements, the polarizers have uniform performance of R_s = 54.7 % and R_p = 0.3 % at their Brewster's angle of 68 degree, which is close to the theoretical value in Bridou *et al.* [2]. We conclude that we have successfully developed the high performance reflective polarizer which has high efficiency and high polarization power ($\equiv$ $(R_s - R_p) / (R_s + R_p)$) of 98.9 %.

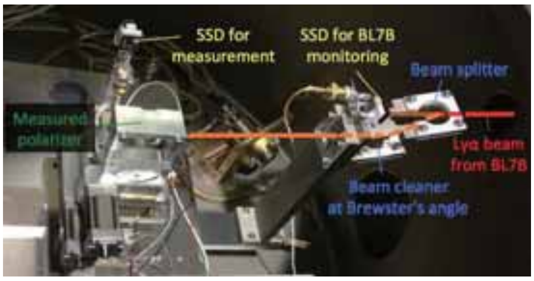

Fig. 1. Our measurement system.

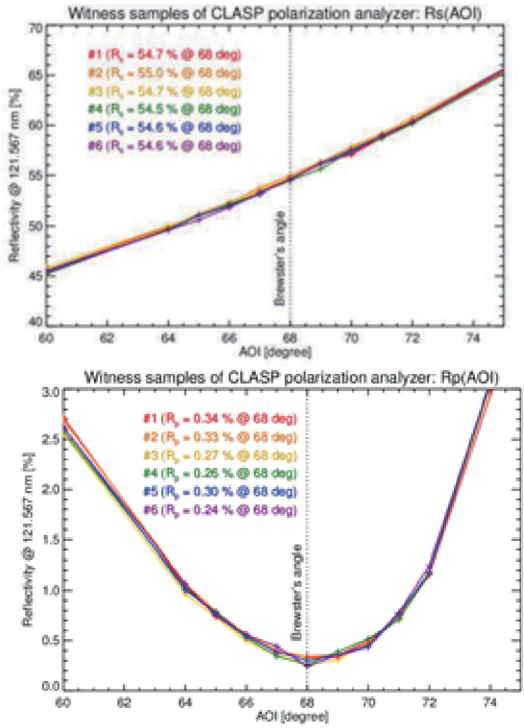

Fig. 2. Measured reflectivity of witness samples for s- (top panel) and p- (bottom panel) polarized beams.

[1] R. Kano *et al.*, SPIE **8443** (2012) 84434F.

[2] F. Bridou *et al.*, Applied Physics A **102** (2011) 641.

[3] H. Watanabe *et al.*, UVSOR Activity Report **37** (2009) 51.