Development of a Compact Scanning Transmission X-Ray Microscope

Y Takeichi¹, N Inami¹, H Suga², T Ueno¹, S Kishimoto¹, Y Takahashi² and K Ono¹

¹ Photon Factory, Institute of Materials Structure Science, High Energy Accelerator Research Organization, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan
² Department of Earth and Planetary Systems Science, Hiroshima University, 1-3-1 Kagamiyama, Higashi-Hiroshima, Hiroshima 739-8526, Japan
³ National Institute for Material Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

E-mail: yasuo.takeichi@kek.jp

Abstract. We report a compact scanning transmission X-ray microscope newly designed and developed at the Photon Factory. The microscope has very compact size and is equipped with fully digitized control electronics to realize high stability, precise positioning and fast data acquisition. The hardware design of the microscope is described in detail. Results of measurement using test samples are also presented.

1. Introduction
Recent developments in microscopic measurement techniques using focused X-rays from a synchrotron radiation source opened a wide field of research in physics, chemistry, biology and environmental science. These methods, which are often referred to as X-ray spectromicroscopy or microspectroscopy, enable elemental composition or chemical state mapping, and the local X-ray absorption spectroscopy (XAS) of materials. Scanning transmission X-ray microscopy (STXM) [1] using soft X-rays is a powerful tool with features of high spatial resolution down to nanometer scale, high sensitivity to chemical properties, and relatively small radiation damage to the sample. It also allows a wide variety of advanced measurement techniques such as magnetic-domain imaging [2], time-resolved experiments [3], and luminescence detection [4].

In this report, we describe our STXM installed at the soft X-ray undulator beamline of the Photon Factory, Japan. The STXM is a newly designed microscope with very compact size that (i) improves the vibration behavior by increasing the resonance frequency of the microscope components, (ii) realizes a fast response of the stages driven by piezo motors, and (iii) can be used with a multi-purpose beamline that is already in operation.

2. Design of the compact STXM
Figure 1 shows the optics of the present STXM. The undulator radiation is first monochromatized and focused on the four-way aperture slit. A Fresnel zone plate (FZP) with outermost zone width of 30 nm is placed at 1 m distant from the slit, and is designed to have focal distance of 0.7–5 mm. An order sorting aperture (OSA) is placed between the FZP and the sample to stop the zeroth and higher order diffraction. A sample image is obtained by scanning the sample position and detecting the transmitted X-rays. All components from the aperture slit to the detector are mounted on a single optical table to
reduce the relative motion of the optics by vibration.

Figure 2 shows the scheme of the STXM components, which is in principle the same as the scheme of the common design [1]. In designing the components, computational simulation of the vibration behavior employing the finite element method was performed to keep the resonant frequency higher than 200 Hz. Using piezo-motor-driven linear stages (Attocube ECS series) for all of the coarse stages, the whole system shown in figure 2 is contained in a small vacuum chamber with interior dimensions of 220×310×200 mm³. The ECS stages are capable of positioning with nanometer precision by means of integrated optical encoders. The coarse stages thus enable fast and precise positioning in the optics adjustment and rough imaging of the sample. The sample position is monitored with laser interferometric position sensors (Attocube FPS3010) with sensitivity of 25 pm. The position values are read out at a rate of 52 kHz for feedback positioning by the sample scanner and on-the-fly measurement.

To realize transmitted X-ray detection with good linearity in a wide range of intensity, fast pulse counting using a silicon avalanche photodiode (APD) [5] is implemented. The APD is capable of pulse counting up to 10⁷ cps, which corresponds to the maximum intensity of focused X-rays expected at the sample. The APD is also suitable for our system because it is much smaller and lighter than the scintillator–photomultiplier assembly commonly used in a commercial STXM.

The pulse counting function, scanner drive and position read-out from the interferometric sensor are all integrated in a field-programmable-gated-array (FPGA) circuit. The FPGA implementation and control program of the STXM are developed on a National Instruments LabVIEW platform.
3. Experiment

3.1. Microscopic image

The experiment was performed at BL-13A and BL-16A of the Photon Factory. Energy resolution was set to $E/\Delta E \sim 5000$. Figure 3(a) shows an oblique-view scanning electron microscopy (SEM) image of a test sample fabricated with a focused ion beam (FIB). The logos are 200 nm deep and patterned on $\approx 300$-nm-thick W film deposited on SiN membrane. Figure 3(b) shows the STXM image taken with photon energy of 350 eV. Figure 3(c) is an enlarged image of the area indicated by the dashed square in figure 3(b). A structure with width of 100 nm was resolved. The spatial resolution of the microscope is still being improved by optimizing the focusing optics and vibration isolation according to the circumstances. Using the present FZP, Rayleigh resolution up to 30 nm is fairly possible. To further improve spatial resolution, we also plan to introduce FZPs with a more precise outermost zone width in the future.

At BL-16A, we examined the stability of the image while changing the polarization mode of the undulator. By taking images of a PtIr standard sample (not shown here), the lateral image shift by resulting from changing the helicity of the circular polarized radiation was estimated to be 80 nm in the horizontal direction, which is acceptable for magnetic-domain imaging.

3.2. Microspectroscopy

To perform microspectroscopy with our STXM, the Na $K$ edge XAS spectrum of NaCl was measured. A solution of NaCl was dropped onto SiN membrane and then air-dried to obtain plate crystals of various sizes. Figure 4(a) and (b) show the oblique-view SEM image and STXM image of the measured sample. Absorption spectra of the area indicated in figure 4(b) were obtained in a line-profile manner; the sample position was scanned at each point of the photon energy. Figure 4(c) shows the spectrum obtained from the region on (solid line) and away from the NaCl solid (dashed line). Using the out-of-the-sample spectrum as a reference ($I_0$), the XAS spectrum of NaCl was obtained as shown in figure 4(d). The spectrum is identical to the XAS spectrum at room temperature reported in the literature [6].

4. Concluding remarks

We designed and developed a compact STXM using piezo-motor-driven linear stages. Structures of 100 nm were shown to be resolved. We also obtained local XAS spectra of NaCl plate crystal. At the beamlines used here, the STXM covers a photon energy range of 250–1500 eV, i.e., from the C $K$
Figure 4. (a) Oblique view SEM image of NaCl dried on SiN membrane. (b) STXM image of the dried NaCl. (c) Transmitted X-ray intensity measured on and off the NaCl sample at the designated area in (b). (d) Na K edge XAS spectrum obtained from (c).

edge to the $M$ edges of rare earths. Our STXM will be used for the magnetic domain imaging and nanometer-scale chemical characterization of rare-earth permanent magnets using circular polarized radiation [2] and the chemical mapping of materials in environmental sciences [7].

5. Acknowledgements
This work is partly supported by the Japan Science and Technology Agency (JST) under Collaborative Research Based on Industrial Demand "High Performance Magnets: Towards Innovative Development of Next Generation Magnets". The experiment was performed with the approval of the Photon Factory Program Advisory Committee (Proposal No. 2013S2-003). YT thanks J. Raabe and B. Watts for fruitful discussion. C. David is also acknowledged for providing the Fresnel zone plate.

References
[1] Kilcoyne A L D, Tyliszczak T, Steele W F, Fakra S, Hitchcock P, Franck K, Anderson E, Harteneck B, Rightor E G, Mitchell G E, Hitchcock A P, Yang L, Warwick T and Ade H 2003 J. Synchrotron Rad. **10** 125
[2] Ono K, Araki T, Yano M, Miyamoto N, Shoji T, Kato A, Manabe A, Nozaki H, Kaneko Y and Raabe J 2011 IEEE Trans. Magn. **47** 2672
[3] Raabe J, Quitmann C, Back C H, Nolting F, Johnson S and Buehler C 2005 Phys. Rev. Lett. **94** 217204
[4] Vaz C A F, Moutafis C, Quitmann C and Raabe J 2012 Appl. Phys. Lett. **101** 083114
[5] Baron A Q R, Kishimoto S, Morse J and Rigal J M 2006 J. Synchrotron Rad. **13** 131
[6] Murata T, Matsukawa T and Naoe S 1988 Solid State Commun. **66** 787
[7] Kikuchi S, Makita H, Mitsunobu S, Terada Y, Yamaguchi N, Takai K and Takahashi Y 2011 Chem. Lett. **40** 680