Fast X-Ray Fluorescence Camera Combined with Wide Band Pass Monochromatic Synchrotron Beam

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Abstract. A double W/B$_4$C multilayer monochromator (2d=50.4Å) was commissioned for non-scanning X-ray fluorescence (XRF) imaging experiments. The combination of a brilliant multi-pole wiggler source and the present wide band pass monochromator permits 1.2×10^{13} photons/sec at the sample position for 8.04 keV X-rays. Energy resolution ΔE and ΔE/E are 300~500 eV and ~5%, respectively. The exit beam height is constant for X-ray energy ranging from 5.5 to 13.0 keV. Indirect cooling of the 1st multilayer works successfully. In addition, a new fast CCD camera was developed for quick readout and transfer of the image data. It was found that the typical exposure time for one XRF image with 1000 × 1000 pixels is 0.03~1 sec. This permits *in-situ* movie recording for the distribution of elements.

INTRODUCTION

A recently developed non-scanning X-ray fluorescence (XRF) microscope is a new procedure for chemical composition imaging [1-3]. Unlike conventional scanning microscopes, it does not require a microbeam. In contrast, it uses quite a wide beam (typically 8mm (H) × 0.2mm(V)), which illuminates the whole sample surface in a low-angle-incidence arrangement (0.5~1.5 deg). Another difference is the detector system. The present microscope employs a CCD camera equipped with a collimator inside, and the distance between the sample surface and the detector is set extremely close, in order to enhance both spatial resolution and detection efficiency. The advantage of the present non-scanning method is its potential capability to perform quick imaging, typically 0.03~1 sec for 1 image with 1000×1000 pixels [4,5].

One of the most important requirements for this imaging is extremely high flux photons with uniform intensity distribution and a wide linear shape. In the present research, the use of a wide band pass monochromator was examined in order to increase the photon flux effectively with only a slight sacrifice of energy resolution. This report describes the instrumental details and the performance of the mutilayer monochromator commissioned at the multi-pole wiggler (MPW) beamline, BL-16A1 at the Photon Factory, KEK, Tsukuba, Japan.

INSTRUMENTATION

Figure 1 schematically shows the beamline layout. A single flat mirror (Rh coated, 4.5 mrad fixed incidence) in the optics hutch was used for removing high-energy X-rays from the multi-pole wiggler X-ray source. A W/B$_4$C double multilayer monochromator (2d=50.4Å, 125 layer pairs, Si wafer substrate, 20mm×50mm×5mm, Osmic Co., Ltd.) was installed in the experimental hatch (~35.5m from the source). Although the issue in the initial stages was the method to be used for cooling the 1st multilayer, it has since been found that simple indirect water-cooling (23 ℃) works well in practice. This is mainly because of the shallow incidence geometry, which can release the heat load. Liquid InGa alloy was employed as a contact material between the Si substrate and a copper plate. The entire optics of the multilayer monochromator are in a vacuum tank at a pressure of less than 50Pa. An oil-free scroll pump was used to avoid possible carbon contamination of the multilayer surface. Simple electronics for the interlock were introduced to secure the monochromator from sudden unexpected water and vacuum problems.

A Si PIN detector (XR-100T, Amptek, energy resolution ~200 eV at 5.9keV) was employed for calibrating the incidence angle for the 1st multilayer by ascertaining the photon energy of air scattering from the X-ray path. The 2nd multilayer was adjusted so that the exit beam is always at the same height (6 mm distant from the direct beam) for X-ray energy ranging from 5.5 to 13.0 keV. The typical beam size is 8~12 mm (H) × 0.2 mm (V).

The details of the non-scanning XRF microscope have been described elsewhere [5]. During the present commissioning, a new fast CCD camera based on the TC281 (Texas Instruments) was developed because the speed of the detector can be another limitation when sufficient photon flux is available. As summarized in Table 1, it has 1000×2000 pixels so that one half can be used as a one-time storage area to which the data are transferred from the other half, the exposure area. It is possible to repeat the exposure at a speed of 30 frames/sec. In the previous study, we used a Hamamatsu C4880 system with a TC215 chip [4,5]. It was necessary to close the shutter when reading CCD pixels, and also the data transfer was slow. The present system is clearly very fast. One can note several advancements in the detector technologies. The power source for Peltier cooling is prepared as an independent box, but all other functions are stored in 5 boards inside the camera. There is no need for a so-called controller any more. In addition, no water is required for cooling the Peltier device.

TABLE 1. Main specifications of the CCD detector for XRF imaging

	Conventional camera	Present new camera
CCD chip	TC215	TC281
Pixels	1000×1018	$1000 \times 1000 \times 2$
Size of pixel	12×12 μm^2	8×8 μm^2
Effective area	12×12 mm^2	8×8 mm^2
Peltier cooling	-30 ºC	-30 ºC
	Water cooling	Air cooling
A/D conv.	10 bit / 14 bit	12 bit
Controller	Outside	Stored
Shutter	Required	Not required
Trans. rate	0.25/ 4 fr/sec	30 fr/sec
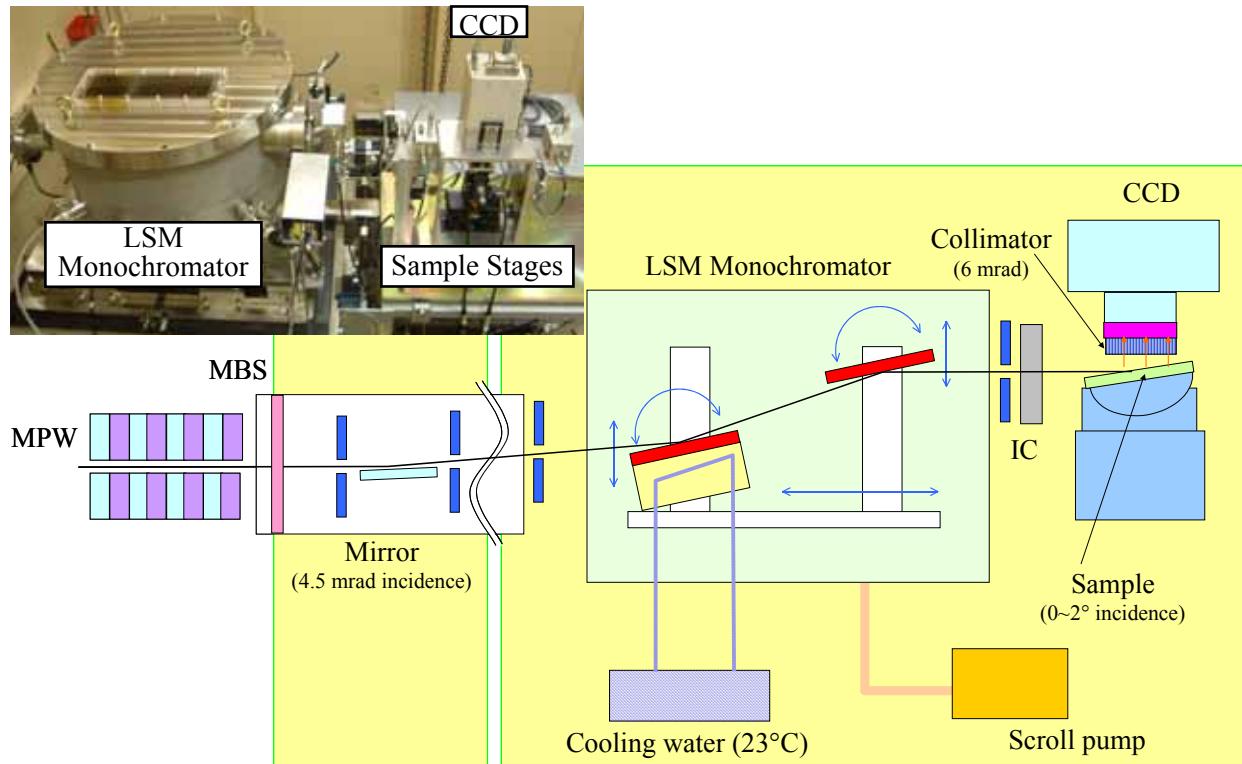
FIGURE 1. Schematic view of beamline optics for non-scanning XRF microscope

10

Intensity [×10³Cts]

Air Scatter
(10 keV)

FWHM
533 eV

Ar

5

0

5   10

Energy[keV]
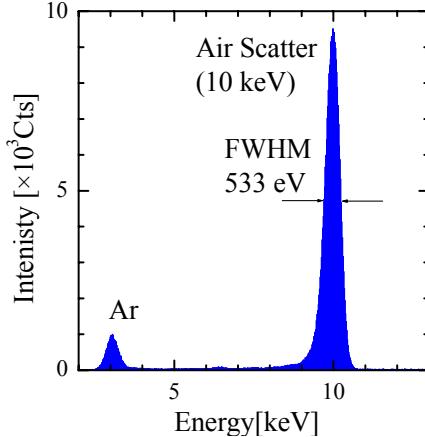
FIGURE 2. Typical X-ray energy spectra for the primary beam measured by Si PIN detector

TABLE 2. Bandwidth of the photon energy monochromatized by multilayers

E [eV]	ΔE [eV]	$\Delta E/E$ [%]
10000	490	4.9
8000	400	5.0
7200	350	4.9
6000	330	5.5

PERFORMANCE

A Si PIN detector placed near the exit window of the vacuum tank for multilayers evaluated the energy resolution of the monochromatized beam. As shown in Figure 2 and Table 2, the FWHM observed was wider than the intrinsic resolution of the detector. The estimated ΔE and $\Delta E/E$ are 300~500 eV and ~5%, respectively. The flux intensity was evaluated by a calibrated ionization chamber under different attenuation conditions and also with different bias voltages. It was found that the flux is 1.2×10^{13} photons/sec for 8.04 keV X-rays at the sample position (8mm(H) × 0.2mm(V)). This is approximately 2 decades larger than that of a normal monochromatic beam at a bending magnet beamline, where our previous experiments have been performed.

Figure 3 shows XRF images of a test pattern (metallic chromium evaporated on the glass substrate) and a piece of printed circuit board. In this case, the images were taken by a fast camera system, and the exposure time was very short, 1 sec and 0.2 sec, respectively. Since the incident energy was 10 keV (above Cu K-edge), one can see that the copper wire looks bright. The quality of the image is quite comparable with our previous data collected by a conventional slow camera system. It was found that optimizing the internal shape of the window plate and the collimator is very important, because the circuit for reading out the CCD data is extremely sensitive and easily degrades the image data, especially when a higher amplification gain is chosen.

The present instruments can be applied to *in-situ* observation of the electrochemical deposition of metals from an aqueous solution. In this experiment, electrolysis was performed using a compact cell with a copper ring anode (i.d. 16mm) and a copper wire cathode (0.7mm dia.) in the center of the ring. An electrolyte consisting of an aqueous solution of 360 mM zinc sulfate was put inside the ring and sandwiched by acrylic resin (bottom) and by a thin Mylar film (6μm thick, on the irradiation side) to form an apprroximately 200 μm thin solution layer. A DC voltage of 2.5 V was applied between the electrodes. XRF movie recording commenced just after applying the voltage. The exposure time was only 0.1 sec for one image, and 600 images were collected to observe the full growing process. Further details will be published elsewhere [6].
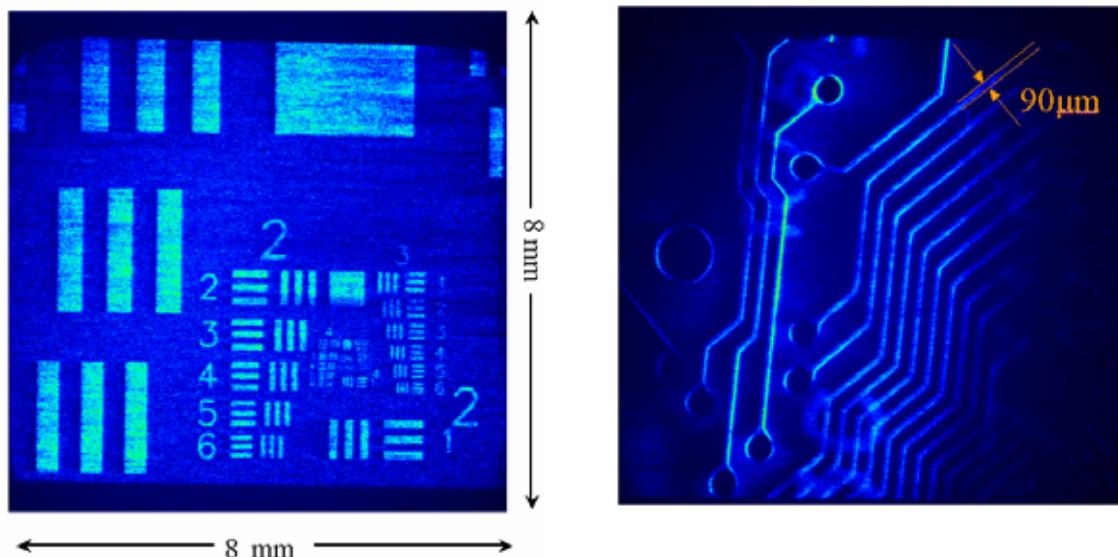
FIGURE 3. XRF images of test pattern (left) and printed circuit board (right). 10 keV excitation.

ACKNOWLEDGMENTS

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