The SR nanobeam analysis center for green/nanotechnologies at BL37XU and BL39XU

The recent progress in X-ray focusing mirrors has achieved a 7 nm X-ray beam [1], showing a path towards X-ray analyses at a sub-10 nm resolution. On the way to the ultimate nanobeam applications, to achieve practical uses of a ~100 nm beam is a current issue to meet requirements in the wide fields of scientific and industrial research studies. Under the circumstances, we upgraded two hard X-ray beamlines, BL37XU (Trace Element Analysis) and BL39XU (Magnetic Materials) for nanobeam applications. At the new experimental stations constructed, an X-ray nanoprobe of 100×100 nm^2 size is provided, and X-ray absorption fine structure (XAFS) measurement, X-ray magnetic circular dichroism (XMCD) spectroscopy, and X-ray fluorescence (XRF) analysis at the ~100 nm spatial resolution are routinely available. These nanoscale spectroscopic analyses would meet the strong demands for structural, chemical, electronic, and magnetic characterizations of novel functional materials with the nanostructure, used in fuel cells, rechargeable batteries, nonvolatile data storage devices, solar cells, and biomaterials, which are key devices of green/nanotechnologies.

For the practical use of the X-ray nanoprobe, the position stability and available photon flux of the focused beam were the primary requirements. To achieve this goal, we made a number of technical developments in the X-ray optics and experimental environments [2]: i) dedicated experimental hutches with a precise temperature control system, ii) Si double-crystal monochromators using a liquid-nitrogen cryogenic cooling system with an improved X-ray beam stability, and iii) X-ray focusing mirrors in the Kirkpatrick and Baez (KB) geometry with a large aperture (see Fig. 1).

The BL37XU and BL39XU beamlines were extended to 76 and 74 m from the source, by the outside wall of the experimental hall, where the dedicated experimental hutches were built. X-ray focusing mirrors were placed in the hutches, and the long distance from the source to the mirrors allowed a small focused beam size with a long working distance. The hutches are equipped with thermal shielding walls with heaters and an air-conditioning system for a precise control of the temperature within ±0.02°C a day.

We achieved a marked improvement in the stability of the monochromatic X-ray beam as well as of the focused nanobeam by adopting the newly developed Si double-crystal monochromators with a cryogenic cooling system. A liquid-nitrogen-cycling refrigerator was optimized in terms of several operation parameters such as the rotation frequency and gas pressure so that the vibration of the Si crystals was minimized. We developed a low-vibration tubing for the liquid nitrogen coolant, effective thermal shields between the Si crystals and the positioning stages, and a high-precision temperature control system, and so on. These means have significantly reduced the fluctuation in the intensity of the monochromatic X-ray beam to 1/10 or less than that for the existing cryogenic monochromators.

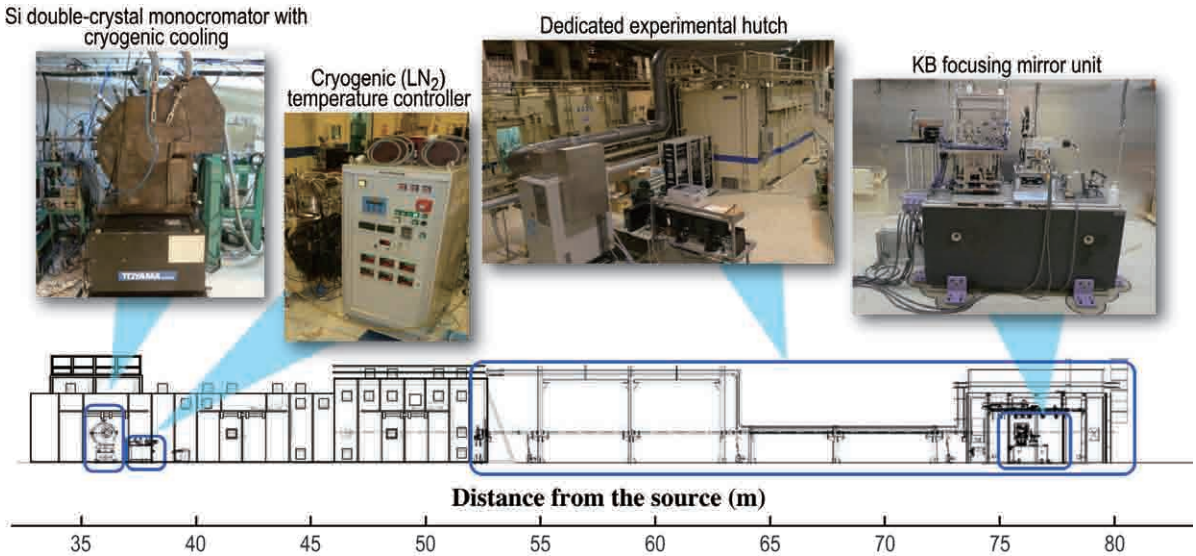

Fig. 1. Upgraded components for the X-ray nanoprobe end station at BL39XU.

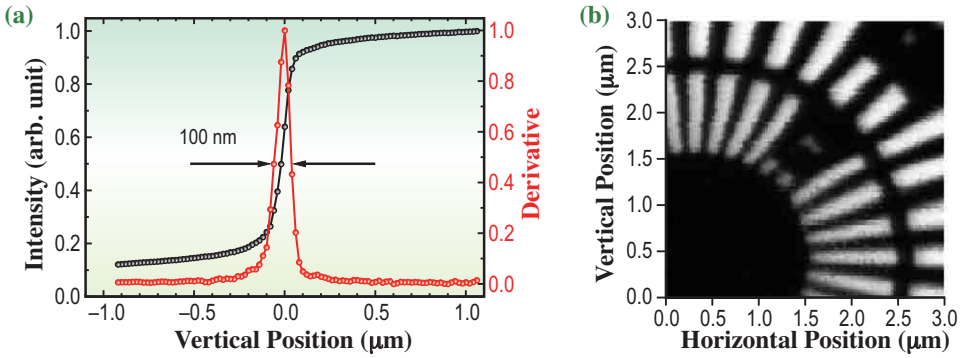

Fig. 2. **(a)** Focused beam profiles in the vertical direction. **(b)** Scanning image of a Ta test chart having the patterns of a 100 nm line and space. The measurement conditions were 100×100 pixels with a 30 nm/pixel step and the acquisition time of 0.1/s pixel.

The KB focusing optics are consists of two elliptical mirrors coated with Rh. The mirrors were fabricated by an elastic emission machining method developed by Osaka University and RIKEN [3]. With the large aperture of 0.76(H)×1.2(V) mm^2, the mirrors can accept almost half of the incoming X-ray flux and focus it into a bright spot on the sample. The mirror units and the sample stages were arranged together on a robust base block made of granite for the isolation of the system from floor vibrations.

Figure 2(a) shows the profiles of a focused X-ray beam in the vertical direction, obtained using the knife-edge method with a gold wire at an X-ray energy of 12.4 keV at BL37XU [2]. The focused beam size of 100(H)×100(V) nm^2 (FWHM) was achieved with the use of the secondary source slits [16(H)×6(V) μm^2] placed just behind the monochromator. The available flux was estimated to be 10^9 photons/s/0.01%bw in the spot. For applications requiring more intense beams, a flux of 10^{12} photons/s/0.01%bw in a 300×300 nm^2 spot can be obtained with the use of the secondary source slit only in the horizontal direction (the vertical slit is open). Figure 2(b) shows a scanning image of a Siemens star test chart, illustrating that our nanoprobe successfully resolves 100 nm line and space patterns. Note that the nearly distortion-free image has proved the high stability of our nanobeam. The apparatus installed in the upgrade, i.e., focusing mirrors, are fully compatible with the X-ray-energy tunability (5–16 keV) and polarization tunability, which are the original useful features of the beamlines.

The X-ray nanoprobe stations were open for public use in May 2011. XAFS measurement at a 100 nm resolution and XMCD experiments at a 300 nm resolution have routinely been carried out. Some preliminary results in several advanced fields have been obtained in the first and second user cycles since the nanoprobe stations opened: Nano-XAFS analysis has revealed the chemical-state distribution inside a single catalyst particle with a size of ~1 μm. The *in situ* observation of the structural transition in submicron-sized phase-change devices by nano-XAFS/EXAFS analysis is ongoing. Bit-patterned magnetic recording media were studied to characterize the elemental, chemical and magnetic states of a single magnetic nanodot using XRF, XAFS, and XMCD techniques, respectively. The magnetization reversal of individual nanodots of perpendicular magnetization was investigated by element-specific XMCD magnetization measurements at external fields up to 1.2 T.

In summary, we have upgraded the two existing beamlines installing the nanobeam capability. Local XAFS/XMCD spectroscopy in a 100 nm region and scanning XAFS/XMCD imaging with a similar resolution will be a powerful tool for the characterization of new functional materials for green/nanotechnologies. Our technical improvements achieved through the upgrade will allow a rapid installation of additional nanoprobe beamlines that will provide different promising applications including nanodiffraction, nanoimaging, and nano-hard-X-ray photoemission spectroscopy in the upcoming years.

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References

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