Development of scanning X-ray microscope using microdiffraction

A strongly correlated electron system often exhibits various interesting physical properties such as superconductivity, dielectricity, magnetism, multiferroic, and so on. To understand these phenomena, it is important to clarify not only the configurations of charge, orbital, and spin, but also its higher order structure (such as domain structure). In order to visualize these higher order structures, a scanning X-ray microscope system using microdiffraction are installed at **BL19LXU**. Here, we report the commissioning results of the microscope system, and the observed image of the charge order domain in $LuFe_2O_4$.

In the diffraction method, the obtained signal is coming from a sample where SR X-ray is irradiated. In an ordinal SR X-ray experiment, the diffraction signal reflects bulk averaged property, because the typical beam size is several hundred μm. If a focused beam less than the size of a domain is available, we can access the property coming from the pure single domain. In addition, if the sample is scanned, we can obtain the spatial distribution of the domain in real space.

In SR X-ray diffraction method, we can probe the information of orbital and magnetic orders in addition to charge order. However, we required the high photon flux of more than 10^{10} photons/s to observe them, because the intensity ratio of a magnetic reflection to the fundamental reflection is typically 10^{-8}. We also required a spatial resolution of 100 nm to achieve a resolution smaller than that of an optical microscope. Moreover, no chromatic aberration is required to execute energy scan, which is important in resonant X-ray scattering method to enhance a weak signal and to probe an element-selective signal. From the reasons stated above, we selected Kirkpatrick-Baez (KB) mirror system as a focusing device. As for the goniometer section with sample manipulation, we built two-axes sample scanning stages perpendicular to the scattering vector on the theta stage of a two-circle goniometer. These scanning stages have a feedback control system using an internal encoder to realize 10 nm resolution.

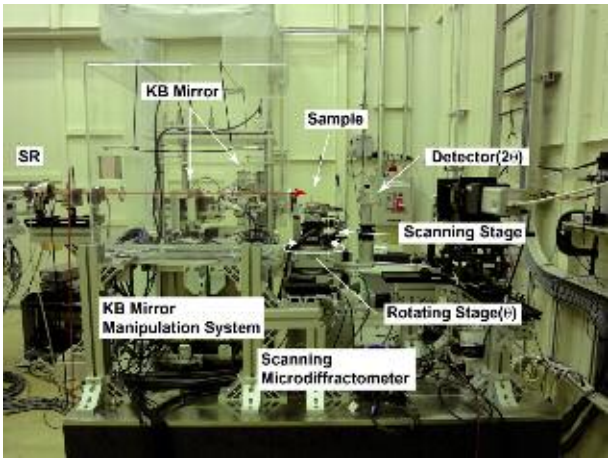

Fig. 1. KB mirror manipulation system and scanning X-ray microscope.

Figure 1 shows the photograph of the installed system. The KB mirror manipulation system is placed at the upstream side of the optical table and the goniometer follows the mirror. To increase the reduction rate of the beam size, a four-quadrant slit as the virtual light source is located 70 m upstream from the mirror, and the working distance of the mirror is designed as 100 mm. Figure 2 shows the schematic

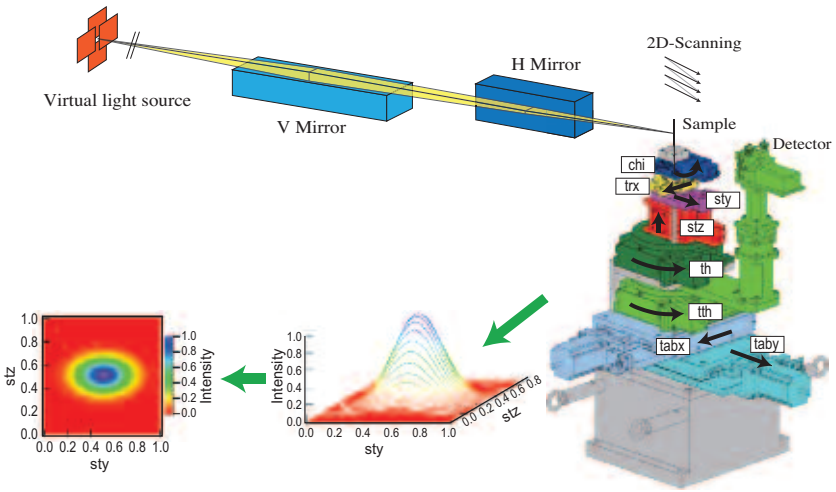

Fig. 2. Schematic of the scanning microdiffraction method.

view of this system. The goniometer consists of two translation axes for alignment of the rotation center and focal point (tabx, taby), three axes for diffraction (chi, theta, two theta), two axes for high resolution scanning (sty, stz) and one axis for sample alignment. A silicon drift detector (SDD) is adopted for energy-selective detection. To obtain a domain map, first, the three axes for diffraction are moved to the angles where a Bragg reflection can be observed. This Bragg intensity at each microregion of the sample is collected by scanning two axes (sty, stz). Then, a two-dimensional intensity map corresponding to the domain map is obtained.

As the evaluation of the KB mirror, we measured the beam profile by means of knife edge (wire) scan and photon flux of the focused beam. In this commissioning, we achieved a beam size of 100×100 nm^2 and a photon flux of 3.7×10^{10} photons/s. For the purpose of microscope evaluation, we also performed the scanning measurement of an X-ray test chart. We measured the transmitted beam intensity through the test chart in which some slit patterns are processed. As can be seen in Fig. 3, this system can be used to resolve a 100 nm structure. Furthermore, by adopting the deconvolution processing (Fig. 3), we determined that the standard deviation of the Gaussian function as a point spread function is 150 nm. It is confirmed from above results that the KB mirror system and the goniometer satisfy the performance for the observation of the higher order structures of charge, orbital and spin orders.

Finally, we show the observed charge order domain in $LuFe_2O_4$ using this system (Fig. 4). There are some superlattice reflections derived from the charge order at (1/3 1/3 *L*/2). In this observation, the goniometer axes are fixed under (1/3 1/3 37/2) reflection condition, and the sample is scanned to collect the intensity from each irradiated region. We are successful in observing some submicron domain with high resolution (the smallest size of the domain is ~300 nm).

This system will provide a new aspect to clarify interesting phenomena, such as phase coexistence or separation, and relations between domain structure and physical properties on strong correlated electron systems.

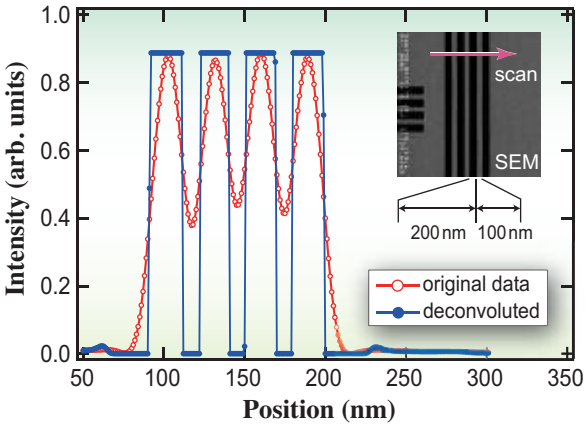

Fig. 3. Transmitted intensity profile of X-ray test chart at 200 nm line and 100 nm space. Inset shows the SEM image of the same position. The red arrow in the SEM image indicates the position of the scan.

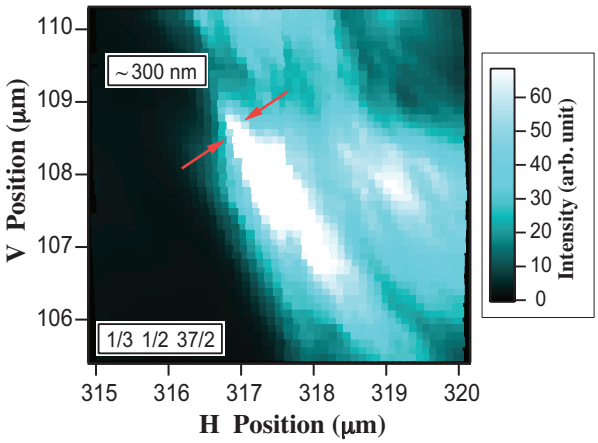

Fig. 4. Intensity map of 1/3 1/3 37/2 reflection of $LuFe_2O_4$. The distance between the arrows is approximately 300 nm.

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