Three-dimensional observation of magnetic domain structure by scanning hard X-ray microtomography

Over a century ago, Weiss theoretically predicted that ferromagnets have internal structures called magnetic domains: magnetization directions are uniform in an individual domain and may be different in different domains. Afterward, researchers found that magnetic domain structures reflect the fundamental magnetic properties of materials. Since the first experimental confirmation of the presence of a domain structure by Bitter, observation of the magnetic domain structure has been an important technique for understanding the magnetic characteristics of systems including practical magnetic materials. Today, observation at a high spatial resolution of a few 10 nm to 100 nm is possible with various magnetic microscopy techniques: magnetic force microscopy (MFM), magneto-optical Kerr microscopy, photoemission electron microscopy (PEEM), and scanning transmission soft X-ray microscopy (STXM). However, most of the existing techniques are limited to observations at the sample surface, where the magnetic domain structures are two-dimensional. The magnetic domain structure inside a bulk sample generally has a three-dimensional (3D) distribution and is closely related to the magnetic properties of the bulk system. For example, the 3D network domains inside sintered permanent magnets are related to the mechanism of magnetization reversal and the emergence of high coercivity. Elucidating 3D magnetic domains in a permanent magnet is an important task in materials science. Moreover, visualizing the topographic spin arrangement in spintronic materials is an intriguing subject in fundamental physics. Such research will also greatly contribute to technological applications. To date, only a few studies of 3D domain observations have been reported [1-3], including the successful 3D vectorial reconstruction of magnetic domains of $GdCo_2$ alloys via X-ray ptychography imaging by Donnelly *et al.* [3] In this study, we present another X-ray tomographic technique that allows observation of the internal magnetic domain structure in a micrometer-size ferromagnetic sample [4]. The technique is based on a scanning hard-X-ray nanoprobe using X-ray magnetic circular dichroism (XMCD). We demonstrate the 3D distribution of a single component of the magnetization vector in a GdFeCo microdisk, which has been reconstructed with a spatial resolution of 360 nm. This technique is applicable to practical magnetic materials including permanent magnets with sintered microstructures. 3D visualization of the magnetic domain formation process under external magnetic fields will be feasible.

Figure 1 shows the experimental setup for the scanning hard-X-ray microtomography constructed at the X-ray nanospectroscopy station of SPring-8 **BL39XU** [5]. The hard X-ray radiation from the in-vacuum standard undulator was monochromatized with a Si 111 double-crystal monochromator. A 0.45-mm-thick diamond X-ray phase retarder was used to generate circularly polarized X-ray beams of switchable photon helicity. Two elliptical mirrors in the Kirkpatrick-Baez configuration were used to focus the circularly polarized X-ray beam in the vertical and horizontal directions. The resulting X-ray beam size

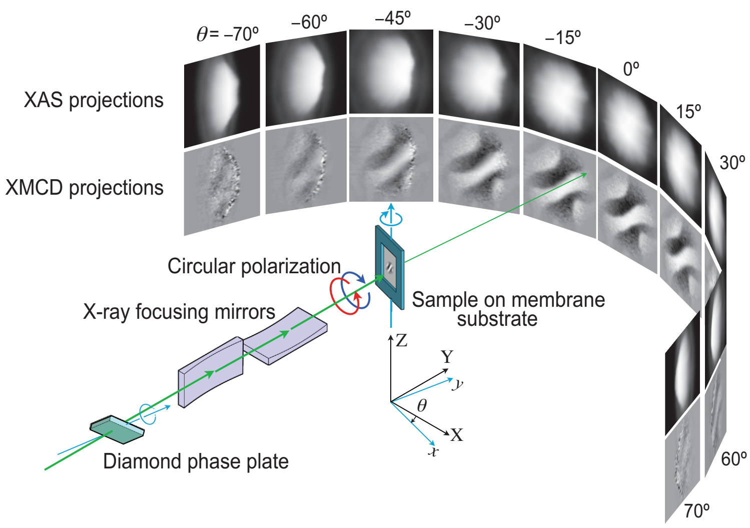

Fig. 1. Experimental setup of scanning hard-X-ray magnetic tomography. A cartoon scheme of a collection of XAS projections and magnetic contrast via XMCD projections at various angles is also shown.

was 130 (horizontal) × 140 (vertical) nm^2 in full width at half maximum (FWHM) at the sample position. To demonstrate the magnetic tomography technique, a microdisk made of $Gd_{22}Fe_{68}Co_{10}$ alloy was used as a sample. This material exhibits perpendicular magnetic anisotropy and maze-like domain structures with stripe widths of 2–3 μm. The sample was grown on a SiN membrane substrate by magnetron sputtering and then patterned into a disk shape by optical lithography and Ar ion milling. The diameter and thickness of the disk are approximately 7 and 3 μm, respectively. The microdisk sample was mounted on high-precision stages with *X*-*Y* translations and a vertical (Z)-axis rotation. X-ray absorption (XAS) images were taken by scanning the sample two-dimensionally in the plane perpendicular to the incident X-ray beam. Scanning magnetic images were simultaneously recorded by monitoring the XMCD signals using the helicity-modulation technique [4]. Figure 1 shows a cartoon scheme of taking images for tomographic reconstruction; the projected XAS and XMCD images were collected as a function of the rotation angle θ of the sample.

Figure 2 shows the 3D reconstruction of the GdFeCo magnetic microdisk. To reconstruct 3D XAS images, the standard algorithm of the algebraic reconstruction technique (ART) was applied to 37 projection images collected at angles from –70 to +70° with a step of 5°. The XAS reconstruction revealed that the sample was mostly homogeneous in composition. To reconstruct a 3D magnetic image, a modified ART algorithm was applied to XMCD projections taken at the same angles as those in XAS. In the modified ART, strong uniaxial anisotropy of the sample is assumed and a correction for the cosθ dependence of the XMCD amplitude is included [4]. In Fig. 2, a cutaway view of the XMCD reconstruction result demonstrates the 3D distribution of the magnetization inside the GdFeCo disk. The color scales correspond to the direction and the amplitude of magnetization perpendicular to the film. Five striped magnetic domains were clearly observed. The spatial resolution of the 3D XMCD reconstructed image was estimated to be 360 nm from the widths of the observed domain boundaries [4].

Our scanning hard-X-ray microtomography technique is applicable to various kinds of ferromagnetic samples including sintered permanent magnets. We are developing magnetic tomographic measurements under an external magnetic field for 3D observation of the evolution of magnetic domains in a bulk. The present setup has a relatively large sample space, making it suitable for introducing a specially designed magnet to allow tomographic measurement under a variable magnetic field. Additionally, the scanning X-ray setup can easily be modified for X-ray fluorescence microtomography and used to study the correlation between the magnetic domains and the elemental distribution via chemical and magnetic 3D imaging. Furthermore, in this study, a sample with uniaxial magnetic anisotropy was assumed and the distribution of only one component of the magnetization vector was obtained. Improvement in the experimental procedure, as well as in the reconstruction algorithm, is ongoing to achieve magnetic vector tomography [2,3].

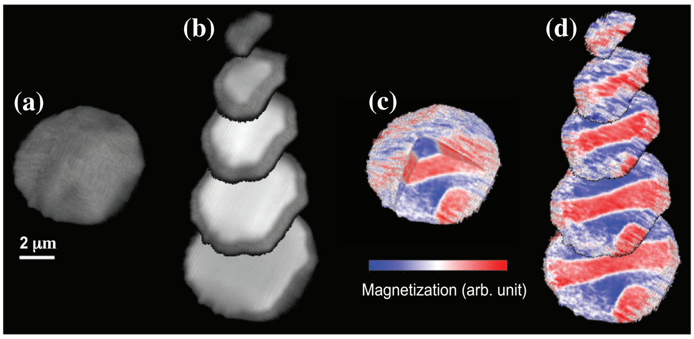

Fig. 2. 3D reconstruction images of GdFeCo microdisk. **(a)** Density distribution obtained by X-ray absorption tomography. **(b)** Cross-sectional images of the density distribution. **(c)** Magnetization distribution obtained by XMCD tomography. **(d)** Cross-sectional images showing the magnetization distribution inside the micrometer-size sample.

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