Application of XMCD-PEEM to the Magnetic-domain Structural Analysis of Nd-Fe-B Sintered Magnets

Nd-Fe-B sintered magnets have been widely applied for various motors, particularly in recent years, such as motors for hybrid electric vehicles, where higher coercivities are required compared with other applications. The microstructure of Nd magnets consists of polycrystalline $Nd_2Fe_{14}B$ with aligned magnetic easy axes. Each grain is surrounded by complementary phases such as an Nd-rich nonmagnetic phase. In order to understand the coercivity mechanism more clearly, it is important to elucidate the relation between these microstructures and the characteristics of magnetic reversal. X-ray magnetic circular dichroism and photoemission electron microscopy (XMCD-PEEM) can provide such information on these characteristics simultaneously, which is one of the most promising methods for the analysis of complicated domain structures and heterogeneous morphologies such as those in Nd magnets. Since the XMCD-PEEM imaging technique has been developed, it has been utilized for various investigations of mainly soft magnetic materials. To the authors' knowledge, there have been few such investigations on hard magnetic materials; thus, this work might be the first reported application of XMCD-PEEM to Nd-Fe-B sintered magnets. The aim of this study is to establish experimental methods for the domain analysis of Nd magnets using XMCD-PEEM techniques. We focused on the difference in domain structures between the thermal demagnetized and DC field-demagnetized states of Nd-Fe-B magnets.

Our experiments on XMCD-PEEM were performed at beamlines **BL25SU** and **BL17SU**. A schematic of the XMCD-PEEM imaging method is illustrated in Fig. 1.

Each beamline is equipped with a helical undulator, which generates circular polarized X-rays. The X-rays are monochromatized into the absorption edge energy of the magnetic elements present in the sample using varied-line spacing plane gratings (VLSPG), then is focused by the optical system. The core electrons of the sample are excited by the incident X-rays, which generate photoemission electrons. These photoelectrons are accelerated by a high voltage to form the XMCD-PEEM image on the screen. The core absorption intensities of the polarized X-rays vary according to the angle between the helicity and the magnetization vectors in the domains, which results in the contrast in the image due to the magnetic domains.

Nd-Fe-B magnets were prepared by powder metallurgical processing. The as-sintered samples were in the thermally demagnetized state. The DC field-demagnetized samples were prepared by a superconducting quantum interference device (SQUID; manufactured by Quantum Design Inc.). The thermally demagnetized samples were fully magnetized once in the positive direction followed by demagnetization in the reverse field, as can be seen from the minor loop in the second and third quadrants of the 4πI-H curve, in Fig. 2. The surface regions of the field-demagnetized samples were mechanically removed to exclude their individual coercivity characteristics. These samples were elementally analyzed by field-emission scanning electron microscopy and energy-dispersive spectroscopy (FE-SEM/EDS) prior to the XMCD-PEEM experiments. As a result, the complementary phases were identified as Nd oxides and Nd-rich regions (Fig. 3(a)). Figures 3(b) and 3(c) show the PEEM images obtained at the

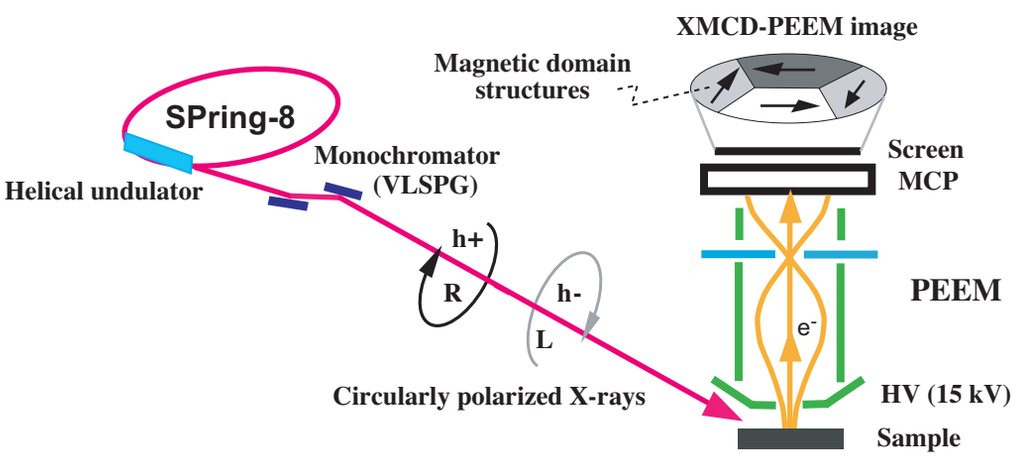

Fig. 1. Schematic illustration of XMCD-PEEM imaging.

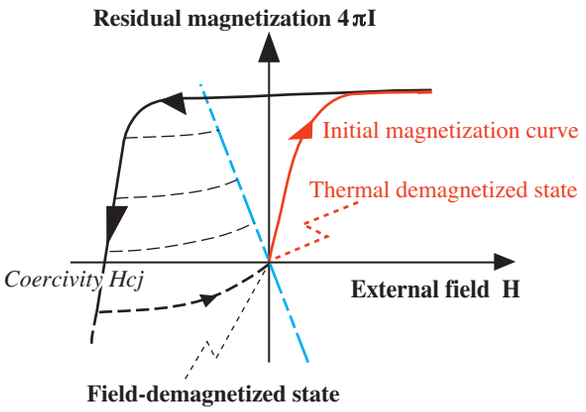

Fig. 2. Schematic magnetic hysteresis of Nd-Fe-B magnets during sample preparation starting from thermal demagnetized state to field-demagnetized state.

energies of the Nd-M_4 and Fe-L_3 edges, which were taken within the yellow- and blue-circled areas in Fig. 3(a), respectively. These images provide information on the elemental distribution upon selecting light with a suitable energy. Figure 3(d) shows the XMCD image of the same area as that shown in Fig. 3(c), which was taken at the energy of the Fe-L_3 edge. The structure appears to have a striped pattern in the lateral direction. In the thermally demagnetized state, each grain sample exhibits a multidomain structure, which is magnetically coupled across the grain boundaries over a wide range of length scales along

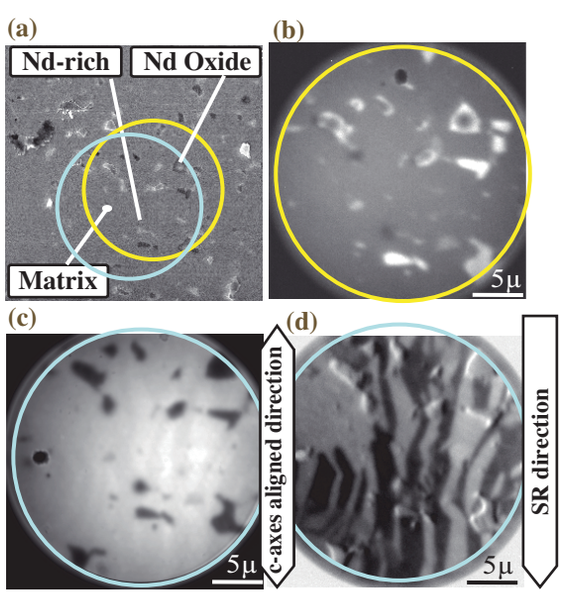

Fig. 3. Structures of magnetic domains and complementary phases of Nd-Fe-B sintered magnet in the thermally demagnetized state; **(a)** FE-SEM image (phases identified by EDS), **(b)** PEEM image of Nd-M_4 within yellow-circled area in Fig. 3 **(a)**, **(c)** PEEM image of Fe-L_3 within blue-circled area in Fig. 3 **(a)**, **(d)** XMCD image of Fe-L_3.

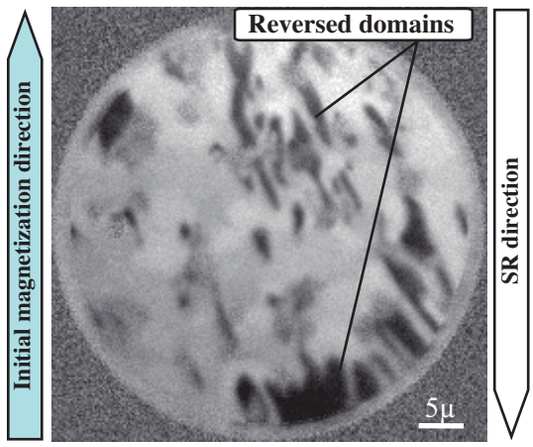

Fig. 4. Structures of magnetic domains of Nd-Fe-B sintered magnet in the field-demagnetized state: XMCD image, SR//c-axis (antiparallel); arrows: examples of reversed domains.

the easy-axis-aligned direction. This may be because of the requirement for preventing the generation of magnetic poles in the domain structure, so that the magnetostatic energy is minimized.

Figure 4 shows the XMCD image in the DC field-demagnetized state, which was taken with the incident X-rays antiparallel to the initial magnetization direction. In this domain image, the white region consists of mainly single domains, and is primary magnetized area, in contrast with the black region of reversed domains that belong to multidomain structures. These reversed domains exhibit a needle-like morphology in some areas, which agglomerate in other areas. Thus, the magnetic domain structures markedly changed after the DC field demagnetization.

The average width of the typical domains and the size of fine Nd-rich regions are approximately 1 μm and of submicron order, respectively. Since these sizes match the resolution of PEEM, XMCD-PEEM is an appropriate method for analyzing such complicated domain structures and the elemental distribution in the microstructures of Nd magnets.

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