Recent progress and future development of synchrotron X-ray analysis of high-performance permanent magnets

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Studies of the magnetization reversal process provide a key clue in uncovering the coercivity mechanism of permanent magnets. The magnetic domain structure inside a magnet forms a three-dimensional network in which one magnetic domain couples with its neighboring ones which have opposite directions of magnetization and are separated by inter- and intra-grain magnetic domain walls. When the magnetization is saturated by an external magnetic field that is sufficiently larger than the magnetic anisotropy field, the magnetic domain structure vanishes. Therefore, a clear-cut aim of studying the magnetization reversal process is to understand how the reversed magnetic domains are initially nucleated as the external magnetic field decreases and how the magnetic domain structure changes with other nucleation events and/or magnetic-domain-wall displacements.

Since their invention in 1984 [1], Nd-Fe-B magnets have been the best permanent magnets and have become an indispensable material for various electric products, hybrid vehicles, and power generators, which are now key technologies for energy sustainability. In the case of Nd-Fe-B sintered magnets, it is known that the coercivity in a polished surface is rather moderate in comparison to that of the bulk [2]. In stark contrast to the significantly decreased coercivity of the polished surface, we presented that the coercivity of the fractured surface closely resembles that of the bulk in a previous study [3]. The higher coercivity of the fractured surface is attributed to the particular way in which Nd-Fe-B sintered magnets fracture, where the majority of the fractured surface remains covered with a thin layer of the grain boundary phase. Although the similarity between the fractured surface and bulk coercivities cry out for magnetic domain observations of the fractured surface, conventional magnetic domain observations using Kerr microscopy, magnetic force microscopy, and photoelectron emission microscopy, have only been conducted on polished surfaces or thin films.

In order to observe the magnetic domain structure in the fractured surface under various magnetic fields, we developed a scanning soft X-ray spectromicroscope equipped with a superconducting magnet with a maximum magnetic field of ±8 T. When used in combination with X-ray photons of opposite helicity, and total-electron-yield detection, magnetic domain observations of the fractured surface become possible. Fig.1 shows the magnetic domain structure of the fractured surface of a Dy-free Nd-Fe-B sintered magnet (with composition $Nd_{14.0}Fe_{79.7}Cu_{0.1}B_{6.2}$) at an applied magnetic field of -0.7 T (after almost saturating the magnet at +3.0 T). In the figure, both the microstructure and the magnetic domain contrast are clearly observed. The magnetic field dependence of the magnetic domains has shown that the precise location in which reversed domains

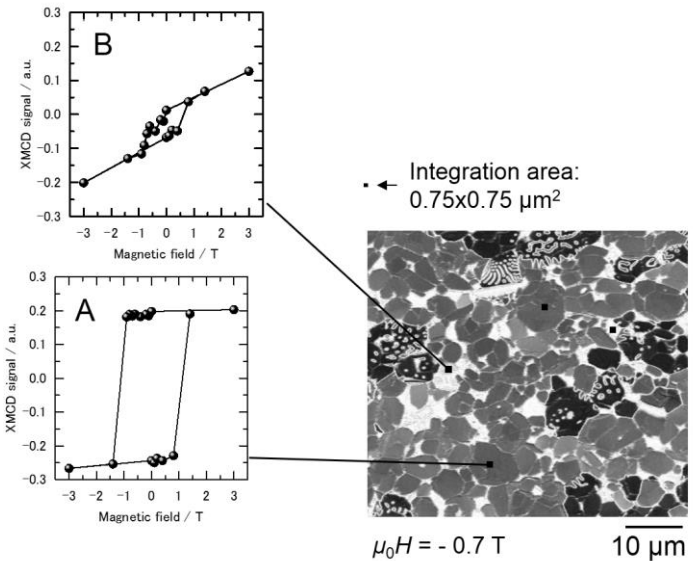

Fig.1 Magnetic domain structure of a Nd-Fe-B sintered magnet under an applied magnetic field of -0.7 T (*right*). Magnetic field dependence of XMCD intensity, A and B, give the local magnetic hysteresis loops in the selected grains.

are initially generated is always identical, and independent of whether the magnetic field is increased or decreased. Further analysis has allowed us to characterize the local magnetic hysteresis (MH) loops for areas ~ 100 nm^2. The observed local MH loops show a wide variety of magnetization reversal characteristics depending on the particular grain. As an example, we have plotted the MH loops for two different grains in Fig.1, labeled A and B, whose magnetization reversal characteristics are rather different. The observed differences between grains suggest that the magnetization reversal of each grain is very sensitive to the local effective magnetic field.

To understand the variety of local MH curves observed, we need to understand the local effective magnetic field. Prohibitively, the local effective magnetic field is very difficult to determine because it depends upon the stray magnetic field from the surrounding magnetic grains, which in-turn depends on the precise orientation of each grain, together with their intrinsic coercivity and the exchange coupling with the grains in the sub-surface layers. Unfortunately, the angle of the easy magnetic (*c*-)axis between each $Nd_2Fe_{14}B$ grain and the intended direction cannot be estimated from the local MH curves. Generally, the electron back-scatter diffraction (EBSD) technique is the most popular method to analyze the grain orientation. However, EBSD cannot be applied to the irregular fractured surface of our sample (which is the favorable target for magnetic domain observations). Therefore, we are developing a scanning X-ray micro-diffraction (SXMD) instrument which can probe any surface, independent of their roughness and irregularity. Moreover, the SXMD has a much longer probing depth than that of EBSD meaning that it is not necessary to worry about the surface state of the target sample, which makes sample preparation much easier. Fig. 2 shows a 3D schematic diagram of the SXMD apparatus under development at SPring-8.

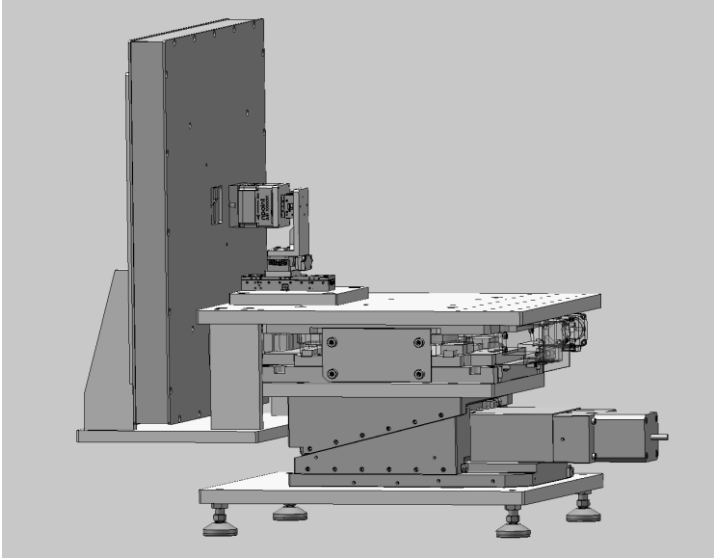

Fig.2 3D schematic diagram of the scanning X-ray micro-diffraction apparatus under development at SPring-8.

In this presentation, I will discuss our research involving the use of synchrotron X-ray diffraction to investigate the crystalline phases that constitute the microstructure, and scanning soft X-ray spectromicroscopy techniques to directly observe magnetization reversal process in Nd-Fe-B sintered magnets. I will also describe the progress made in the development of the scanning X-ray micro-diffraction instrument.

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