10 Elements Strategy Initiative Center for Magnetic Materials (ESICMM)

– in situ analysis using neutrons and X-rays –

PI: Kanta Ono

The goals of the Elements Strategy Initiative Center for Magnetic Materials (ESICMM) at the National Institute of Material Science (NIMS) are: (1) laboratory-scale synthesis of mass-producible high-performance permanent magnets without using critical rare-earth elements for the next generation and (2) framework-building and provision of basic science and technology for industrial R&D. To achieve these goals, ESICMM focuses on theoretical research and mining of new permanent magnet materials, and simultaneously pursues various processing technologies to improve the existing high-performance permanent magnet materials through cooperative activities in the three research fields of computer physics, structural and property characterization, and material processing. Another important mission of ESICMM is to train scientists who will contribute to the future development of magnetic functional materials.

In CMRC, the In-situ Analysis Using Neutrons and X-rays Project was started in July 2012 as an analysis group of ESICMM. The complementary use of neutrons at J-PARC/MLF and other facilities as well as synchrotron X-rays at the Photon Factory is very useful for characterizing magnetic materials from the atomic scale to micrometer scale.

We present two results of small angle neutron scattering (SANS) of Nd-Fe-B magnets.

10-1 Magnetization reversal process of Nd-Fe-B nanocrystalline magnets observed by magnetic SANS

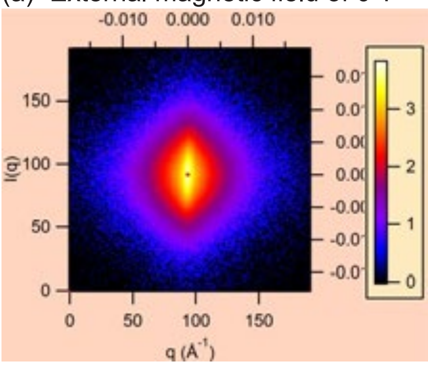
There is strong demand for the development of permanent magnet materials with high coercive force for industrial applications such as motors for hybrid and electric vehicles. The following phenomenological model of the coercive force was proposed by Kronmüller: $H_c = \alpha H_a - N_{eff} M_s$. The coercive force ($H_c$) is determined from the anisotropy field (H_a), the saturation magnetization (M_s), and microstructural parameters: α and the effective demagnetization factor (N_{eff}). Rare-earth iron boride $Nd_2Fe_{14}B$ exhibits both a high anisotropy field and high saturation magnetization, and $Nd_2Fe_{14}B$-based permanent magnets are widely used. However, in order to increase the coercive force further, the heavy rare-earth element Dy, which is one of the critical elements with an unstable supply, is substituted by Nd. Therefore, there is an urgent need to develop Dy-reduced and/or Dy-free permanent magnet materials. Reduction of the $Nd_2Fe_{14}B$ grain size is one way to realize a Dy-free Nd–Fe–B magnet with high coercive force. Hot-deformed Nd–Fe–B nanocrystalline magnets consist of $Nd_2Fe_{14}B$ grains of several 100 nm. Furthermore, the infiltration of RE–Cu (RE = Pr or Nd) eutectic alloy enhances their coercive force.

In order to unveil the microscopic mechanism of the coercive force, we have conducted a magnetic small-angle neutron scattering (SANS) study on as-deformed and RE–Cu-infiltrated Nd-Fe-B nanocrystalline magnets. The SANS experiment was performed at the V4 beamline at the BER-II in Helmholtz-Zentrum Berlin. SANS intensities were obtained during the magnetization reversal process from the fully magnetized initial state. Scattering intensities along the direction perpendicular to the nominal [001]-axis were maximized at the coercive force in all samples. External magnetic field dependence of the magnetic scattering intensity showed the suppression of intensity variation in the RE–Cu-infiltrated samples compared to the as-deformed sample [1,2]. This indicates that the $Nd_2Fe_{14}B$ grains are magnetically isolated in the RE–Cu-infiltrated Nd-Fe-B nanocrystalline magnets, resulting in the higher coercive force than that of the as-deformed one.

10-2 Initial magnetization process of Nd-Fe-B nanocrystalline magnets observed by magnetic SANS

Since the discovery of $Nd_2Fe_{14}B$, enormous efforts have been made to improve the magnetic performance of Nd-Fe-B permanent magnets in response to the growing need for strong permanent magnets for building an energy-efficient society [3,4]. However, some basic phenomena such as initial magnetization and reversal processes in the magnets have not been deeply understood due to the complexity arising from the inhomogeneity and multiscale structures of real magnets. Most of the related researches are based on microscopic observations; there have only been a few bulk experiments except some attempts to understand the physical picture of initial magnetization via macroscopic measurements [5,6]. SANS is a suitable probe to investigate initial magnetization and reversal processes inside bulk magnets which involve the development of magnetic domains with nano/micrometer size. We have conducted SANS experiments on the initial magnetization process in nanocrystalline Nd-Fe-B magnets with different grain boundary width to investigate the relation between microstructure and initial magnetization. SANS experiments were carried out at QUOKKA, ANSTO. 2D SANS images for a sample with the shortest grain boundary width are shown in Figure 1. Strong intensities along the direction perpendicular to the easy axis are observed for the thermally demagnetized state at 0 T (upper image) and it decreases with increasing external field *H* (lower image).

(a) External magnetic field of 0 T



(b) External magnetic field of 1.6 T

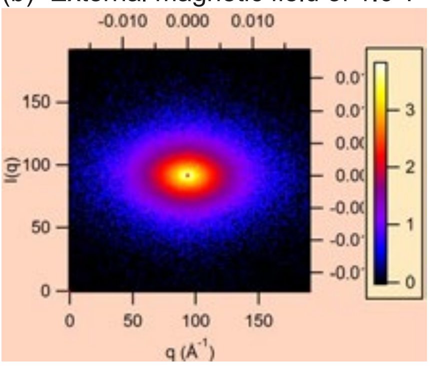


Fig. 1: SANS images of a nanocrystalline Nd-Fe-B magnet in the initial magnetization process.

References
[1] T. Ueno et al., IEEE Trans. Magn. **50** (2014) 2103104.
[2] K. Saito et al., J. Appl. Phys. **117** (2015) 17B302.
[3] M. Sagawa et al., J. Appl. Phys. **55** (1984) 2083.
[4] O. Gutfleisch et al., Adv. Mater. **23** (2011) 821.
[5] K. Kobayashi et al., J. Appl. Phys. **113** (2012) 023907.
[6] K. Kobayashi et al., J. Appl. Phys. **113** (2013) 163910.