

Visualization of the magnetization reversal processes in He jet-milled Nd-Fe-B sintered magnet by X-ray magnetic tomography

M Suzuki¹, M. Takeuchi², S. Kobayashi^{1,3}, R. Haga², Y. Kotani¹, T. Nakamura^{1,3,4}, N. Kikuchi², T. Sasaki^{3,5}, T. Ohkubo^{3,5}, Y. Une⁶, and S. Okamoto^{2,3}

¹Japan Synchrotron Radiation Research Institute, 1-1-1 Kouto, Sayo, 679-5198, Japan.

²IMRAM Tohoku University, Sendai 980-8577, Japan

³Elements Strategy Initiative Center for Magnetic Materials, NIMS, Tsukuba 305-0047, Japan.

⁴International Center for Synchrotron Radiation Innovation Smart, Tohoku University, Sendai 980-8577, Japan

⁵Research Center for Magnetic and Spintronics Materials, NIMS, Tsukuba 305-0047, Japan.

⁶Daido Corporate Research and Development Center, Steel Co., Ltd., Nagoya 457-8545, Japan

Introduction

The magnetic properties of Nd-Fe-B sintered magnets are closely related to the microstructure. To unveil the mechanism of the high coercivity emerged, the nucleation and evolution processes of reversed magnetic domains need to be clarified concerning the microstructure of a sintered magnet. Magnetic domain observation under an external magnetic field has so far been performed using Kerr microscopy and soft-X-ray scanning XMCD microscopy [1-4]. However, these existing techniques are limited to observe the domain structure on the surface of the sample, and it was not possible to investigate the magnetization reversal process inside bulk magnets. In this study, we used a recently developed hard X-ray magnetic tomography technique [5, 6] to directly observe the internal magnetic domain structure of Nd-Fe-B sintered magnets in a three-dimensional (3D) manner.

Experiment

Anisotropic Nd-Fe-B sintered magnet with an averaged particle diameter of 1 μm was prepared by the He-jet mill and pressless process [7]. A bulk specimen (remanent magnetization 1.5 T and coercivity 1.4 T) was micro-fabricated into a square column of $20 \times 20 \times 57 \mu\text{m}^3$ by a focused ion beam (FIB) method. A first order reversal curve (FORC) measurement was performed for another cutting sample with the same aspect ratio and larger dimensions, and it was confirmed that the FIB process has given little damage and the magnetic properties of the sample were almost unchanged. The hard X-ray magnetic microtomography experiment at the Nd L_2 edge (6.725 keV) was carried out at BL39XU of SPring-8 [5, 6]. The application of magnetic fields to the sample was performed off-line, and the magnetic domain structure of the remanent magnetization state at zero fields was observed by the X-ray tomography measurements.

Results

The formation and reversal process of the magnetic domains inside the bulk Nd-Fe-B sample was successfully observed in 3D. The widths of magnetic domains are comparable to the particle diameter, suggesting that the magnetization reversal of each particle has been visualized. Some inner grains are found to reverse independently. Thus, the nucleation points of magnetization reversal are directly demonstrated. In this talk, the correlation between the sintered microstructure and the magnetic domain structure will be discussed.

Part of this work is supported by ESICMM under the outsourcing project of MEXT (JPMXP0112101004).

[1] M. Suzuki *et al.*, *Acta Mater.* **106**, 155 (2016). [2] Y. Kotani *et al.*, *J. Synchrotron Rad.* **25**, 1444 (2018). [3] D. Billington *et al.*, *Phys. Rev. Mater.* **2**, 104413 (2018). [4] S. Okamoto *et al.*, *Acta Mater.* **178**, 90 (2019). [5] M. Suzuki *et al.*, *Appl. Phys. Express* **11**, 036601 (2018). [6] M. Suzuki *et al.*, *Synchrotron Rad. News* **33**, 4 (2020). [7] Y. Une *et al.*, *J. Jpn Inst. Met.* **76**, 12 (2012).

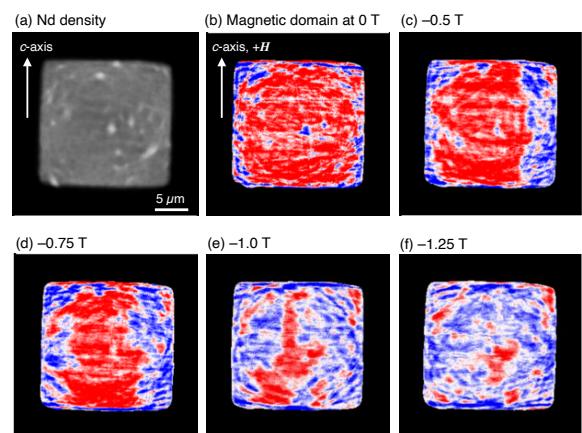


Fig. 1 Tomographic reconstructions of He-jet milled Nd-Fe-B sintered magnet. Cross sections of a square column sample at a plane parallel to the c-axis are shown.