# Observation of magnetic domain reversals in Nd-Fe-B hot-deformed and infiltrated magnets by SANS

M. Yano[1], T. Ueno[2], K. Saito[3], K. Ono[3], M. Harada[4], A. Manabe[1], T. Shoji[1], N. Sakuma[1], A. Kato[1], and U. Keiderling[5]

[1] Advanced Material Engineering Div., Toyota Motor Corporation, Susono 410-1193, Japan
[2]Elements Strategy Initiative Center for Magnetic Materials, National Institute for Materials Science, Tsukuba 305-0047, Japan
[3] High Energy Accelerator Research Organization (KEK), Tsukuba 305-0801, Japan
[4] Toyota Central R&D Labs. Inc., Nagakute 480-1192, Japan
[5]Helmholtz-Zentrum Berlin für Materialien and Energie, Berlin 14109, Germany

Nd-Fe-B magnet in motors in hybrid or electric vehicles requires Dy to increase its coercivity to maintain their magnetization in an operating temperature. However, Dy reduces magnetization in the magnet due to anti-parallel coupling of the magnetic moment between Dy and Fe [1]. To understand the coercivity mechanism in the Nd-Fe-B magnet to satisfy both high coercivity and magnetization, we have studied microstructures and magnetic domains in the magnet.

In general, the coercivity of Nd-Fe-B magnets can be enhanced by reducing the grain size of $Nd_2Fe_{14}B$ phase but it was found that only the grain size reduction cannot realize enough coercivity because of the reversal domain connections during demagnetizing procedures [2]. To reveal factors for higher coercivity, it is important to show the relation among magnetic properties, microstructures and magnetic domain reversals by using several kinds of methods because the microstructures and the magnetic domains are correlated to the coercivity.

Small-angle neutron scattering (SANS) is one of the complementary method to two-dimensional observations using microscope and it has a possibility to quantify the three-dimensional and multi-scaled microstructures and magnetic domains. The SANS enables us to investigate the bulk magnets using high transmission neutrons. Moreover, SANS can be performed under various sample environments of magnetic fields or temperatures.

In order to clarify the effect of grain isolation in nano-crystalline Nd-Fe-B magnets, we have prepared samples with different grain isolation and have performed SANS measurement to observe the difference in microstructures and magnetic domain reversals in these samples.

The hot-deformed Nd-Fe-B magnets have been made from rapidly quenched melt-spun ribbons. The melt-spun ribbons were crushed into a few hundred μm and then sintered at 873 K under a pressure of 100 MPa. The sintered bulk was hot-deformed with height reduction of about 80% to develop a (001) texture of phase. The hot-deformed magnet was soaked into the molten Re-Cu (Re=Pr or Nd) alloys to infiltrate into the grain boundaries to isolate grains [3]. The magnetic properties of as-deformed and Pr-Cu infiltrated samples are shown in Fig. 1 (a).

The SANS was performed at the V4 beamline of the research reactor BER-II at Helmholtz-Zentrum Berlin (HZB), Germany. The samples were fully magnetized along the easy magnetization axis ([001]-axis) at 10 T in advance. The sample temperature was set to 300 K. Neutron beam with the wavelength of 1.147 nm was used. Incoming beam direction was perpendicular to the [001]-axis and the irradiated area at the sample was set to 8 mm in diameter by the neutron window. The typical q range was 0.013–0.165 $nm^{-1}$ with a sample-detector distance of 15.76 m. External magnetic fields of ± 5 T were applied along the [001]-axis to obtain magnetic field-dependent SANS patterns.

The magnetic field dependences of intensity I(q) along the [001]-perpendicular direction for as-deformed and Pr-Cu infiltrated samples are shown in Fig. 1 (b). Note that the scattering vector $q$ and the dimension in real space $d$ can be converted using Bragg equation, $d = 2\pi/q$. The variation of the intensities in a specific $q$-range corresponds to the variation of the microstructure or magnetic domains of the dimension $d$. As shown in Fig. 1 (b), the intensities increase by applying the reversal field for as-deformed sample and become maximum at the coercive field of $\mu_0H_c$ = -1.46 T. For the infiltrated sample, intensities along the [001]-perpendicular direction also increase by applying the reversal field, as observed in the as-deformed sample, but the intensity difference is smaller. To magnify the magnetic field dependence as a function of the *q* or *d*, the intensity difference from that at 5 T are shown in Fig. 1 (c). The intensity difference becomes maximum at the coercive field of each sample. The intensity of smaller *d* range is dominant for as-deformed sample although the intensity of larger *d* range is dominant for infiltrated sample. These results indicate that the single particle reversal is dominant in the infiltrated sample implying that the larger ratio of the isolated grains than that of the interacting grains in the [001]-perpendicular direction, as compared to the as-deformed one.

These SANS results will be compared with the results of Nd-Cu infiltrated samples or direct magnetic domain observation by microscope.


Reference

[1] J. F. Herbst and W. B. Yelon, J. Appl. Phys. **57**, 2343 (1985).

[2] M. Yano *et al.*, IEEE Trans. Mag., **48**, 2804 (2012).

[3] H. Sepehri-Amin *et al.*, Acta Materia., **61**, 6622 (2013).


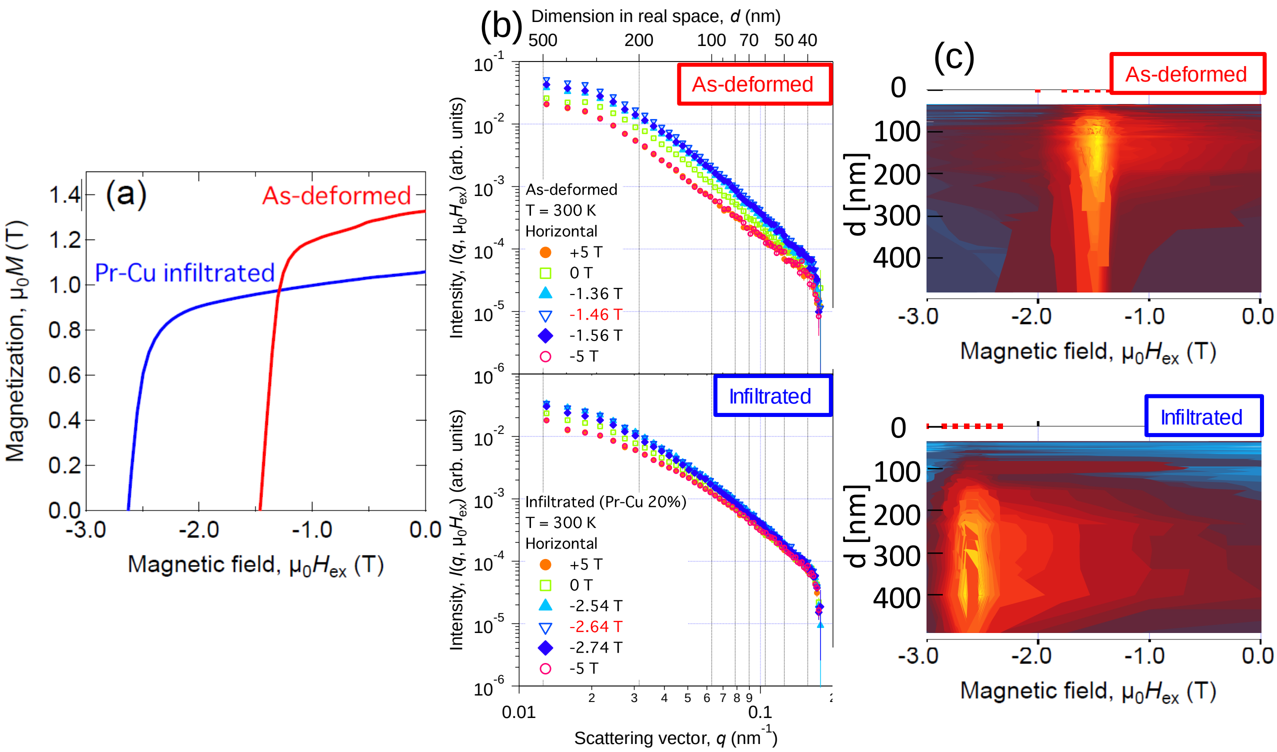


Fig. 1 (a) Demagnetization curves for as-deformed and Pr-Cu infiltrated Nd-Fe-B nano-crystalline magnets. (b) Magnetic field dependence of SANS intensities of as-deformed and infiltrated magnets. (c) Intensity difference between fully magnetized state and each demagnetization field. These intensity maps can be produced by (b).