Indirect Observation of Inner Domain Structure in Nd$_2$Fe$_{14}$B Sintered Magnets

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Abstract

Domain structures in sintered Nd$_2$Fe$_{14}$B magnets were studied using Kerr effect microscopy (KEM) and magneto-force microscopy (MFM). In the direction perpendicular to the crystal c-axis (c-plane), a fine maze domain structure was observed using KEM, but an only unclear complex structure can be obtained using MFM. After 5T pulse magnetization, both observation methods gave clear monotonous images of many magnetized grains and local fine domain structures with strong contrast in limited numbers of grains. On the other hand, in the direction of parallel to the c-axis (in a sense, a-plane), only bright or dark grains as a whole grain, were observed using both methods. These observations can be explained as that the surface demagnetized grains have a role of permeable medium of the magnetic fluxes from the inner domains. As a conclusion, when the inner grains generate sufficient magnetic fluxes to magnetize the surface no-coercive grains, the inner domain structures of the sintered Nd$_2$Fe$_{14}$B magnets can be observed using KEM and MFM.

Key words: Nd-Fe-B sintered magnets, Domain structures, Kerr effect microscopy, Magneto-force microscopy

1. Introduction

Crystal grains in the sintered Nd$_2$Fe$_{14}$B magnets lose their coercivities when the Nd-rich grain boundary layers are removed from the surface of NdFeB grains by polishing (1), (2). Based on this well-known fact, it has been often argued to be impossible to observe inner domain structures in the sintered Nd$_2$Fe$_{14}$B magnets, since the observable domain structures on the surface polished grains losing coercivity should be completely different from the inner domain structures of the sintered Nd$_2$Fe$_{14}$B magnets.

Nevertheless, the domain structures in the sintered Nd$_2$Fe$_{14}$B magnets were reported to be successfully observable by some researchers (3)-(5). However, they never discussed nor intended to observe the inner domain structures distinguishing from the domain structures in the surface grains.

In this paper, the authors will try to find the method to observe the inner domain structure in the sintered Nd$_2$Fe$_{14}$B magnets using Kerr effects microscopy (KEM) and magneto-force microscopy (MFM). The authors finally recognized that the inner domain structures could be indirectly observed through the surface demagnetized, almost no coercive, grains by polishing. The observed domain structures can be attributed to the magnetic flux propagation from the inner grains that keep the original domain structures even after the polishing.

2. Experimental and Results

The samples for this study are Dy-free Nd$_2$Fe$_{14}$B sintered magnets of NEOMAX-50 specification (containing a slight of Al and Cu doped elements) by NEOMAX Co., Ltd., Japan. The sample grains were highly oriented to the crystal c-axis before sintering, and the orientation was well kept after sintering also.

Fig.1 is a schematic representation of the observed and measured directions in this study. The a-plane (we are aware that the plane is not “a-plane” in strict sense, i.e. (hk0) directions, then we call them as “A-side”) and c-plane (B-side) are parallel and perpendicular planes to the crystal c-axis, respectively. The size of samples are approximately 5mm × 5mm × 6mm that is sufficiently large to inhibit the surface effects on hysteresis curves. The samples were embedded in epoxy resin and polished for KEM (Olympus Co., Ltd., BX60M) and MFM (Seiko Instruments Co., Ltd., SPI-3800N) observations using emery papers and alumina suspended liquids (minimum : 0.1 μm Al$_2$O$_3$ particles).

Fig.1 A schematic representation of the sample and directions of observations

Fig.2 (a) shows magnetic hysteresis curves measured in the directions of c-axis (//) and in the a-axis (⊥), respectively, measured using VSM-5-15, TOEI Industry Co., Ltd.. As shown in the hysteresis loops, the orientation of the crystal grains are very well, but the rectangularity of the loops is strongly...
influenced by the magnetization fields of 1.7T and of 5T pulse-field as shown in the figure. The corresponding coercivities for the applied fields are about 1.15T ($\mu_0 H_{appl}$) and 1.25T, respectively. The dispersion angle of crystal c-axes in the grains that was determined from the residual magnetizations in the orientated and in the perpendicular directions is less than 1.6 degree. The observations of domain structures were conducted in the samples shown in Fig.2 (b), i.e. the sample in thermally demagnetized state (“Initial” in the figure) and the residual magnetization state after the 5T-pulse magnetization (“5T-Mag” in the figure). In the magnetization curves in Fig.2, the demagnetizing fields were not corrected.

In the a-plane (A-side), the domain patterns are not observed even in the thermally demagnetized state as shown in Fig.3 (c). The contrast of KEM images changes in each whole grain, but the fine maze patterns as Fig.3 (a) was never observed in this plane. The same type of images were taken by Guttfleisch et al. (6). They, however, have no awareness about the surface polishing effects on the observed domain structures.

Fig.4 shows a portion of a-plane where the stripes of domain, about 1 μm thickness parallel to polished surface, can be noticed faintly. The Fig.4 (a) and (b) are the enhanced contrast images of KEM using a computer software. The enhancement grades at the same position are different between the figures. The domain stripes, however, have a clear connection between the grains. The stripes should correspond to domains parallel to crystal c-axis, but being originated the Kerr effect by the magnetic fluxes from the slightly tilting c-axis of the grains and/or under-layer grains that propagate the magnetic fluxes.

Fig.5 shows MFM images of the same type of polished sample. Fig.5 (a) and (b) show the thermally
demagnetized c-plane that corresponds to the KEM image shown in Fig.3 (a). The signals of MFM are finely vibrated on the image, and the domain structures shown in Fig.3 (a) cannot be clearly observed.

In Fig.5 (c) and (d), many surface grains are magnetized into single-domain state and give monotonous bright signal as a whole grain, and some grains show a clear characteristic domain structures that are obviously discriminated from those in Fig.5 (a) and (b). This observation is basically same with Fig.3 (b) by KEM.

At the top of cantilever in the MFM instrument, Pt-Co material is coated for magnetic flux sensing. Since this Pt-Co head interacts with the fluxes from the domains in crystal grains of the sample, the domain walls in the demagnetized state, in Fig.5 (a) and (b), should be quite sensitive to keep local energy balance between the head and domains.

On the other hand, the domain wall positions observed in Fig.5 (c) and (d), are fixed by the surrounding magnetized grains. Therefore, these domain images are very clear compared with these in Fig.5 (a) and (b) (see ref. (7)). The conditions of observation such as the distance of cantilever from the sample surface, and so on, are the same in both observations.

The MFM images of the thermally demagnetized a-plane (A-side), Fig.5 (e) and (f), are completely different from these of c-plane as shown in Fig.3 (c). Especially, Fig.5 (f) shows only grain morphologies that can be seen by slightly generated magnetic fluxes from the grain surfaces and grain boundaries, and there is no domain structure inside of each grain. This observation has good correspondence with the KEM observation shown in Fig.3 (c).

3. Discussion

The important points to understand the observations in this study are the followings. First, the polished surface grains lose their coercivity, and the multi-domain state should be the natural state without an external magnetic field for them. Second, the KEM is optical observation method, but the MFM is magnetic one, then the interaction between the magnetic fluxes from crystal grains and the light in the former method should be completely different from that in the later method, i.e. that between the magnetic fluxes and that from Pt-Co material at the top of cantilever.

The observed KEM image of the thermally demagnetized c-plane shown in Fig.3 (a) is clearly different from the MFM images of the same plane in Fig.5 (a) and (b). The maze patterns in Fig.3 (a) can easily be interpreted as the domain structures in the surface grains and/or in the inner grains. Both the surface and inner grains are thermally demagnetized into multi-domain state, and the coercivities of the surface grains should be very small caused by the polishing effects on them. Both the grains form the unique domain structures from inner to surface grains, to keep the energy minimum in the group of grains.

Therefore, the ambiguous domain structures in the MFM images in Fig.5 (a) and (b) only suggest the strong magnetic interaction between the magnetic fluxes from the grains, including both the surface and inner grains, and that from the Pt-Co material at the top of cantilever, but it is impossible to distinguish whether the interaction affects to the domain structure in the
surface grains or that in the inner grains.

When we observe Fig.3 (b) and Fig.5 (c) and (d), it is possible to discuss whether the above interactions affect on the surface grains or on the inner ones. Even after the magnetization under 5T-pulse field, the surface grains should return to multi-domain state in zero applied field because of their coercivities are very small. That is to say, there is no reason the grains magnetized into single-domain state that can be observed in both KEM and MFM images after 5T-pulse magnetization as shown in Fig.3 (b) and Fig.5 (c) and (d). Therefore, we should conclude that the observed single-domain state comes from the inner grains that still keep their coercivities, and the surface demagnetized grains should have a role of permeable media of the domain structures, i.e. magnetic fluxes, in the under (inner) layers.

If we reach to the discussion above, it should be very interesting that some grains after 5T-pulse magnetization still keep multi-domain state as shown in Fig.3 (b) and in Fig.5 (c) and (d). And also the clearness of the domain images in the residual multi-domain grains in these photographs, that is completely different from the MFM images in Fig.5 (a) and (b), should be caused by the strong magnetic fluxes from the surrounding grains in single-domain state. The domain walls in the residual multi-domain grains cannot move by the magnetic fluxes from the Pt-Co material at cantilever, then that may be expressed as a “frozen” domain structure.

The A-side observations shown in Fig.3 (c) and in Fig.5 (e) and (f) revealed that the magnetic fluxes in this side are very limited amount, and it is very difficult to obtain clear KEM and MFM images of the inner grains, except the edges of grains where the fluxes comparatively easily generate to outside of the plane. The apparent single-domain state in the surface grains in this side is important to understand the energy state as a whole sample. The observations on this side (a-plane), however, are important for the discussion of magnetic reversal mechanism in the samples, and should be developed to obtain clearer images of domain structures.

A schematic representation of the observed domain structure in the sintered magnets in this study is shown in Fig.6. Fig.6 (a) shows the relationship between the demagnetized, no coercive, surface grains and inner grains that still keep their coercivities. When the sample magnets are polished, the situation should correspond to Fig.6 (b) in which the surface demagnetized grains play a role of permeable media of magnetic fluxes from the inner, under layer, grains. In the case of the 5T-pulse field was applied, the surface demagnetized grains are also magnetized by the magnetic fluxes from the inner magnetized grains into single-domain state (Fig.6(c)).

**Conclusion**

In the cases of the domain structures in the inner grains generate sufficient magnetic fluxes to magnetize
the surface no-coercive grains, the domain structures in the inner grains in the sintered Nd-Fe-B magnets can be observed indirectly through the surface grains by polishing on the surface of magnets. The domain walls in thermally demagnetized state easily move by the application of magnetic field from the Pt-Co material at the top of cantilever of MFM. The domain walls in the residual multi-domain grains after 5T-pulse magnetization, however, cannot be moved by the flux from the cantilever, and it may be called as a “frozen” domain structures by the fluxes from the surrounding magnetized grains.

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<References>

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