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Application of MFM for studying Nd–Fe–B magnets

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Abstract

Magnetic force microscopy (MFM) was used to study the magnetic microstructure of sintered Nd–Fe–B permanent magnets at the surfaces perpendicular to the alignment axis. The coarse domain pattern is observed in the form of maze domains (of the order of 1 μm) and reverse spikes (of the order of 0.5 μm). It is similar to those present in thick uniaxial crystals with high magnetic anisotropy, reported in earlier investigations. In addition to this coarse domain pattern, a fine domain structure is detected. The MFM images are correlated with the topographic data recorded by atomic force microscopy.

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Keywords: Domain structure; Permanent magnets; Magnetic force microscopy

1. Introduction

There are a variety of techniques for the observation of magnetic domains. Among these methods, magnetic force microscopy (MFM), based on the magnetostatic interaction between the magnetic specimen and a magnetic tip placed over the specimen surface, is currently one of the most widely used, mainly because of its high spatial resolution (routinely better than 100 nm), high surface sensitivity and ease of application. The method has proved to be a useful characterization tool in both basic research and industrial applications [1].

A detailed knowledge of the magnetic microstructure is of great interest from both fundamental and technological points of view. In particular, it is very important for the development of high-performance permanent magnets. The purpose of this paper is to study the domain structure of sintered Nd–Fe–B permanent magnets at the surfaces perpendicular to the alignment axis, using MFM. In general, such an investigation presents two difficulties [2]. The first difficulty is associated with the fact that Nd–Fe–B magnets need careful surface preparation for magnetic domain observation. This is mainly because they are multiphase and chemically very reactive. The second difficulty is related to the presence of strong stray fields, of the order of 1 T, close to the specimen surface. As a consequence, this leads to perturbation of the magnetic state of the tip during MFM study.

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2. Experimental

The specimens studied were sintered $(Nd_{0.85}Dy_{0.15})_{14.5}Fe_{79}B_{6.5}$ magnets (from Sumitomo Special Metals Co., Ltd.) in the shape of cuboids a few millimetres in size. They consisted of large grains with an average size of about 10 μm. At room temperature the specimens had a coercivity ${}_JH_c > 1.67$ MA/m, a remanence $B_r = 1.26$ T and a maximum energy density $(BH)_{max} = 305$ kJ/m^3.

The magnetic microstructure was studied in the thermally demagnetized state at the surfaces perpendicular to the alignment axis. The observation surfaces were carefully polished to reduce the topographic contrast using successively finer abrasive papers and diamond powders (3, 1 and 0.25 μm in average diameter) with a water-free lubricant.

MFM measurements were carried out by an NT–MDT instrument operated in the dynamic mode using MikroMasch silicon cantilevers with tips magnetized along the tip axis, which was perpendicular to the specimen surface. In this case, MFM senses the vertical component of the derivative of the force between the specimen and the tip. The image signal was detected as the phase or amplitude shift of an oscillating cantilever. The tips used were coated with a Co film of about 60 nm in thickness, onto which a Cr protective film of about 20 nm in thickness was deposited. The coercivity of the tips was approximately 32 kA/m.

3. Results and discussion

Fig. 1a presents an MFM image of the domain structure of a sintered Nd–Fe–B magnet, while the corresponding atomic force microscopy (AFM) image is shown in Fig. 1b. It is seen in Fig. 1a that the domains form a maze structure on a scale of about 1 μm. The maze structure is characteristic of uniaxial materials having strong magnetic anisotropy. The maze domains are generally imaged as bright, while the domain walls between them are displayed as dark. The former means that, as expected, during the scanning process the tip magnetization was reversed by the stray field of maze domains, resulting in the loss of information on their sign. Within the maze domains there are smaller domains (called reverse spikes) visible in the form of circles. The reverse spikes are relatively small, conically shaped, surface domains and therefore contain only a small amount of material. As a consequence, the stray fields generated by the reverse spikes are insufficient to remagnetize the tip and they are displayed as dark, indicating that their magnetization is opposite to that of the maze domains within which they lie. Moreover, the reverse spikes are substantially displayed as unsharp, as reported and explained in Ref. [3]. The reason for the presence of the maze domain structure and reverse spikes at the surface perpendicular to the alignment axis is the reduction in the magnetostatic energy at the cost of a larger total Bloch wall area.

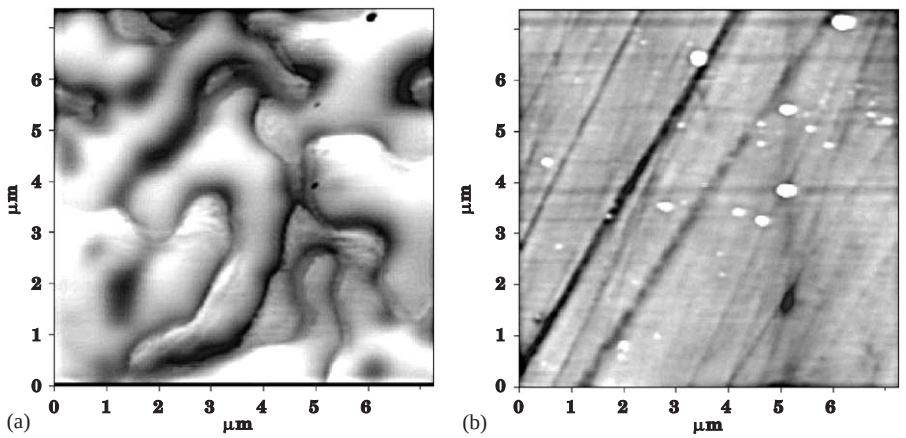

Fig. 1. (a) MFM image of a sintered Nd–Fe–B magnet collected with a tip-specimen separation of 100 nm and (b) the corresponding AFM image.

The MFM images are found to be consistent with those obtained by magneto-optic Kerr microscopy, as can be seen by comparing Figs. 1a and 2. The domain structure in Fig. 2 was made visible using the polar Kerr effect, which maps the perpendicular component of magnetization. Here the domains of opposite magnetization, along the axis perpendicular to the specimen surface, are displayed as dark and bright regions; in the image there are also present some topographic features (sharply delineated, mainly black, but also white) caused by the polishing process. In comparison with MFM, Kerr microscopy has the advantage of producing signal which is directly related to the magnetization but it possesses much worse spatial resolution as an optical technique.

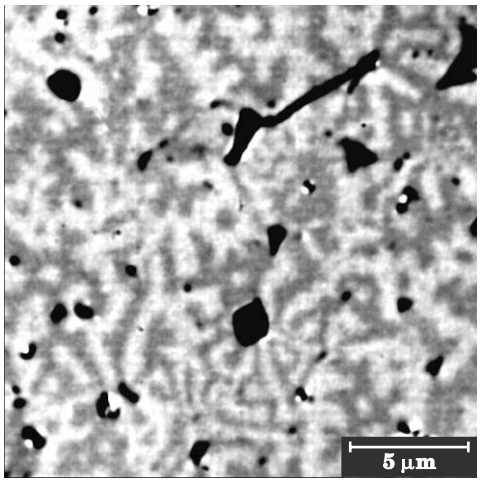

Fig. 2. Image of the domain structure of a sintered Nd–Fe–B magnet obtained by magneto-optic Kerr microscopy.

Comparison of MFM image with the corresponding AFM image allows to directly correlate the domain structure with the topographic data. In the case presented in Fig. 1, it is to be noted that some topographic features, visible as bright circles in the upper right quadrant of the AFM image, are reflected in the MFM image. In general, however, the magnetic domain structure is not influenced by the surface topography.

Interestingly, MFM observations show that in addition to the coarse maze domains and reverse spikes, there is a fine surface structure in the form of curved stripes. This fine scale structure is presented in a high-resolution image in Figs. 3a, and b shows the corresponding AFM topographic data. For a given MFM image with the fine domain structure, no such structure was observed in the corresponding AFM image, and moreover we did not observe any change of the MFM image with changing scan direction. This in turn means that the fine structure is of magnetic origin, and not an image artefact. Similar fine domain structure was reported for the first time by the authors of Ref. [4] for a Nd–Fe–B specimen of different chemical composition than the specimens

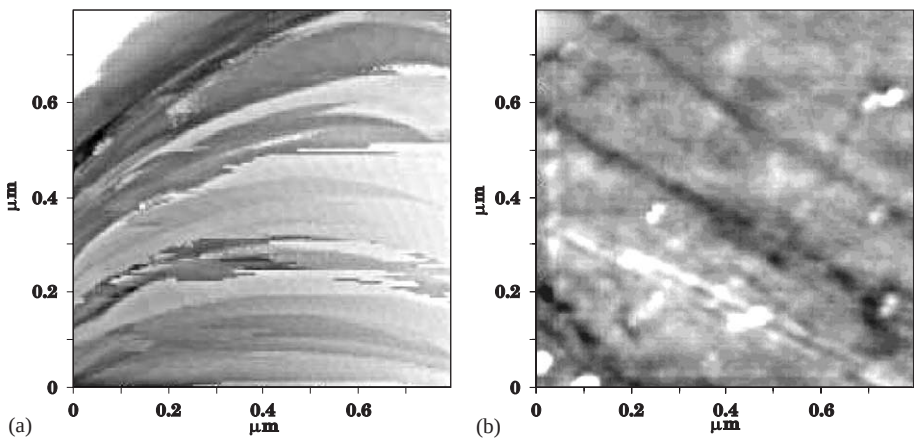

Fig. 3. (a) High resolution MFM image of a sintered Nd–Fe–B magnet recorded with a tip-specimen separation of 100 nm and (b) the corresponding AFM image.

used by us in this work. The MFM method with the tip magnetization perpendicular to the specimen surface is insensitive to in-plane components of magnetization [5]. As a consequence, the fine scale domains have their magnetizations perpendicular to the surface. They are found to reduce the magnetostatic energy in close proximity to the specimen surface.

4. Conclusions

We applied the MFM technique to observe the domain structure of sintered Nd–Fe–B permanent magnets at the surfaces perpendicular to the alignment axis. The domain structure is found to be complex. It consists of the coarse maze domains (of about 1 μm in size) and surface reverse spikes (of about 0.5 μm in size), which were also observed with magneto-optic Kerr microscopy. This structure resembles those for sufficiently thick uniaxial crystals with high magnetic anisotropy, for example, magnetoplumbite or barium ferrite, reported in earlier studies [6].

In addition to the mentioned structure, there are fine scale surface domains (of about 50–150 nm in size). Their observation was possible thanks to the fact that MFM possesses the high spatial resolution and high surface sensitivity, much better than other methods which rely on the specimen stray field, such as Bitter pattern technique or type-I magnetic contrast in the scanning electron microscope (SEM) [7]. Moreover, thanks to simultaneous collection of the topographic data by AFM, it was possible to directly correlate the MFM images with the surface topography.

Acknowledgements

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