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Magnetic domain observations of $(Tb_{0.3}Dy_{0.7})Fe_{1.95}$ alloys

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The magnetic domain structures of $(Tb_{0.3}Dy_{0.7})Fe_{1.95}$ alloys were investigated using scanning probe atomic and magnetic force microscopies. Results demonstrate that the specimen surface presented corrugated and spiked domain configurations interpreted in terms of surface closure domains in cubic magnetic materials after the specimen was annealed at 673 K for 2 h in an ultra-high vacuum of 5×10^{-5} Pa. However, a thin soft magnetic Fe layer appeared on the specimen surface after annealing at 723 K for 2 h in a relatively high vacuum of 5×10^{-3} Pa. As a result, the demagnetized energy in the specimen surface is largely reduced and the interior magnetic domain configurations of the specimen are observed. The results maybe provide an approach for detecting the interior magnetic domain structures of TbDyFe alloys or other cubic magnetic materials.

Key words: magnetic domain, magnetic force microscopy (MFM), demagnetized energy, TbDyFe.

Introduction

The first reported domain studies on TbDyFe alloys were carried out by Clark et al. using synchrotron radiation X-ray topography [1, 2]. Later, many papers reported the magnetic domain observation of TbDyFe samples using Kerr Magneto-optics [3, 4], Bitter technique [5], differential interference contrast microscopy [6, 7], transmission electron Lorentz microscopy [8-10], and scanning probe microscopy [11-13]. However, the relations between the observed surface closure and interior or bulk magnetic domain structures are not clear. It is of significance to reveal the bulk domain structure of $Tb_{0.3}Dy_{0.7}Fe_2$ crystals since it is related to the magnetostrictive properties directly. In general, the surface domain configuration can be revealed easily using magnetic force microscopy (MFM). However, the interior magnetic domains of cubic materials are not generally detected using MFM when their thickness exceed the critical value of domain branching.

In this study, the magnetic domain structures of $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ alloys were investigated using AFM and MFM. We found by chance that the interior magnetic domain structures could be detected by MFM when a thin soft-magnetic Fe film appeared on the surface after the sample was annealed at 723 K for 2 h in a vacuum of 5×10^{-3} Pa. The interior domain configurations were interpreted in terms of low-energy 71° and 109° domain walls in the <110> plane surface of $(Tb_{0.3}Dy_{0.7})Fe_{1.95}$ alloys. The mechanism of the appearance of the interior domains will also be discussed.

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Experimental

The master alloys were prepared using 99.9% pure Tb, Dy and Fe in the proportion of $(Tb_{0.3}Dy_{0.7})Fe_{1.95}$. The experimental alloy rods were fabricated in a directional solidification furnace specially designed for the "one-step" process [14]. The specimens for testing were taken out 120 mm away from the bottom of the rods. The crystal preferred orientation was detected using a Bruker AXS D8 GADDS X-ray apparatus form the transverse section of the specimen. The Curie temperature of the specimen was measured to be about 658 K by thermo-magnetic analysis, as listed in Table 1. The surface roughness and magnetization distribution of the specimen were observed using a Digital Instruments NanoScope IIIa-D3000 scanning probe atomic and magnetic force microscopies after the speci-

Table 1. Surface roughness (R_q) and corresponding phase roughness ($R_q(\Delta\phi)$) and the magnetic domain width W_d of MFM contrasts of <110> oriented $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ alloys after being annealed at 673 K in an ultra-high vacuum of 5×10^{-5} Pa and 723 K in a vacuum of 5×10^{-3} Pa, respectively. The Curie temperature T_c of the specimen is also listed

Condition of annealing	R_q (nm)	$R_q(\Delta\phi)$ (°)	W_d (μm)	T_c (K)
673 K, 5×10^{-5} Pa	3.8	8.1	0.4	658
723 K, 5×10^{-3} Pa	11.3	2.8	2.6	

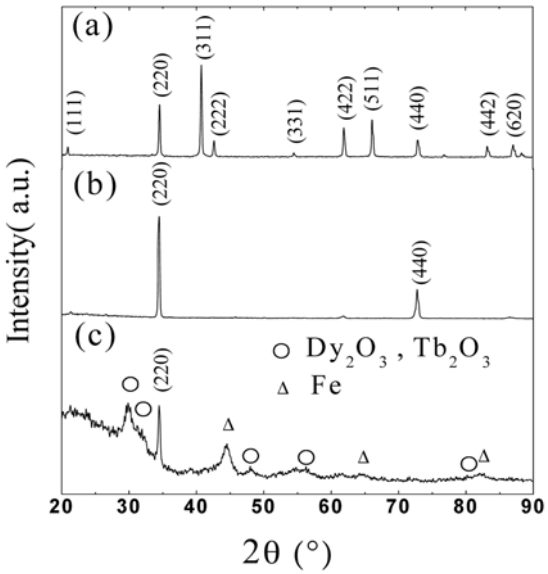

Fig. 1. X-ray diffraction patterns of <110> oriented $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ specimens. (a) the powders of the specimens; (b) after annealing at 673 K for 2 h in a vacuum of 5×10^{-5} Pa; (c) after annealing at 723 K for 2 h in a vacuum of 5×10^{-3} Pa.

men was cut, ground, polished and annealed above the Curie temperature. The magnetic probes used were micro-fabricated Si cantilevers with a pyramidal tip coated with about a 40 nm thick magnetic Co-Cr film and magnetized downward prior to imaging. The lift-height of the tip was 100 nm.

Results and Discussion

The result of X-ray diffraction analysis of the $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ alloys is presented in Fig. 1. It can be seen clearly from Figs. 1(a) and 1(b) that the sample possesses a <110> preferred orientation. No oxide and second-phase were detected in the X-ray patterns after the sample was annealed at 673 K for 2 h in an ultra-high vacuum of 5×10^{-5} Pa. However, the oxides of Tb and Dy elements as well as soft magnetic Fe appear besides the (220) peak after the specimen was annealed at 723 K for 2 h in a vacuum of 5×10^{-3} Pa, as shown in Fig. 1(c).

Figure 2 shows a typical 80 mm × 80 mm AFM and the corresponding MFM image of a $(Tb_{0.3}Dy_{0.7})Fe_{1.95}$ sample after it was annealed at 673 K for 2 h in an ultra-high vacuum of 5×10^{-5} Pa. The sample surface, as shown in Fig. 2(a), is relatively smooth besides the scratch line in the left-top region. The surface roughness R_q was measured to be about 3.8 nm, as listed in Table 1. The magnetic configurations shown in Fig. 2(b) are the characteristics of the surface closure domain in cubic magnetic materials. The surface domain width W_d and the phase roughness $R_q(\Delta\phi)$ of the specimen are measured to be about 0.4 mm and 8.1°, respectively (see Table 1). The internal domains incline towards the surface due to the magnetization perpendicular to the

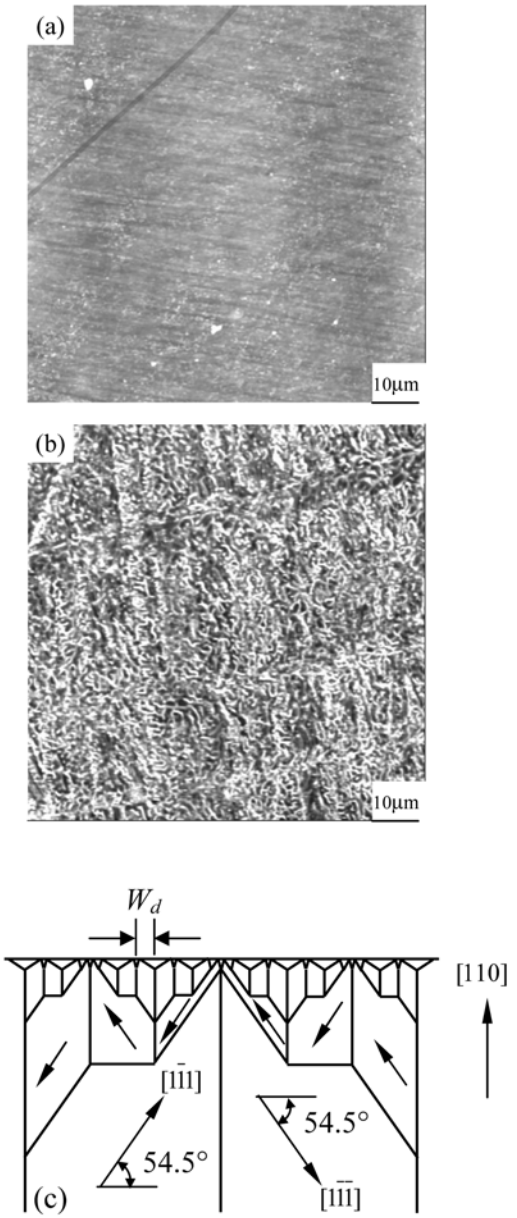

Fig. 2. Typical 80 μm×80 μm AFM (a) and corresponding MFM (b) images of the <110> oriented specimens at room temperature. The z range of AFM and MFM images is 100 nm and 60°, respectively. A model of domain refinement of Fig. 2(b) is proposed as (c) according to the echelon pattern.

specimen surface. As a result, the sample surface presents corrugated and spiked domain configurations in order to decrease the demagnetized energy. A model of the domain refinement of Fig. 2(b) is proposed in Fig. 2(c) according to the echelon pattern [15].

The typical 80 mm × 80 mm AFM and the corresponding MFM images of the sample after annealing at 723 K for 2 h in the relatively high vacuum of 5×10^{-3} Pa are given in Fig. 3. As shown in Fig. 3(a), the surface of the specimen became rougher compared with Fig. 2(a). The surface roughness R_q, as listed in Table 1, is about 11.3 nm. Furthermore, the phase roughness $R_q(\Delta\phi)$ of the specimen is decreased to be

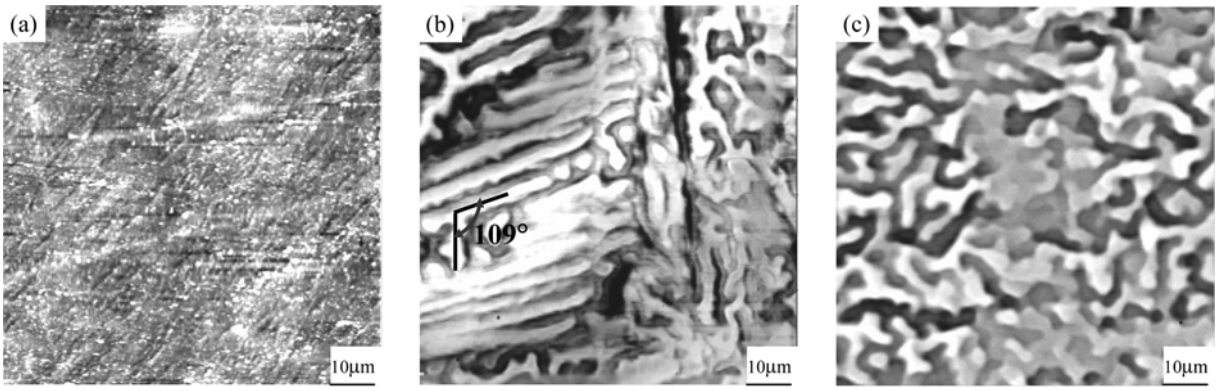

Fig. 3. Typical 80 μm×80 μm AFM image (a) and two MFM images (b, c) of the <110> oriented specimens at room temperature after annealing at 723 K for 2 h in a vacuum of 5×10^{-3} Pa. The z range of AFM and MFM images is 100 nm and 20°, respectively.

about 2.8°. It is greatly less than that of Fig. 2 and indicates that the stray field of the specimen surface became weak. Figures 3(b) and 3(c) show two typical interior domain configurations of the <110> oriented specimen. In Fig. 3(b), the 180°, 71° and 109° domain walls can be found clearly, which is consistent with the fact that the <110> plane surface contains two easy axes with angles of 71° or 109° because the easy axis of the magnetization of the Laves phase $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ crystals is <111> direction at room temperature. The results conform to the model of 71° and 109° walls in the <110> surface of the $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ crystals, as illustrated in Fig. 4. However, the magnetic configuration shown in Fig. 3(c) is striped domains. This may be the reason that the other two easy axes of the $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ crystal play a leading role in this region. It is obvious that the magnetic domain width of Fig. 3(b) and 3(c) is much more than that of Fig. 2(b). The corresponding domain width was measured to be about 2.6 mm (see Table 1).

To explain the appearance of the interior domains, the surface compositions of the specimens annealed in the relatively high vacuum of 5×10^{-3} Pa were analyzed by XRD, as shown in Fig. 1(c). As we know, the oxides of Tb and Dy elements, Tb_2O_3 and Dy_2O_3, are paramagnetic, and the Fe film is a soft-magnet with in-plane magnetization at room temperature. In our case, this soft magnetic layer is a "capping" layer, and actually acts as a shielding layer for the stray fields emerging from the underneath plate-like domains, which may be verified by the fact that the phase roughness of the MFM image (Fig. 3(b)) of the specimen annealed in the vacuum of 5×10^{-3} Pa is only 2.8°. Therefore, the demagnetized energy in the surface layer is largely reduced and the formation of a surface closure domain pattern is no longer necessary. As a result, the inner plate-like domains of the specimen are presented and detected by the magnetic tip as striped domains, which is similar to the results observed in the surface layer of

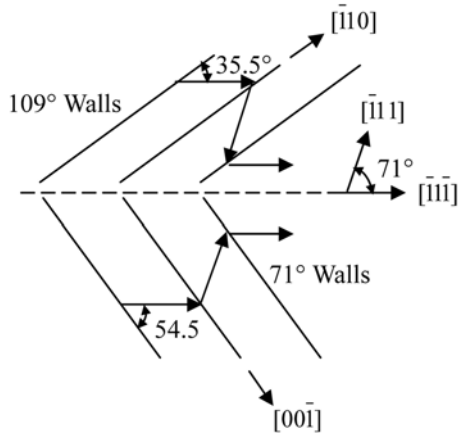

Fig. 4. Model of 71° and 109° walls in the <110> surface of the specimen.

Nd-Fe-B magnets due to the implantation of Fe ions [16].

Conclusions

The sample surface presented corrugated and spiked closure domain configurations to decrease the demagnetized energy after the sample was annealed at 673 K for 2 h in an ultra-high vacuum of 5×10^{-5} Pa. A thin soft magnetic Fe layer appeared in the specimen surface after annealing at 723 K for 2 h in a relatively high vacuum of 5×10^{-3} Pa. As a result, the demagnetized energy in the specimen surface is largely reduced and the interior magnetic domain configurations of the specimen are revealed by MFM because of the thin Fe layer shielding in part the internal stray field of the crystals. The results indicate that the $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ alloys are sensitive to the degree of vacuum when being annealed. Furthermore, this study maybe provides us with an approach for detecting the interior magnetic domain structures of $Tb_{0.3}Dy_{0.7}Fe_{1.95}$ alloys or other cubic magnetic materials by scanning probe microscopy.

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