Stress-induced magnetic domain structure in $DyFe_{11}Ti$ compound

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Abstract. In this work, we present the results of the $DyFe_{11}Ti$ compound magnetic domain structure (DS) exploration. It was found that mechanical polishing leads to a change of the anisotropy of the surface layer, which affects the magnetic DS configuration. It is established that the equilibrium structure of the surface domains is restored through time under unchanged external conditions. It is suggested that the mechanism of DS relaxation has thermal fluctuation nature.

1 Introduction

The analysis of magnetic domain structure (DS) allows to estimate some intrinsic parameters of the obtained phases [1, 2]. Among a large number of the experimental techniques for DS investigation, magneto-optical Kerr microscopy is the most appropriate, because it can provide the researchers with a high-throughput characterization data.

Nevertheless, there are many reasons why the quantitative analysis of magnetic properties from domain structure can be erroneous. It is known that the magnetoelastic contribution can change the magnetic anisotropy type of the sample surface layer in comparison with the anisotropy of the sample volume [3, 4]. In this work the results of experimental study of the magnetic domain structure of the $DyFe_{11}Ti$ compound, as well the analysis of the mechanical stress influence on the surface DS of bulk crystals are presented.

The $RFe_{11}Ti$ intermetallic compounds are now reconsidered as promising materials for permanent magnets with low contents of rare earth materials. This compound has $ThMn_{12}$-type tetragonal crystal structure (space group I4/mmm) and is characterized by reasonably high values of the Curie temperature and of the spontaneous magnetic moment [5-7]. $DyFe_{11}Ti$ compound has uniaxial magnetic anisotropy at room temperature. This compound is characterized by the two spin-reorientation phase transitions (SRT). According to the literature data, SRT temperatures significantly vary: the SRT1 is observed in the temperature range of 98–120 K, SRT2 – 214–250 K [5-10]. Besides, $DyFe_{11}Ti$ compound possess the large magnetostriction [11]. Temperature study of the magnetic DS of $DyFe_{11}Ti$ single crystals was carried out in [3,4], where it was shown by means of magnetic measurements that two SRT occurred in the sample (T_{srt1}=275 K and T_{srt2}=125 K), and at the same time two types of areas with different SRT temperatures were observed by means of DS study. The presence of impurities of the second phase can be excluded since the studies were carried out on single crystals.

2 Experimental

The $DyFe_{11}Ti$ compound was synthesized by induction melting of stoichiometric composition under argon atmosphere. The resulting ingot was sealed in an evacuated quartz tube and annealed in a resistance furnace. Several large grains were extracted and checked for single crystallinity. According to metallographic, thermomagnetic and X-ray analyses the samples were single phased and had $ThMn_{12}$ structure.

The preparation of the sections was carried out by using the set of diamond pastes. After polishing the sample was cleaned with ethanol. The time between the final stage of preparation procedure of the sample surface and the beginning of observations did not exceed 1 minute. To verify the effect of mechanical stress on surface of the section this procedure was repeated several times on the samples with different crystallographic orientation of the observation plane. It was established that even after a brief polishing the visible transformation of the surface DS began only 30 minutes after starting of the observations.

The magnetic domain structure of the samples was investigated by means of the polar magneto-optical Kerr effect on Axiovert 200MAT (Zeiss) in the temperature range from 273 K up to 423 K.

MFM measurements were carried out at room temperature on Scanning Probe Microscope Solver Next (NT-MDT (Russia)) using magnetic cantilevers. The measurements were executed by two-pass method.

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During the first pass, the topography is studied by standard tapping AFM mode. In the second pass the cantilever is lifted to a certain height (150 nm) for each scan line and performs the scanning, subtracting the stored topography.

3 Results and discussion

The surface domain configuration of a uniaxial magnetic material is determined by the quality factor $Q = K_1/(2\pi I_S^2)$ (K_1 – magnetocrystalline anisotropy constant, I_S – magnetization saturation value). $DyFe_{11}Ti$ compound reveals $Q \sim 2.98 > 1$, thus it is a highly anisotropic magnetic material, in which the surface domain configuration is determined by the energy of the stray fields of the sample. At room temperature, the equilibrium surface DS on the basal plane of the samples with uniaxial anisotropy demonstrate "stars-like" domain. The impact of abrasive materials on the sample surface leads to the formation of stressful surface layer. This effect can be attributed to tensile stresses, which affect the domain configuration. In particular, the magnetic domains on the basal plane of the $DyFe_{11}Ti$ single crystal are not immediately detected by the polar Kerr effect after mechanical polishing of the surface. However, the labyrinth domain structure is detected by the Bitter method and by using ferrite-garnet indicator tape. This is because the surface layer is thin enough for the Bitter method and indicator tape to depict the stray field of the main domains. The main feature of the presented experimental DS observation was the fact that the surface domain structure returns to its equilibrium state through time without impact of an external magnetic field and at constant temperature. Fig.1a presents the labyrinth domain structure on the $DyFe_{11}Ti$ single crystal 1 hour after a short polishing. After 3 hours, the contrast of domains significantly increased (fig.1b), while the boundaries of the labyrinth domains began to take on sharp outlines, in which one can see the emerging "star" domains. The typical surface domain structure formed on the basal plane after 10 hours under constant external conditions (fig.1c). The reason that the method of the polar Kerr effect does not depict the domains in the basal plane immediately after mechanical polishing is that the magnetic moment is oriented in the observation plane due to stresses. Through time, the magnetic moment begins to deviate from this direction and DS is visualized in polarized light. The necessary relaxation time depends on the duration and severity of

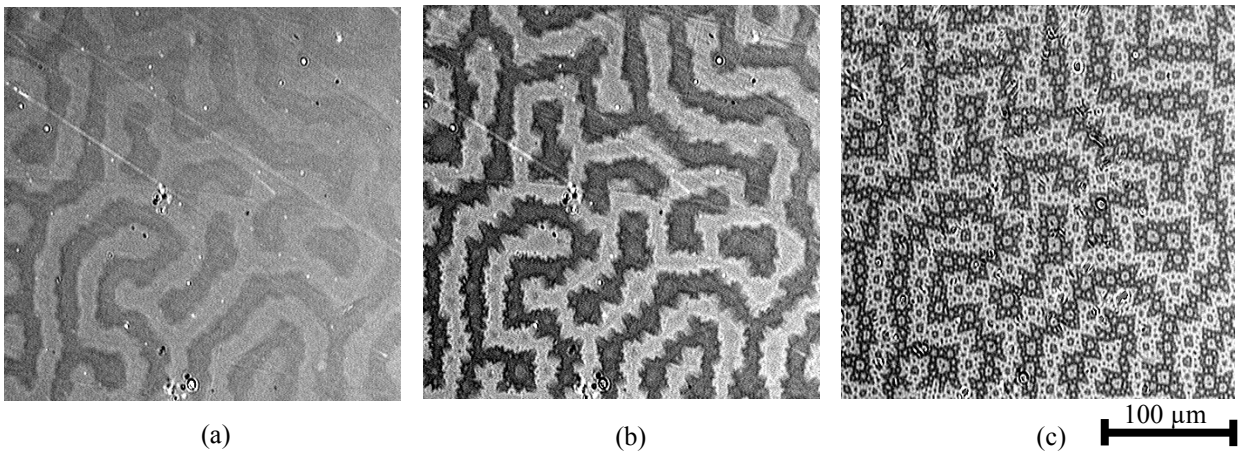

Fig. 1. The magnetic domain structure transformation on the basal plane of $DyFe_{11}Ti$ single crystal detected by Kerr magneto-optical microscopy: a) after 60 min, b) after 3 h, c) after 10 h.

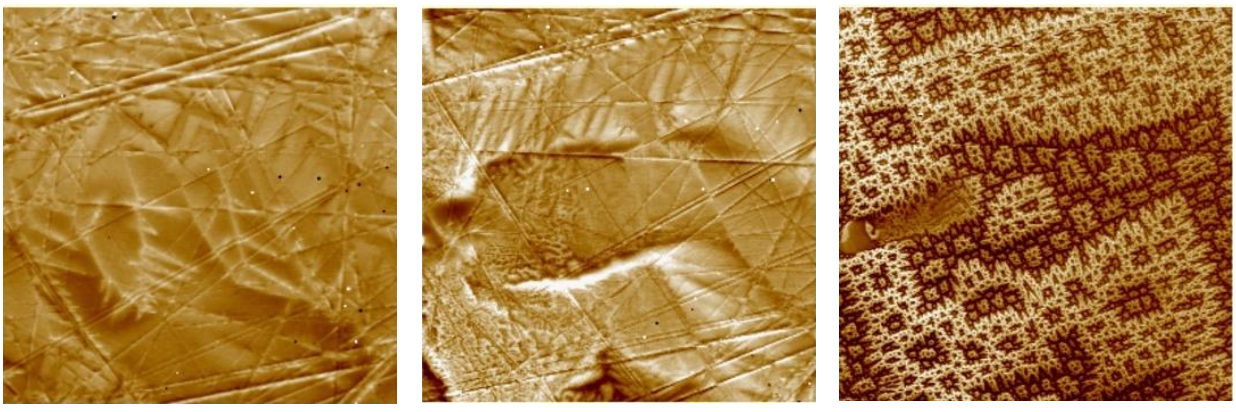

Fig. 2. MFM images of the magnetic domain structure relaxation process on the basal plane (001) of $DyFe_{11}Ti$ single crystal at room temperature: a) after mechanical polishing immediately, b) after 45 h, d) after 300 h (100x100 μm)

mechanical polishing and temperature of the sample.

It should be noted that the heating of the single crystalline specimen up to 423 K after mechanical polishing the relaxation time reduced down to 1-2 minutes. Moreover, the stages of the DS relaxation transformation remain exactly the same as at the room temperature. The restoration of the equilibrium structure always begins at the edge of a single crystal or a grain.

An MFM study allowed to identify the DS on the (001) surface of the sample just after polishing. Registration of magnetic stray fields was carried out at the distance of 150 nm above the surface. This allows to visualize not only the surface defects, but also the domain boundaries.

In contract with Fig.1, where the relaxation of DS after a short-term effect of the polishing on surface of the section was shown, Fig. 2 depicts DS images obtained after a complete surface treatment of the same single crystal, including the grinding and polishing steps. The degree and duration of the impact on the surface significantly affect the speed of the relaxation process.

As it is shown in Fig.2a, the $DyFe_{11}Ti$ sample demonstrates typical multi-axis magnetic DS. After 45 hours in the same region of the surface domain structure has changed: the resulting image corresponds to the stray fields of the labyrinth structure. After 300 hours the equilibrium typical branched (closure-type or star-like) domains structure has formed on the surface. Thus, the rate of restoration of the equilibrium domain structure has increased by a factor of 30.

Moreover, the relaxation process study of the polycrystalline samples has shown that the relaxation rate of the grains with various crystallographic orientations differs: restoration of the surface DS of the grains with an orientation close to the basal plane was faster than for grains with the prismatic orientation.

Analysis of the surface domain parameters allowed us to calculate values of the surface density of energy (γ) and width (δ) of domain walls. To determine the values of γ and δ theoretical and experimental approaches were used.

For the theoretical calculation the expression obtained for the 180° domain walls was applied:

$$\gamma = 4\sqrt{A \cdot K_1} \quad (1)$$

$$\delta = \pi\sqrt{A/K_1} \quad (2)$$

where A=159,12×10^{-13} J/m is the exchange parameter, K_1= 8,31×10^5 J/m^3 is magnetocrystalline anisotropy constant. The set of obtained parameters values is summarized in Table 1.

The second Bogenberger-Hubert method based on the analysis of experimental images of the magnetic domain structure gives the following equation:

$$\gamma = \frac{L\, I_S^2}{4\,\pi\,\beta} \quad (3)$$

where L is the average distance between domain boundaries, I_S = 665 Gs – saturation magnetization value, β=0,31±0,02.

Table 1. The surface density of the energy (γ) and width (δ) of domain walls

δ, Å	γ, J/m^3	
	calc.	exp.
137	145,0	108,0

On arbitrarily oriented plane, the structure of closure-type domains of the sample and the DS of surface stressed layer is identified by the polar effect Kerr technique at the same time. Fig.3a shows images of the nonequilibrium DS obtained in the grains of polycrystalline sample, which orientations are close to the prismatic plane. It is seen that the walls of surface layer domains are oriented perpendicular to the walls of the main domains.

Analysis of all obtained data suggests that the mechanism of relaxation of the surface DS has thermal fluctuation character.

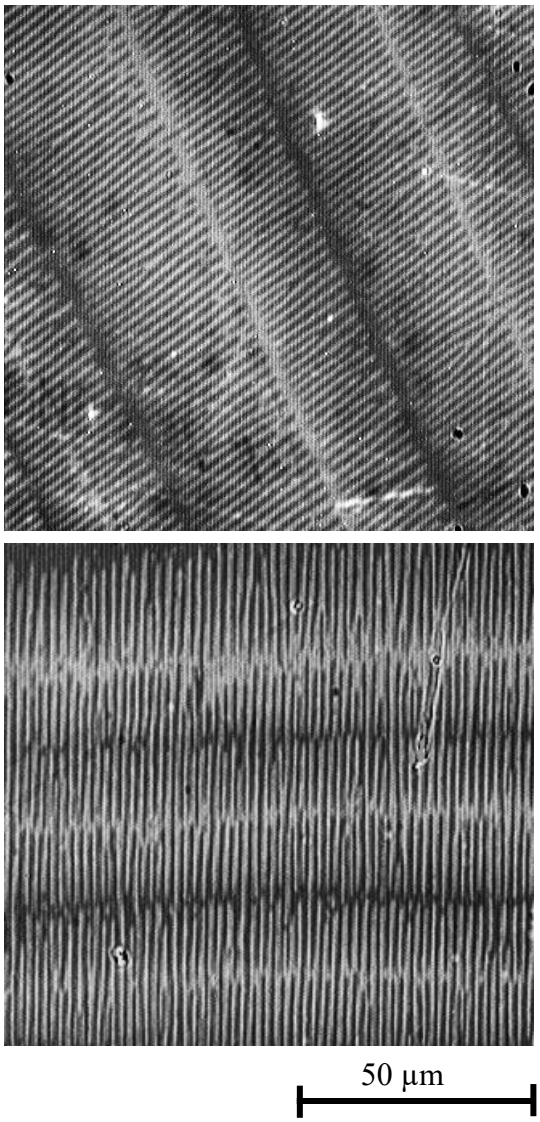

Fig. 3. Magnetic domain structure on the arbitrary plane of $DyFe_{11}Ti$ polycrystalline sample detected by Kerr magneto-optical microscopy at room temperature after mechanical polishing.

Conclusions

It was established that the mechanical impact induced during polishing in the single crystal and polycrystalline $DyFe_{11}Ti$ compound leads to configuration change of the surface domain structure.
Through time in the absence of external magnetic fields stress-induced surface domain structure relaxes into an equilibrium configuration that is typical for a magnetic material with the uniaxial anisotropy.
The relaxation time of the surface domain structure depends on the duration and severity of exposure to the surface, and the sample temperature. Heating the sample enhances the recovery process of the surface domain structure, which indicates the thermal fluctuation nature of the relaxation process.
Summarizing the obtained results, the observation of domain structure by means of magneto-optical Kerr microscopy can be used as high-throughput analysis technique for characterization of new uniaxial phases within combinatorial analysis. Nevertheless, it should be mentioned that detection and quantitative analysis of uniaxial phases can be erroneous due to the impact of external stresses during polishing. To eliminate the possible errors one need to use some chemical etching or thermal or magnetic treatments.

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