

CONDENSED MATTER PHYSICS

Influence of Annealing on Magneto-Optical Properties of $\text{Fe}_{72.4}\text{Ti}_{5.4}\text{B}_{19.2}\text{O}_{3.0}$ Nanocrystalline Films

N. N. Perova^{1*}, E. A. Ganshina^{1**}, I. M. Pripechenkov¹, D. M. Gridin¹,
E. V. Harin², V. A. Tejetov², E. N. Sheftel², and A. B. Granovsky^{1,3}

¹Department of Magnetism, Faculty of Physics, Lomonosov Moscow State University, Moscow, 119991 Russia

²Baikov Institute of Metallurgy and Materials Science, Russian Academy of Sciences, Moscow, 119334 Russia

³Institute for Theoretical and Applied Electromagnetics, Russian Academy of Sciences, Moscow, 125412 Russia

Received May 6, 2024; revised July 8, 2024; accepted July 11, 2024

Abstract—The results of the study on the magnetic properties of $\text{Fe}_{72.4}\text{Ti}_{5.4}\text{B}_{19.2}\text{O}_{3.0}$ nanocrystalline films, subjected to annealing in vacuum at 200, 300, and 400°C, are presented. Films with a mixed (nanocrystalline + amorphous) structure, 0.52 μm thick, were obtained by magnetron deposition on glass substrates. Hysteresis loops were measured using a vibrating magnetometer, and the field and spectral dependences of the transverse Kerr effect (TKE) were determined. Magnetization reversal processes were visualized using a magneto-optical Kerr magnetometer. It was shown that the processes of partial crystallization of the initially amorphous phase and the redistribution of Ti and B within crystalline grains and grain boundaries, leading to the formation of new phases as a result of annealing, manifest themselves in a two-stage magnetization reversal process and in the modification of the TKE spectra.

Keywords: soft magnetic films, hysteresis loop, transverse Kerr effect, magneto-optical Kerr magnetometer

DOI: 10.3103/S0027134924701935

INTRODUCTION

Films of Fe-based alloys with nanocrystalline and dispersion-hardened structures, obtained by magnetron deposition followed by annealing, can exhibit high saturation magnetization, low coercivity, high magnetic permeability up to gigahertz frequencies, and stability [1, 2], making them competitive with the best amorphous-nanocrystalline soft magnetic materials, which are mainly produced in the form of ribbons by melt quenching [3]. The structure and properties of Fe-based films are optimized by varying the conditions and modes of deposition and subsequent annealing. The magnetic properties of thin film alloys critically depend on the chemical and phase composition of the material, its structure (grain size, microstrain within the grain, interphase boundary structure, etc.), and micromagnetic structure (bulk and surface) [4, 5]. In this work, $\text{Fe}_{72.4}\text{Ti}_{5.4}\text{B}_{19.2}\text{O}_{3.0}$ films obtained by magnetron deposition were chosen as the model system. Magnetostatic measurements were carried out, and, using a set of magneto-optical

methods, the bulk and near-surface magnetic properties were studied, which made it possible not only to identify their differences but also to visualize the magnetization reversal process.

1. SAMPLES AND MEASUREMENT TECHNIQUE

Samples, in the form of films with a thickness of 0.52 μm on glass substrates, were obtained by magnetron deposition at MISiS following the technique described in detail in [2]. For comparison, a nanocrystalline Fe film was also produced. The samples were annealed at temperatures of 200, 300, and 400°C for 1 h in a vacuum of 2×10^{-4} Pa. Before annealing, the films were characterized by a mixed structure consisting of nanocrystalline and amorphous phases. The ratio of these phases changes as a result of annealing [6]. Hysteresis loops were measured using a Lake Shore 7407 vibrating magnetometer. Three magneto-optical methods were used in the geometry of the transverse and longitudinal Kerr effects (TKE/LKE):

1. measurement of field dependences at different wavelengths (TKE);

*E-mail: perova.n@physics.msu.ru

**E-mail: eagan@mail.ru

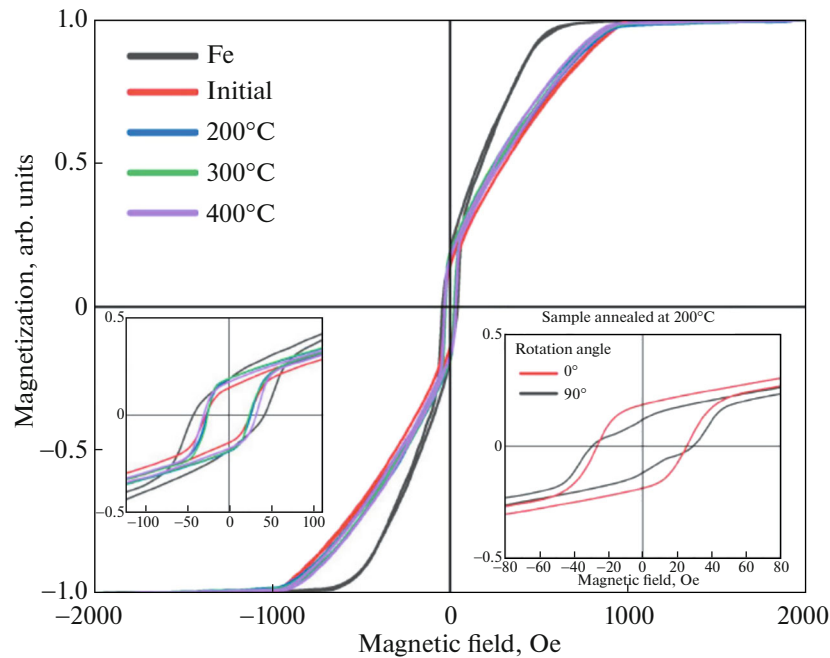


Fig. 1. Bulk hysteresis loops for nanocrystalline Fe and $\text{Fe}_{72.4}\text{Ti}_{5.4}\text{B}_{19.2}\text{O}_{3.0}$ films in the initial state and after annealing at different temperatures. The left inset shows an enlarged central part of the loops, while the right inset illustrates the anisotropy of the hysteresis loop after a 90° rotation of the sample annealed at 200°C .

2. measurement of spectral dependences of TKE in the range of 0.5–3.5 eV;
3. measurement of hysteresis loops and visualization of magnetization reversal using an Evi-comag magneto-optical Kerr magnetometer (LKE).

All measurements were performed at room temperature.

2. RESULTS AND DISCUSSION

Figure 1 presents the bulk hysteresis loops obtained using a vibrating magnetometer. The loops are shown in normalized form and generally have a similar shape. Among them, the loop of the nanocrystalline Fe film, provided for comparison, stands out due to its higher coercivity. It is also worth noting that all the films exhibit similar hysteresis loops regardless of the angle at which the field is applied in the plane of the film, indicating that they are isotropic. The exception is the film annealed at 200°C , which shows slight angular anisotropy when measured with the magnetometer (see the left inset in Fig. 1).

Figure 2 shows the hysteresis loops of the near-surface region, where the magneto-optical signal is generated. The magneto-optical signal is formed at a depth of approximately $\lambda/4k$, where λ is the wavelength of the radiation, and k is the absorption coefficient at this wavelength [7], which is no

more than 20–30 nm. In other words, the hysteresis loops determined using LKE reflect the magnetization reversal of the near-surface layer with a thickness

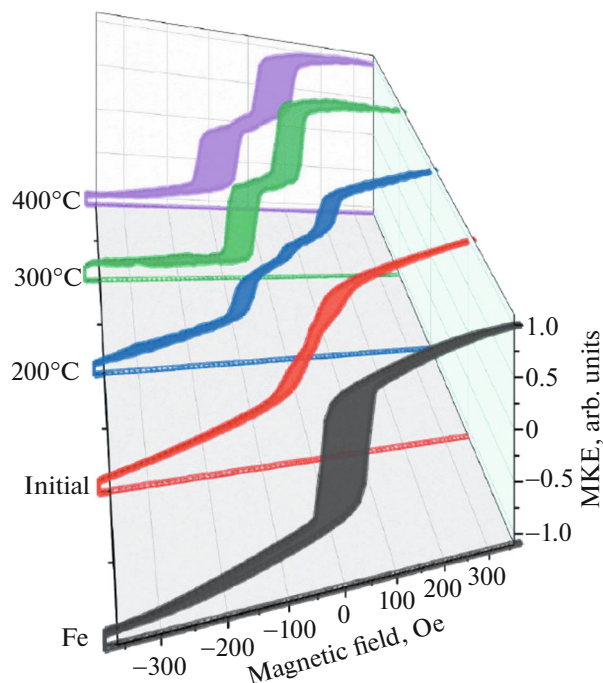


Fig. 2. Near-surface hysteresis loops for $\text{Fe}_{72.4}\text{Ti}_{5.4}\text{B}_{19.2}\text{O}_{3.0}$ films in the initial state and after annealing at different temperatures.

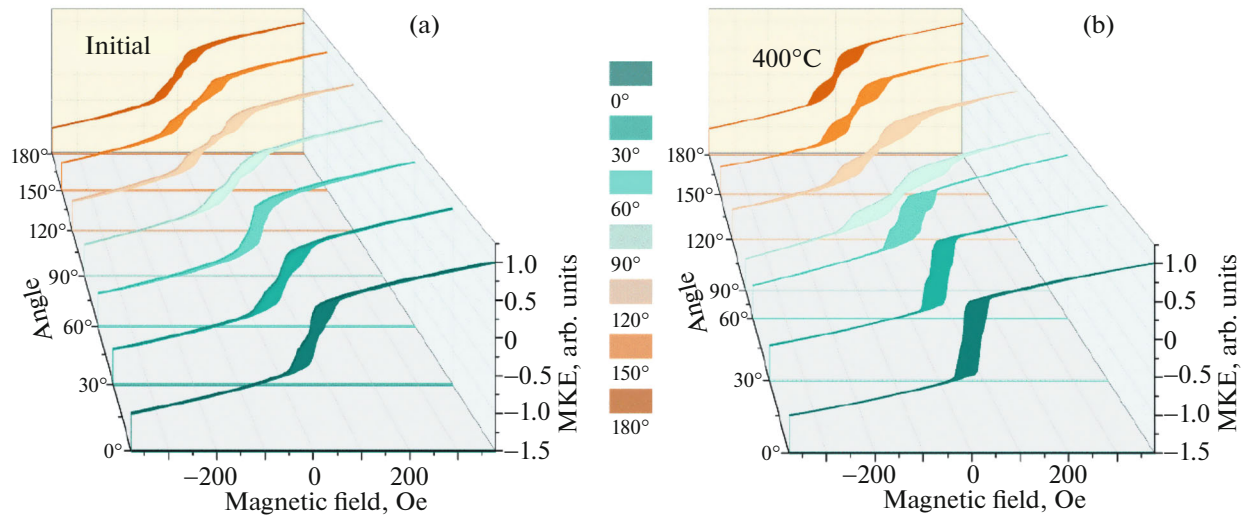


Fig. 3. Near-surface hysteresis loops for $\text{Fe}_{72.4}\text{Ti}_{5.4}\text{B}_{19.2}\text{O}_{3.0}$ films (a) in the initial state and (b) after annealing at 400°C . The rotation angle step is 30° .

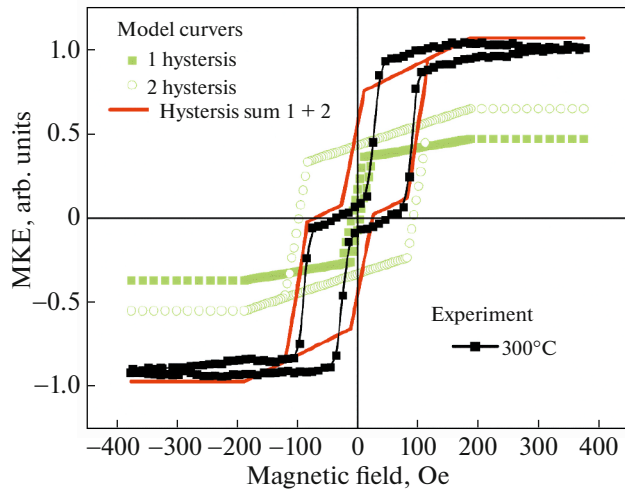


Fig. 4. Model of a two-stage loop as the sum of two hysteresis loops with different coercive forces.

of 20–30 nm. Annealing results in changes in the shape of both bulk and near-surface hysteresis loops, with the near-surface hysteresis loops differing significantly from the bulk ones. First, they are anisotropic (see Figs. 3a, 3b), and second, they exhibit a kink when the field is applied in one of the in-plane directions, whereas it is almost unnoticeable in the bulk loops. Third, their coercive force values differ from those of the bulk loops.

This kinked loop shape can be interpreted as a superposition of two loops with different coercive forces. As an example, Fig. 4 shows the construction of such a hysteresis loop using the method described in [8]. It should be noted that this construction does not take into account for the magnetization of one phase by

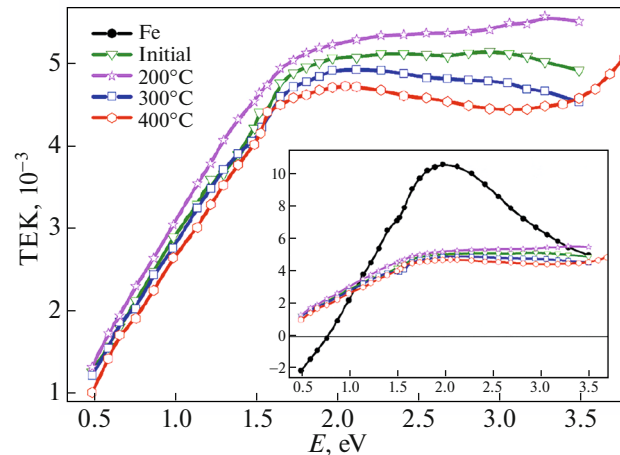


Fig. 5. Spectral dependences of the transverse Kerr effect (TKE) for $\text{Fe}_{72.4}\text{Ti}_{5.4}\text{B}_{19.2}\text{O}_{3.0}$ films in the initial state and after annealing at different temperatures. The inset compares the TKE spectra of nanocrystalline $\text{Fe}_{72.4}\text{Ti}_{5.4}\text{B}_{19.2}\text{O}_{3.0}$ films with that of nanocrystalline Fe.

the other, so it does not fully match the experimentally measured curves but only demonstrates the formation of two phases with different coercive forces.

The presence of a kink in the near-surface hysteresis loops in weak magnetic fields indicates a two-stage magnetization reversal process, suggesting the formation of an additional magnetic phase in these layers. The more pronounced “pinching” of the hysteresis loop in the magneto-optical loops indicates that the formation of the additional phase during film annealing begins from the surface. As shown in [6], annealing results in the partial crystallization of the initially amorphous phase, as well as the redistri-

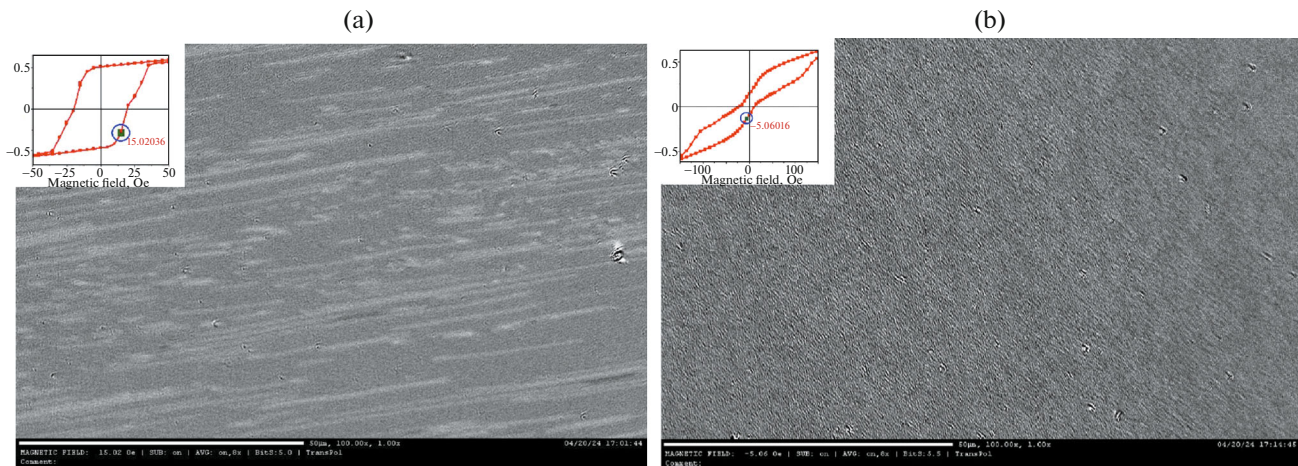


Fig. 6. Visualization of near-surface magnetization reversal of the sample annealed at 400°C using a magneto-optical Kerr magnetometer: (a) sample position corresponds to the nominal 0°, (b) after a 90° rotation. The spot area is 110 × 70 μm.

bution of Ti within the grains and B at the grain boundaries, leading to the formation of an additional phase/phases. This is confirmed by the spectral dependences of the TKE (Fig. 5). As the annealing temperature increases, the TKE spectrum changes, and the peak characteristic of nanocrystalline iron at 2.0 eV becomes more pronounced.

Figure 1 shows that after rapid magnetization in the plane of the film by a field near the coercive force, the magnetization increases monotonically and almost linearly in relatively strong fields. However, the magnetization of nanocrystalline iron reaches saturation in weaker fields. This behaviour suggests the presence of a soft magnetic phase and a phase with perpendicular magnetic anisotropy in the annealed samples. The presence of perpendicular anisotropy explains the stripe domain structure visualized using a Kerr microscope (Fig. 6a). Previously, a stripe domain structure for a similar class of films was observed using a magnetic force microscope [9, 10].

Figures 6a, 6b show the magnetization distribution images in two orthogonal directions. While in one direction, a predominant orientation of stripe domains is clearly visible, in the other direction, a more chaotic structure is observed. This is the reason for the anisotropy observed in the hysteresis loops.

CONCLUSIONS

Magneto-optical studies have shown that the magnetic properties of the near-surface region of $\text{Fe}_{72.4}\text{Ti}_{5.4}\text{B}_{19.2}\text{O}_{3.0}$ films differ significantly from those of the bulk. For the near-surface hysteresis loop, a loop with a pinching effect was obtained, indicating the presence of two distinct magnetic phases. The processes of partial crystallization of the initially amorphous phase and the redistribution

of Ti and B within the crystalline grains and at the grain boundaries, leading to the formation of new phases as a result of annealing, begin at the surface of the films. The presence of a stripe domain structure, which is anisotropic, is confirmed both by magnetic measurements and by direct visualization of the domain structure.

FUNDING

This work was carried out with the financial support of the Russian Science Foundation (grant no. 23-23-00434, <https://rscf.ru/project/23-23-00434>) using equipment purchased with funds from the development program of Lomonosov Moscow State University.

CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

REFERENCES

1. E. N. Sheftel, E. V. Harin, V. A. Tedzhetov, P. V. Kiryukhantsev-Korneev, K. N. Rozanov, S. Y. Bobrovskii, and P. A. Zezyulina, *J. Magn. Magn. Mater.* **561**, 169700 (2022). <https://doi.org/10.1016/j.jmmm.2022.169700>
2. E. N. Sheftel, E. V. Harin, S. Yu. Bobrovskii, K. N. Rozanov, V. A. Tedzhetov, I. O. Bannykh, and Ph. V. Kiryukhantsev-Korneev, *J. Alloys Compd.* **968**, 171981 (2023). <https://doi.org/10.1016/j.jallcom.2023.171981>
3. J. Zhou, J. You, and K. Qiu, *J. Appl. Phys.* **132**, 040702 (2022). <https://doi.org/10.1063/5.0092662>

4. G. Herzer, in *Handbook of Magnetic Materials*, Ed. by K. H. J. Buschow (Elsevier, 1997), Vol. 10, p. 415.
[https://doi.org/10.1016/s1567-2719\(97\)10007-5](https://doi.org/10.1016/s1567-2719(97)10007-5)
5. T. Liu, H. Zhang, F. Kong, A. Wang, Ya. Dong, A. He, X. Wang, H. Ni, and Yo. Yang, *J. Mater. Res. Technol.* **9**, 3558 (2020).
<https://doi.org/10.1016/j.jmrt.2020.01.093>
6. E. N. Sheftel, V. A. Tedzhetov, E. V. Harin, P. V. Kiryukhantsev-Korneev, O. M. Zhigalina, and G. Sh. Usmanova, *Coatings* **14**, 475 (2024).
<https://doi.org/10.3390/coatings14040475>
7. G. Traeger, L. Wenzel, and A. Hubert, *Phys. Status Solidi (a)* **131**, 201 (1992).
<https://doi.org/10.1002/pssa.2211310131>
8. R. V. Lapshin, *Rev. Sci. Instrum.* **91**, 065106 (2020).
<https://doi.org/10.1063/5.0012931>
9. C. B. Craus, A. R. Chezan, M. H. Siekman, J. C. Lodder, D. O. Boerma, and L. Niesen, *J. Magn. Mater.* **240**, 423 (2002).
[https://doi.org/10.1016/s0304-8853\(01\)00882-4](https://doi.org/10.1016/s0304-8853(01)00882-4)
10. N. G. Chechenin, *Magnetic Nanostructures and Their Applications* (GrantViktoriaTK, Moscow, 2006).

Publisher's Note. Allerton Press, Inc. remains neutral with regard to jurisdictional claims in published maps and institutional affiliations. AI tools may have been used in the translation or editing of this article.