

Domain Structure of Magnetic Nanocrystalline Materials Studied by Electron Holography

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Recently, various advanced magnetic materials have been developed by fabricating fine grains in nanometer scale. Fe-based alloys $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ prepared by rapid quenching and annealing at appropriate temperatures show good properties for soft magnets [1]. It was reported that the magnetic properties drastically changed with the change of the annealing temperature or the change of the microstructure [2, 3]. In order to understand the magnetic properties of these materials, it is important to investigate not only the microstructure but also the magnetic domain structure. In this paper, by using the electron holography method, the magnetization distribution of $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ is investigated. Electron holography experiment was carried out with a JEM-3000F transmission electron microscope installed with a field emission gun and a biprism. The magnetic field around the specimen was reduced to 0.2 mT by switching off and then degaussing the objective lens.

Figures 1 (a) and 1 (b) show the coercive force (H_c) and the permeability (μ_r) of $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ with various heat treatments. The lowest coercive force is obtained at the annealing temperature of 823 K. It is also noted that the permeability takes large values at this temperature. Figure 2 (a) - (d) show TEM images of $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ with various heat treatments. While an as-quenched specimen (Fig. 2(a)) is amorphous state, fine grains of about 10 nm and enlarged grains of 50 Å` 200 nm are observed in Figs. 2 (b) and 2 (d), respectively. Figure 3 (a) - (c) show the reconstructed phase images of $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ films with the increase of tilting angle. In the top of Fig. 3 (a) of the as-quenched specimen with no tilt, monotonous closure domains are clearly seen. It is noted that the magnetic lines of force are parallel to the specimen edges, thereby eliminating the surface magnetic charge. It is interesting to note that the shape of the closure domain starts to change at the magnetic field of 8.3 A/m which well corresponds to the coercive force of the bulk specimen of 6.9 A/m (Fig. 1 (a)). On the other hand, it is seen that the magnetic domain of the specimen annealed at 823 K is more sensitive to the magnetic field as seen in Fig. 3 (b), well corresponding to low coercivity and high permeability. In the case of the specimen annealed at 973 K, the size of magnetic domains becomes smaller, and the magnetic lines of force deviate significantly from the monotonous line shape. The irregularity in the shape of the magnetic lines of force is considered to result from the inhomogeneous magnetization distribution due to the bcc Fe and the Fe-B compounds. Being different from Figs. 3(a) and 3 (b), the magnetic lines of force do not change so much for the tilt. The difference directly indicates the strong pinning of magnetic domain walls due to the precipitates, resulting in the drastic increase of the coercive force and the decrease of the permeability [4].

References

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- [4] The author wishes to thank Dr. Y. Yoshizawa and Mr. Y.-G. Park for providing the specimens and for the cooperation.

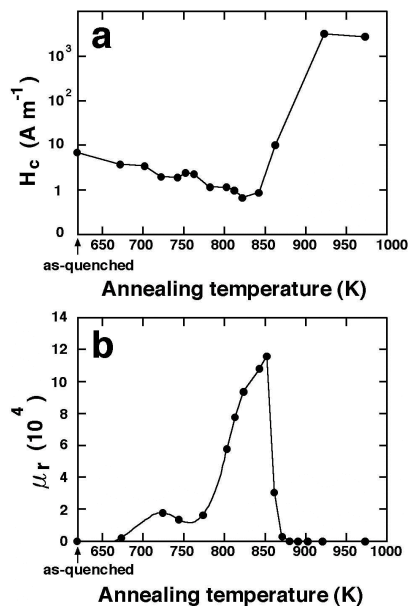


Fig. 1. Coercive force (a), permeability (b) of Fe_{73.5}Cu₁Nb₃Si_{13.5}B₉ as a function of annealing temperature.

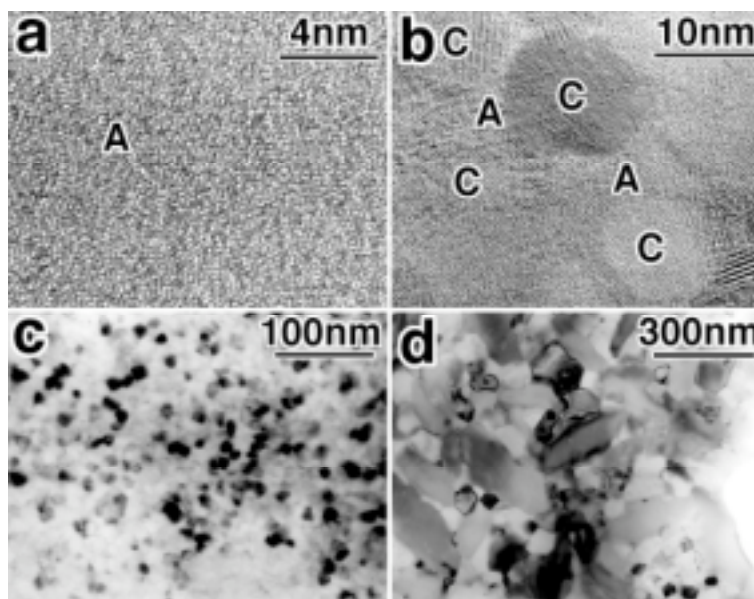


Fig. 2. TEM images of Fe_{73.5}Cu₁Nb₃Si_{13.5}B₉ as-quenched (a), annealed at 823 K (b), 923 K (c) and 973 K (d).

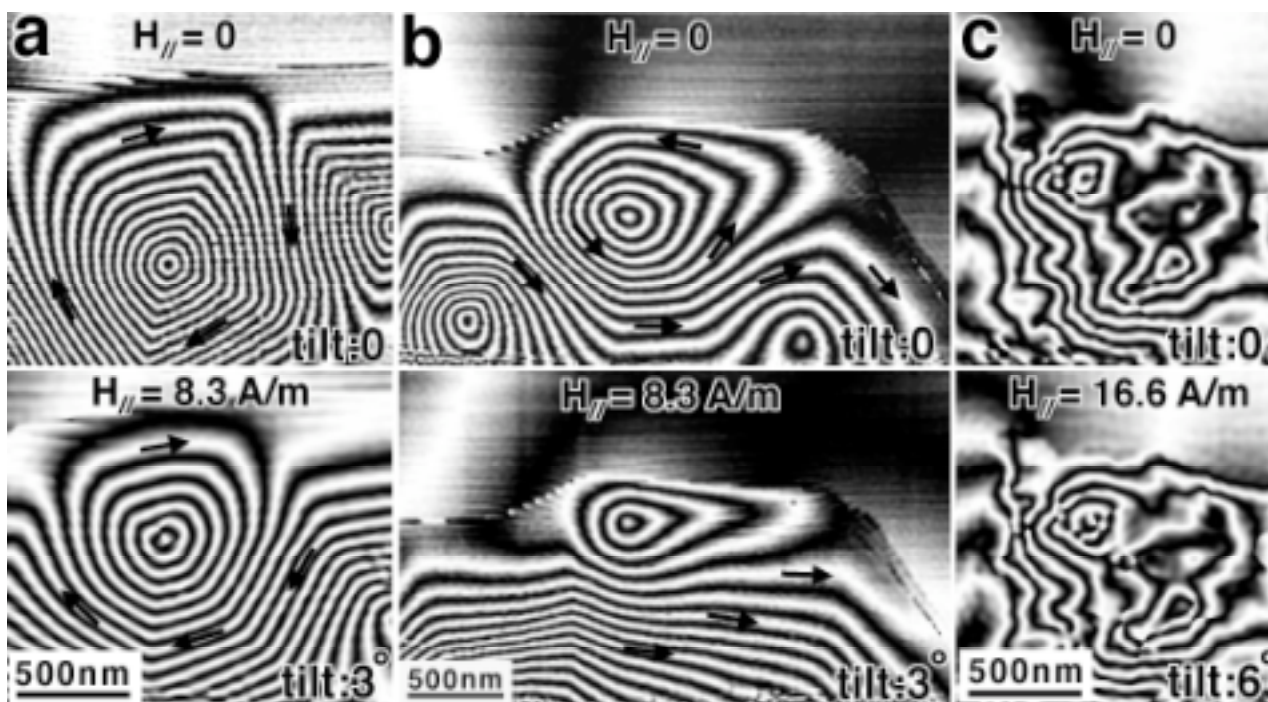


Fig. 3. Reconstructed phase images of Fe_{73.5}Cu₁Nb₃Si_{13.5}B₉ as-quenched (a), annealed at 823 K (b), and 973 K (d) with the increase of tilting angle. The direction of the magnetic lines of force is indicated by arrows. H_y indicates the magnetic field in the film plane due to the tilt.