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INFLUENCE OF FILM THICKNESS ON THE MICROSTRUCTURE AND MAGNETIC PROPERTIES OF FINEMETIC THIN FILMS

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The aim of this work was to study the influence of film thickness on the structure and magnetic properties of finemetic thin films after annealing. Thin films with the various thickness (from 20 nm up to 700 nm) were prepared by DC sputtering method. The heat treatments of the films for further structural and magnetic observations were performed at the temperature range 300–500°C for 15 min in vacuum furnace. Structural observations were carried out by transmission electron microscopy. Coercivity was determined from hysteresis loops traced with fluxmeter and Kerr magnetooptical hysteresisgraph. All the experimental results confirm a different magnetic behaviour of the thin films according to their thickness.

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1. Introduction

It is generally known that in amorphous and crystalline ferromagnetic thin films like permalloy, coercivity increases with decreasing film thickness. This fact can be attributed to preparation related defects, like surface roughness, the relative contribution of which increases with decreasing thickness [1, 2].

In this paper we investigate the influence of film thickness on the structure and magnetic properties of annealed finemet-type thin films.

2. Experimental methods

Thin films of composition $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ of 25 nm, 100 nm, 200 nm, 700 nm thickness were prepared by ion beam sputtering onto glass and freshly cut rock salt (for magnetic measurements and transmission electron microscopy (TEM) observations, respectively) at room temperature using target which was made of about 30 μm thick amorphous ribbons of the same composition. The samples for magnetic measurements were covered by the protective SiO film after film sputtering immediately. In order to change the microstructure the films were

annealed at temperatures 250°C, 300°C, 350°C, 400°C, 450°C, and 500°C for 15 min in vacuum furnace. Structural observations were carried out by TEM. To compare results obtained by surface layer and bulk measurements of coercivity Kerr magnetooptical hysteresisgraph and fluxmeter were used. Dependence of the Hall coefficient R_1 on the annealing temperature was obtained from the Hall curves measured by DC method at room temperature.

3. Results and discussion

TEM confirmed amorphicity of all the prepared samples. No significant influence of the film thickness (up to 100 nm) on their structure was observed. Amorphicity of thicker samples was confirmed by X-ray diffraction (XRD). Figure 1 shows influence of annealing treatment on the film structure. Heat treatment at 350°C (Fig. 1a) starts the partial crystallization of all the samples. It is an initial stage of the crystallization process with low volume fraction of homogeneously dispersed FeSi crystals in amorphous matrix with high nucleation density, similarly as for nanocrystalline finemet-type ribbons. The mean grain size of the crystals is about 10 nm. Figure 1b shows nanocrystalline structure of the samples annealed at 400°C with the presence of crystals of about 30 nm. Annealing at 450°C caused that all the samples were fully crystallized with mean grain size of about 50 nm (Fig. 1c). Annealing at 500°C leads to grain coarsening up to 100 nm and precipitation of borides (Fig. 1d).

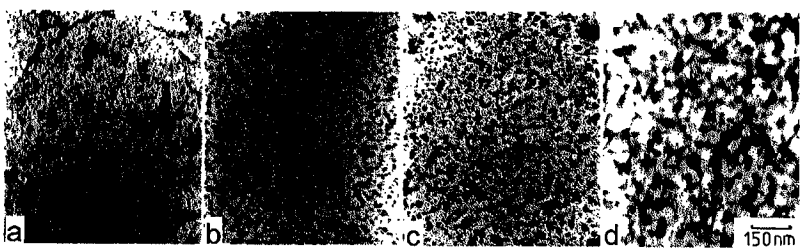

Fig. 1. TEM microphotographs of the samples annealed at (a) 350°C, (b) 400°C, (c) 450°C, and (d) 500°C.

Figure 2a shows the temperature dependence of surface layer coercivity on film thickness. The decrease in coercivity after annealing up to 300°C for all the samples is caused by the structural relaxation processes which leads to elimination of internal stresses induced in the films during their preparation. The minimal value of H_c was observed after annealing at 350°C due to crystallization of FeSi phase, however, low volume fraction of the phase was observed. Slight grain coarsening observed at 400°C caused smooth increase in H_c. Further grain coarsening at 450°C caused steep increase in H_c. It can be explained by the fact that the grains are coupled only in two dimensions because thin thickness and grain size are of the same order of magnitude. This leads to an increase in the magnetocrystalline energy and therefore to higher values of H_c. This process can be seen more clearly in the samples annealed at 500°C with higher grain size [3].

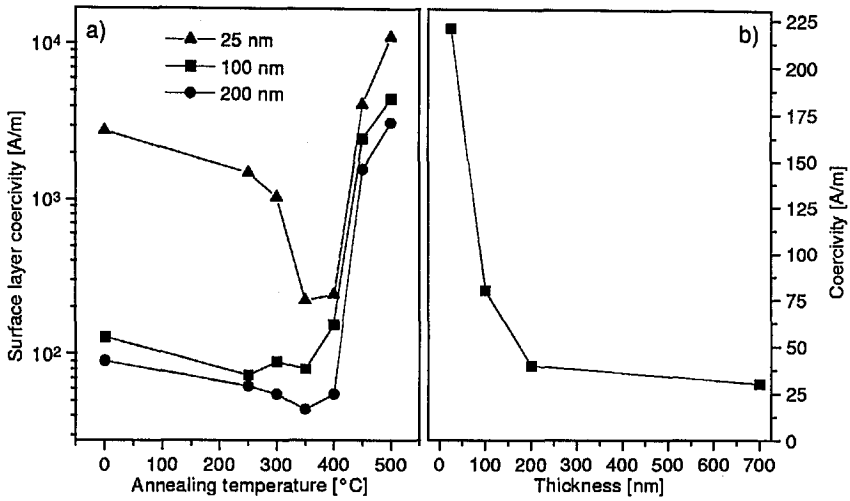

Fig. 2. (a) The dependence of surface layer coercivity on annealing temperature. (b) Thickness dependence of coercivity of the samples annealed at 350°C.

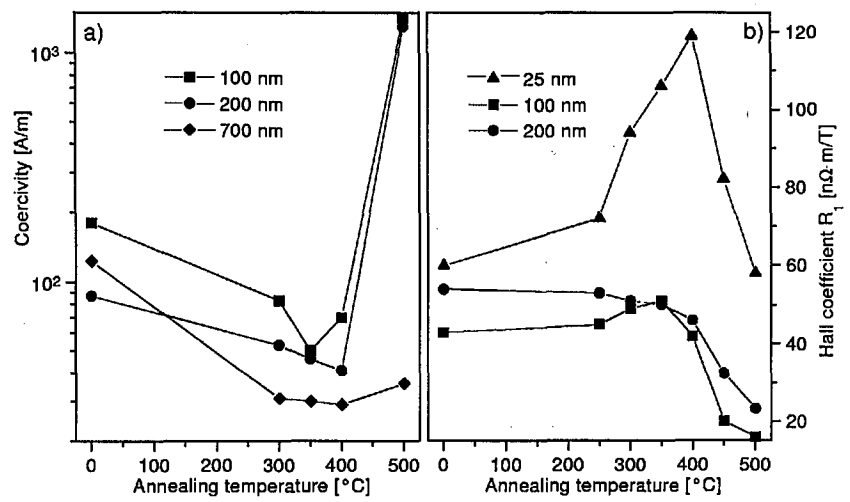

Fig. 3. (a) The dependence of bulk coercivity on annealing temperature. (b) The dependence of the Hall coefficient R_1 on annealing temperature.

Figure 2b illustrates thickness dependence of H_c after annealing at 350°C.

Figure 3a shows the temperature dependence of bulk coercivity on film thickness. It can be seen that surface layer H_c and bulk H_c are almost of the same magnitude for samples up to 100 nm thick. On the other hand, the bulk value of H_c decreases with increasing film thickness and above 200 nm is lower than for surface one. It can be explained by the fact that the Kerr magnetooptical hysteresisgraph gives information from about 50 nm thick surface layer of the sample. Moreover, the values of H_c are significantly influenced by the surface roughness. There is also an interesting fact that value of H_c for 700 nm thick sample does not increase up

to 500°C. It can be attributed to significantly lower value of magnetocrystalline energy because of convenient ratio between thickness and grain size of crystals.

Figure 3b presents the temperature dependence of extraordinary Hall coefficient R_1 for all samples. Initial increase in R_1 could be connected with generation of clusters of crystalline phase [4]. The lower thickness, the more significant increase in R_1. It can be seen that dependence of R_1 for 200 nm sample is already typical as for amorphous ribbons [5].

4. Conclusion

1. No influence of film thickness on crystallization process in the range 25–700 nm after annealing was observed. Crystallization process starts at lower temperature as for finemet ribbon (350°C) and typical nanocrystalline structure was observed at the temperature range 350–500°C but with higher grain size (10–50 nm) as for finemet-type ribbons.

2. It has been proved that grain size and thickness of the films influences the value of magnetocrystalline energy (due to two-dimensional coupling in the case that thickness of the film corresponds with grain size of crystals). The higher thickness, the lower magnetocrystalline energy and value of coercivity, respectively.

3. Hall effect measurements confirmed higher sensitivity for indication of generation of crystallization nuclei (clusters) for very thin films (of about 25 nm).

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