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STRUCTURE AND MAGNETIC PROPERTIES OF Fe/Zr MULTILAYERS

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In this paper we report results of investigation into structure and magnetic properties of as-deposited Fe/Zr multilayers with different wavelength of modulation, λ, and thicknesses ratio of sublayers $r = d_{\mathrm{Fe}}/d_{\mathrm{Zr}}$. Three series of samples with r equal to 0.5, 1, 2 and λ from 12 nm to 1.2 nm were prepared by sputtering technique. The samples were studied by X-ray diffraction and torque curve measurements. It is shown that during deposition amorphization process takes place at interfaces. For nominal Fe sublayer thickness d_{Fe} smaller than 2 nm the multilayers are found to be completely amorphous independently of r, and consist of alternating Fe-rich and Fe-poor amorphous sublayers.

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1. Introduction

Polycrystalline Fe/Zr multilayers undergo a transition to amorphous state through the process of solid state reaction [1]. For multilayers (Mls) of sufficiently small wavelength of modulation this process plays an important role also in as-deposited films [2]. Magnetic measurements performed at room temperature (RT) for Fe/Zr Mls permit determination of the contribution of the ferromagnetic phase, in the case considered — of iron, as amorphous $\mathrm{Fe}_{100-x}\mathrm{Zr}_x$ is nonmagnetic in the whole range of concentration and at RT [3]. We report magnetic measurements performed not only at RT but also in lower temperatures (down to 100 K) which permitted approximate determination of the composition of the formed amorphous phase.

2. Experiment

Fe/Zr Mls were deposited with dc double "face to face" sputtering system. Sequential deposition of elemental iron and zirconium was performed by rotation of the substrate under two sources, which were shielded so that co-deposition was completely avoided. Three series of multilayers were prepared with r equal to 2, 1, 0.5, which corresponds to the mean composition of $\mathrm{Fe}_{80}\mathrm{Zr}_{20}$, $\mathrm{Fe}_{67}\mathrm{Zr}_{33}$,

$Fe_{50}Zr_{50}$. For each series λ was changed from 12 nm to 1.2 nm and repetition of bilayers was increased with decreasing λ to deposit approximately equal amount of Fe for samples within each series.

The structure of the samples was determined by standard Θ–2Θ X-ray diffraction. The wavelength of modulation λ and sublayer thicknesses were determined by small angle X-ray diffraction (SAXRD). The structure of Fe and Zr sublayers was examined by high angle diffraction. The Fe and Zr layer thicknesses were also determined using X-ray fluorescence analysis.

Magnetic properties of the samples were studied with an automatic torque magnetometer in the temperature range 100–300 K. Torque curves were taken in applied field up to 15 kOe, which was rotated in a plane perpendicular to the Mls surface.

3. Results and discussion

Structure of Fe and Zr sublayers in multilayers with large λ was determined by high angle ($20° < 2\Theta < 50°$) X-ray diffraction. Two predominant Fe(110) and Zr(002) Bragg peaks were observed. Both peaks were rather broad indicating a small crystallite size. Intensities of the peaks were diminished with decreasing λ. For multilayers with $\lambda < 4$ nm no crystalline phases were revealed. This is consistent with the result of conversion electron Mössbauer spectroscopy (CEMS) studies presented in the papers [4, 5], where the as-deposited iron was shown to be amorphous in Mls with 10 monolayers thick sublayers of Fe and Zr.

A good structure periodicity of the samples was found by small angle ($1° < 2\Theta < 8°$) X-ray diffraction, which confirmed our technological prediction. Several peaks were typically revealed, which reflects a strong composition modulation in the growth direction (Fig. 1). A number of peaks decreased with decreasing λ, implying intermixing on interfaces. A small angle peak was observed for a sample with $\lambda = 1.2$ nm, which had a nominal Fe sublayer thickness as small as 0.8 nm (Fig. 1, inset). The values of modulation wavelength determined from SAXRD are consistent with these calculated from X-ray fluorescence analysis.

The torque curves obtained for the studied Mls revealed the features typical for thin magnetic films without distinct perpendicular anisotropy as for the films whose hard axis of magnetization is perpendicular to the plane of the film. This result is consistent with those reported by Clemens [5], who proved that magnetic moments of iron in amorphous Fe/Zr Mls lie in the plane of the sample. Thus, the measurements of torque curves should provide the information on the effective shape anisotropy of the Mls. On the basis of obtained torque curves we determined the effective shape anisotropy constant $K_{\text{eff}} = 2\pi M_{\text{eff}}^2$, where M_{eff} is effective magnetization of Fe layer. To be able to determine this constant we have taken the following assumptions: (i) magnetic torque comes from Fe sublayers, (ii) the volume of the magnetic phase is equal to the nominal volume of the deposited iron, (iii) amorphization via the reaction in solid state leads only to a decrease in the nominal thickness of Fe sublayers. The amorphous $Fe_{100-x}Zr_x$ phase under formation could not disturb the measurements as for $10 < x < 55$ its magnetic ordering temperatures T_c are below RT [3]. It should be pointed out that the tem-

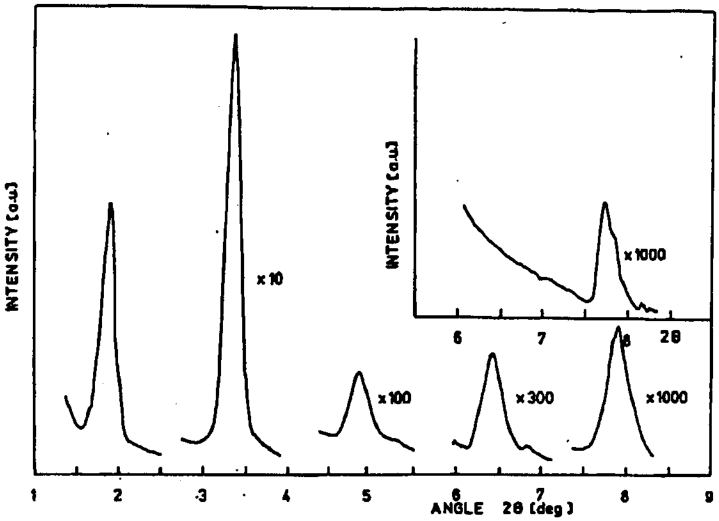

Fig. 1. X-ray spectra for Fe/Zr multilayers of $r = 2$ and $\lambda = 6.4$ nm or $\lambda = 1.2$ nm (inset).

perature dependence of K_{eff} can give information on T_{c} of the formed amorphous alloy as this dependence is analogous to that of $M^2(T)$ commonly reported to be used for determination of T_{c} of amorphous alloys.

Figure 2a presents K_{eff} as a function of the nominal thickness of Fe sublayer, determined at RT. For Mls of $\lambda < 4$ nm a drastic reduction in K_{eff} is seen to occur, independently of r. It should be remembered that for all Mls the total thickness

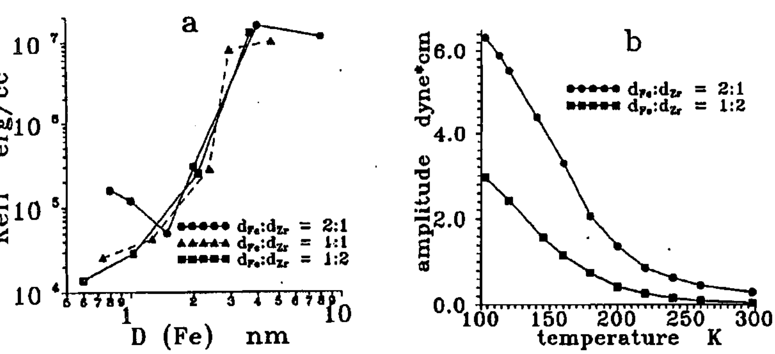

Fig. 2. Effective anisotropy constant K_{eff} as a function of nominal Fe-sublayer thickness d_{Fe} (a) and its temperature dependence (b). K_{eff} is proportional to the amplitude of torque curve. The nominal thicknesses of Fe-sublayers are $d_{\mathrm{Fe}} = 1.5$ nm and 1.3 nm for samples with $r = 2$ and 0.5, respectively.

of Fe sublayers was constant. This result together with the outcome of the X-ray studies indicate that an amorphous Fe–Zr alloy at interfaces is formed during deposition of Mls. As the only contribution to the torque curves comes from the unreacted fraction of Fe, one can suppose that the samples of $d_{Fe} < 2$ nm are almost completely amorphous. In fact, for these samples K_{eff} amounts to only 0.5% of its value for samples of $d_{Fe} > 10$ nm.

For multilayers of $\lambda < 2.2$ nm and $r = 2$ ($d_{Zr} < 0.7$ nm), K_{eff} increases with decreasing λ. It seems that the Ml of $d_{Fe} = 1.5$ nm can be treated as fully amorphous as its K_{eff} is lower than 0.3% of its value expected for unreacted Fe. Further decrease in the thickness of Zr sublayers leads probably to the appearance of discontinued structure which results in the effective increase in the thickness of certain Fe sublayers. As a consequence, a certain amount of Fe remains unreacted and brings a contribution to K_{eff}. Thus, for Mls of $r = 2$ and $\lambda < 2.2$ nm, K_{eff} increases with decreasing λ (Fig. 2a). More detailed information concerning local magnetic structure of the studied films can be obtained from the FMR [6] and CEMS [7] studies.

The above results allow us to conclude that the as-deposited Mls of $d_{Fe} < 2$ nm consist of alternating Fe-rich and Fe-poor amorphous sublayers. Additional confirmation of this conclusion are the results of torque curves measurements as a function of temperature. These measurements were done for Mls of $d_{Fe} = 1.5$ nm ($r = 2$) and 1.3 nm ($r = 0.5$). Figure 2b shows temperature dependence of torque curves amplitude, which is a measure of K_{eff}. It is seen that in both samples an amorphous Fe–Zr alloy is present. The shape of these curves and the fact that we cannot determine one definite T_c indicate that magnetic sublayers show a composition gradient. However, in the cases of both curves we can determine T_c of dominant phase in the temperature range 100–300 K by extrapolation of the linear part of the curve. For curve describing a Ml of $r = 2$, T_c of the dominant phase is 230–240 K which corresponds to the composition $Fe_{75}Zr_{25}$. For curve describing a Ml of $r = 0.5$ T_c of the dominant phase is 180–190 K which indicates the phase $Fe_{70}Zr_{30}$ [3]. In the former Ml the dominant phase has a composition close to the mean composition of the sample: $Fe_{80}Zr_{20}$. In the latter the dominant phase composition is significantly different from their mean one: $Fe_{50}Zr_{50}$. Such substantial difference may be explained as resulted from strong composition inhomogeneity of an amorphous phase formed at interfaces.

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