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STRUCTURE AND MAGNETIC PROPERTIES OF Ni/Ti MULTILAYERS

B. Szymański, F. Stobiecki

Institute of Molecular Physics, Polish Academy of Sciences
Smoluchowskiego 17/19, 60-179 Poznań, Poland

T. Otto and K. Röll

University of Kassel, FB Physik, Kassel, Germany

We report a study of structure and magnetic properties of Ni/Ti multilayers with different modulation wavelength, 10 nm $\leq \lambda \leq$ 40 nm, and constant sublayers thickness ratio $d_{\mathrm{Ni}}/d_{\mathrm{Ti}} = 1$. Specimens were characterized with X-ray diffraction, vibrating sample magnetometer and torque meter in as-deposited state and in successive stages of isothermal annealing at 423 K. Hysteresis curves of as-deposited samples revealed the typical feature of ferromagnetic films with predominant shape anisotropy. During annealing very pronounced changes in the hysteresis loops were observed. An analysis of the loops indicates the occurrence of distinct changes in magnetic anisotropy of Ni sublayers: their effective shape anisotropy is drastically reduced due to a development of the perpendicular anisotropy and/or a modification of the microstructure. The effect can possibly be applied to achieve desirable variation of perpendicular anisotropy in multilayers which consist of Ni sublayers.

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The transition of polycrystalline metallic multilayers (Mls) to amorphous state via solid state reaction (SSR) has been intensively investigated in the last decade (see for an example [1]). It was also reported that magnetic properties of Mls are altered by SSR [2]. In our previous study we found that in Ni/Zr Mls an effective perpendicular magnetic anisotropy arised as a result of isothermal annealing at 455 K [3, 4]. Ni/Ti Mls seems also to be a system of interest because it was demonstrated that dissolution of Ti in Ni occurs simultaneously with formation of the amorphous phase, and this takes place both along the original interfaces and along the intralayer grain boundaries [5]. Moreover, the SSR is associated with development of stresses in Mls [6]. In the present paper the changes in structure and magnetic properties of Ni/Ti Mls upon isothermal annealing at 423 K are studied. The temperature of annealing is chosen very low compared with that mainly used in amorphization experiment ($\approx$ 500–600 K) because we would like to moderate the diffusion processes.

A set of Ni/Ti Mls with $d_{\mathrm{Ni}} \approx d_{\mathrm{Ti}}$ was deposited onto glass substrate by rf sputtering. The modulation wavelength λ was varied from 10 to 40 nm. The samples were annealed at 423 K in high vacuum furnace at a pressure of better than 3×10^{-6} hPa. A good periodicity of the samples was confirmed by small angle X-ray diffraction. Several peaks were typically revealed, reflecting a strong composition modulation in the growth direction. The values of λ, d_{Ni} and d_{Ti} were determined from small angle diffraction and X-ray fluorescence (XRF) method. The structure of Ni and Ti sublayers in Mls was determined by standard θ-2θ, high angle X-ray diffraction (HAXRD). The well pronounced Ni (111) and Ti (002) Bragg peaks were observed for all our Mls. No other peaks were found except the corresponding second order, which indicates strong texturing of Ni and Ti sublayers.

Figure 1 shows HAXRD spectra for as-deposited and annealed Mls with $\lambda = 20$ nm. The intensity of Ni (111) peak is significantly higher than that of Ti (002), suggesting a stronger texture of Ni sublayers. Moreover, small peaks can be seen on the left side of Ni (111) peak. These can be interpreted as the Laue satellites, implying constant Ni-crystallite size in the direction normal to the sample plane. The Laue interference function gives rise to satellites at both sides of the main peak. The observed asymmetry can be explained, according to the model of Hollanders et al. [5], by the changes in the spacing of atomic planes at the top and bottom sides of diffracting crystallites. Such spacing changes can occur due to dissolution of Ti in the Ni crystallites near the Ni/Ti interfaces. As a result, coherency strains develop in the Ni crystallites parallel to the interfaces: compressive stresses occur in the interface-adjacent region, and tensile stresses occur in the center. The development of tensile planar stresses on annealing can also be revealed from the positive shift (about 0.4%) of Ni (111) Bragg peak (Fig. 1), since it suggests a decrease in the lattice spacing of Ni crystallites. The absence of Laue satellites for the Ti (002) peak indicates that the Ti sublayers have less uniform crystallite size distribution in the growth direction than the Ni sublayers. The integrated intensity I_{int} and the integral width $\beta = I_{\mathrm{int}}/I_{\mathrm{max}}$ of Bragg peaks did not change under annealing. It means that volume and the size z of the coherent diffracting crystallites in the growth direction did not change. Since the thickness of sublayers is small ($d \leq 20$ nm) we can assume that the

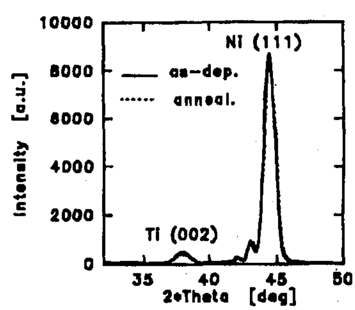

Fig. 1. X-ray diffraction spectra of as-deposited and annealed (11 h at 423 K) multilayers with $\lambda = 20$ nm.

peaks broadening is caused by the small size of crystallites only and Scherrer's law $z = \lambda/(\beta \cos\theta)$ (λ is the wavelength of the X-radiation) can be applied to calculate z_{Ni} and z_{Ti}. For sample with $\lambda = 14.9$ nm (as determined from small angle XRD and XRF) the sum of the calculated $z_{\mathrm{Ni}} = 6.0$ nm and $z_{\mathrm{Ti}} = 4.9$ nm is equal to 10.9 nm, i.e. is about 4 nm smaller than λ. It means that due to the SSR which has already occurred during deposition some part of both sublayers, most probably that adjacent to interfaces, is not textured.

Magnetic properties of the Mls were measured with a vibrating sample magnetometer (VSM) and a torque magnetometer. Measurements were performed for as-deposited samples and after different stages of annealing. The results are shown in Figs. 2 and 3. Hysteresis curves and their changes on annealing are qualitatively similar to that of Ni/Zr Mls [4]. As follows from the linear extrapolation of magnetic moment of as-deposited Mls as a function of the λ^{-1} [3], about 3.2 nm of the nominal thickness of the Ni sublayers is nonmagnetic. It can also be noted (Fig. 2) that anisotropy field H_k of as-deposited Mls is unexpectedly larger than $4\pi M_s = 6100$ Oe, indicating, at variance with [5] the presence of a certain compressive stress in Ni sublayers. After annealing the pronounced changes in magnetic moment and H_k were found. The greatest variation of those parameters took place on the early stage of annealing despite the low annealing temperature. The de-

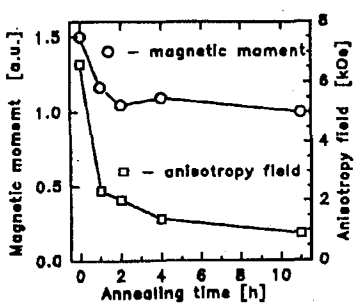

Fig. 2. Magnetic moment and anisotropy field changes on annealing at 423 K.

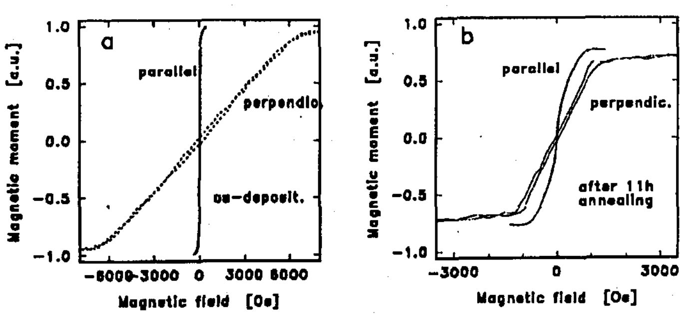

Fig. 3. VSM hysteresis loops for as-deposited (a) and annealed (b) Mls ($\lambda = 14.9$ nm) measured in the magnetic field applied parallel and perpendicular to the film surface.

crease in H_k could be qualitatively explained as a consequence of tensile stress in Ni sublayers, which develops on annealing. However, the alteration of H_k amounts to 5.5 kOe and is too large to be exclusively caused by tensile stress. The H_k is the sum of three contributions: (i) the shape anisotropy field $H_{\rm sh} = (D_\perp - D_\parallel)M_s$, where $D_\perp$ and $D_\parallel$ are the demagnetization factors in direction perpendicular and parallel to the sample plane, respectively ($D_\perp - D_\parallel = 4\pi$ for continuous Ni sublayers); (ii) the stress-induced anisotropy field $H_\sigma = 3\lambda_{111}\sigma/M_s$, where $\lambda_{111} = -29.5 \times 10^{-6}$ is magnetostriction constant of Ni; (iii) the magnetocrystalline anisotropy field $H_{\rm mk} = 2K_1/M_s$, where $K_1 = -4.9 \times 10^4$ erg/cm^3. $H_{\rm mk} \approx -0.2$ kOe and could be neglected as caused by texture which does not change appreciably on annealing. The second term H_σ may attain a value of about -3.5 kOe if one assumes the extreme value of tensile stress $\sigma = 2 \times 10^{10}$ dyne/cm^2 [7]. Thus $H_{\rm sh}$ should decrease from 6.1 kOe to about 4.5 kOe on annealing to explain the observed value of $H_k \approx 1$ kOe. The change in $H_{\rm sh}$ is assumed to be related to some modification of the microstructure of Ni sublayers. On annealing, they have converted into quasi-layers which consist of ferromagnetic grains separated by nonmagnetic boundaries. Most probably the modification of microstructure is caused by grain boundary diffusion of both Ni and Ti. It could also account the decrease in magnetic moment on annealing because the magnetic moment of Ni decrease very rapidly when alloying with Ti [8].

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