Technical Physics Letters, Vol. 29, No. 4, 2003, pp. 263–266. Translated from Pis'ma v Zhurnal Tekhnicheskoĭ Fiziki, Vol. 29, No. 7, 2003, pp. 1–9.

The Magnetic Structure of Ferromagnetic Filaments of a CoNi(P) Alloy in a Porous Silicon Matrix

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Received October 8, 2002

Abstract—The magnetic and resonance properties of CoNi(P) alloys, synthesized by chemical deposition as films on single crystal silicon substrates and as filaments in linear pores of porous silicon substrates, were studied by magnetization and ferromagnetic resonance measurements. It is established that CoNi(P) alloys of the same composition but different morphologies occur in states characterized by different degrees of nonequilibrium, which is manifested by different modes of the magnetization approach to saturation. © *2003 MAIK "Nauka/Interperiodica".*

In the past decade, there has been extensive development of the technology of novel magnetic materials representing magnetic filaments formed through chemical deposition of fine particles possessing desired properties inside linear pores of a matrix, for example, porous silicon (por-Si) [1]. Investigation of the structural features of such objects by conventional diffraction methods is rather difficult, because a small volume fraction of the magnetic filaments in a composite sample makes the integral diffraction measurements insufficiently informative. For this reason, it is a usual practice to study the structure of single filaments (isolated from the matrix) by means of transmission and scanning electron microscopy [2, 3]. Experimental investigation of the micromagnetic structure of ferromagnetic filaments in nonmagnetic matrices encounters analogous difficulties. This probably accounts for the fact that, to our knowledge, no experimental data on the magnetic structure of such filaments have been reported so far.

In recent years, the crystal structure and magnetic microstructure of nanocrystalline materials, amorphous alloys, and ensembles of small particles are frequently studied by an indirect method based on the analysis of magnetization approach to saturation [4–7]. We have used this magnetostructural approach to study the magnetic and resonance properties of CoNi(P) alloys chemically deposited as films on single crystal silicon plates and as filaments in linear pores of por-Si substrates. This investigation allowed us to compare the peculiarities in magnetic response related to both geometric shape and structural features of the ferromagnetic material.

The layers of porous silicon were obtained by anodic dissolution in aqueous hydrofluoric acid solutions. Variable parameters included the $HF–H_2O$ solution composition (component volume ratio $x : y$), current density (j = 5–30 mA/cm^2), and anodizing time (t = 10–180 min) at a constant applied dc voltage (U = 10 V). The resulting porous structure was studied in an optical microscope (JENAVERT, Germany) at a magnification of x_{250} and x_{630}. Figure 1 shows the microphotographs of two porous samples cleaved upon the electrochemical treatment in different regimes. These substrates were used for depositing ferromagnetic CoNi(P) filaments. The images reveal well-defined cylindrical pores perpendicular to the sample surface, with a diameter of 1–2 μm and a length (depth) of 10–100 μm.

Ferromagnetic filaments of a CoNi(P) alloy were grown in the pores of silicon plates by means of a heterogeneous reaction of metal reduction from an aqueous solution of cobalt ($CoSO_4$) and nickel ($NiSO_4$) salts under the action of a reducing agent—sodium hypophosphite (NaH_2PO_2) at T = 80°C. Reference films were prepared by chemical deposition from the same system onto a polished single crystal silicon surface. A comparison of the samples obtained on porous and single crystal silicon substrates allowed us to reveal the features of magnetic response related to geometric shape of the ferromagnetic material.

The magnetic properties of the ferromagnetic filaments and planar layers synthesized as described above were studied using the ferromagnetic resonance (FMR) and magnetization techniques. The measurements were performed on an EPA-2M spectrometer operating at a frequency of f = 9.2 GHz and on a vibrating-sample magnetometer in a range of magnetic fields up to 10 kOe.

The magnetization curves and the FMR spectra obtained for various experimental geometries (Figs. 2 and 3) revealed a magnetic anisotropy in the direction of linear pores (oriented perpendicularly to the silicon plate surface). The FMR data allowed the nature of this

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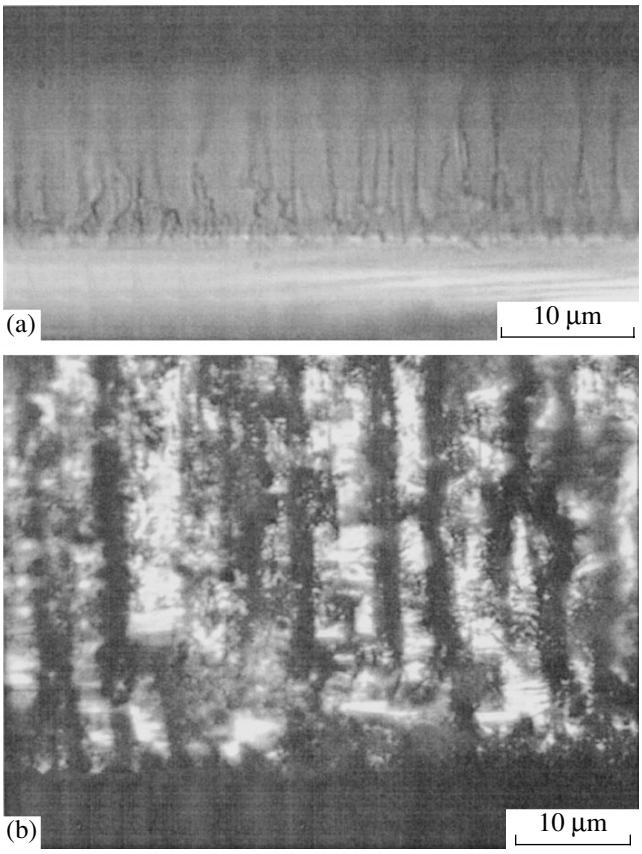

Fig. 1. Microphotographs of cleaved silicon plates upon anodization in an HF–H_2O (1 : 1) solution for 60 min at U = 10 V: (a) j = 64 mA/cm^2 (highly doped silicon); (b) j = 105 mA/cm^2.

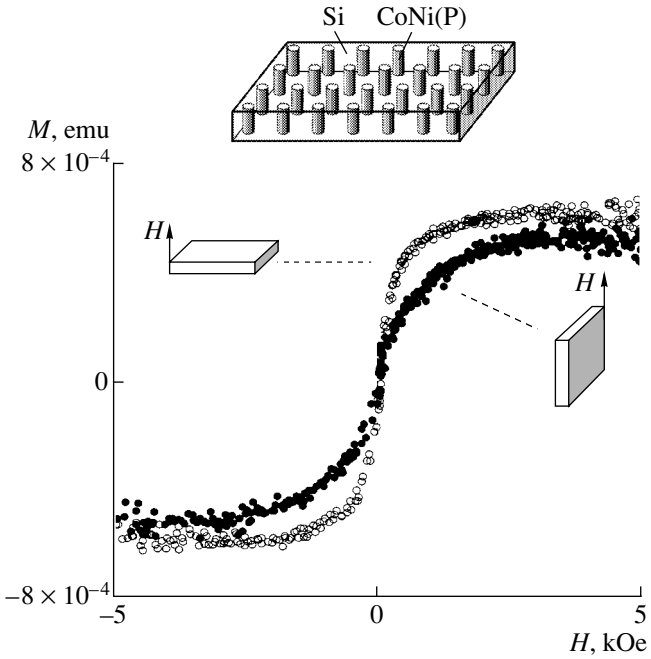

Fig. 2. Magnetization curves of the samples with ferromagnetic CoNi(P) filaments in a por-Si matrix (the inset on top shows a schematic diagram of the system).

anisotropy to be identified as the shape anisotropy. Figure 3 presents the FMR spectra of magnetic filaments of a CoNi(P) alloy in por-Si in comparison to the spectra of films of the same alloy on single crystal substrates, measured in different geometries. As can be seen, the two kinds of samples are characterized by significantly different values of the resonance fields H_r observed for the same material, substrate orientation, and applied field strength. This is explained by the material occurring in different morphological modifications.

The resonance field strength H_r is given by the formula [8]

$$\omega_H = |\gamma|\{[H_r + (N_x - N_z)M_z][H_r + (N_y - N_z)M_z]\}^{1/2}, \quad (1)$$

where $|\gamma|$ = 2.8 MHz/Oe, M_z is the effective magnetization and N is the demagnetizing factor related to the shape of the ferromagnetic material. Substituting the experimental resonance fields into this formula with $N_x = N_y = 0$, $N_z = 4\pi$ for the field **H** perpendicular to a CoNi(P) film and $N_z = N_y = 0$, $N_x = 4\pi$ for the field **H** parallel to the film, we determined the effective magnetization of the alloy studied: M_z = 450 G. Using this experimental value, we calculated the demagnetizing factor N for the CoNi(P) alloy synthesized in porous silicon. Calculated by formula (1) under a natural assumption that $N_x = N_y = N \neq N_z$, this value has proved to be close to 2π, that is, to the demagnetizing factor of the ideal ferromagnetic cylinder (see the inset in Fig. 3). This result indicates that a CoNi(P) alloy synthesized in por-Si represents an ensemble of well-defined ferromagnetic filaments possessing a cylindrical shape. Using specially prepared films of the same CoNi(P) composition, we have measured the spin wave resonance (SWR) spectrum for the field **H** perpendicular to the film plane and, using standard formulas [8], calculated the exchange coupling constant: $A = 0.3 \times 10^{-6}$ erg/cm.

The characteristics of the magnetic anisotropy and magnetic microstructure of CoNi(P) alloys with different morphologies were studied by methods described in [6, 9, 10]. Following an approach developed in these papers, we measured the curves of magnetization to saturation and analyzed their shapes, mostly for the external field oriented parallel to the easy axis of a sample (in order to exclude the influence of the form factor).

The magnetization approach to saturation in the CoNi(P) alloy film deposited onto a silicon plate obeys the law $M \sim H^{-2}$ (Fig. 4). This behavior is characteristic of polycrystalline magnetic materials, which allowed us to describe the experimental magnetization curve by the Akulov formula [11]

$$\frac{M(H) - M_s}{M_s} = \left(\frac{D^{1/2}H_a}{H}\right)^2, \quad (2)$$

where M_s is the saturation magnetization and H is the magnetic field strength. Using this formula, it is possible to determine the rms fluctuation of the local anisotropic magnetic field: $D^{1/2}H_a = 600$ Oe.

For the CoNi(P) filaments in por-Si, the magnetization approach to saturation in the range from 1 to 5 kOe obeys the law $M \sim H^{-1/2}$ (Fig. 4). This power law is typical of the amorphous and nanocrystalline magnetic materials. Indeed, a necessary condition for this asymptotic behavior is $R_c < \delta = (A/K)^{1/2}$ [6], where R_c is the correlation radius of local anisotropy (in nanocrystalline alloys, $2R_c$ approximately equals the grain size) and $\delta = 200$–400 Å (in magnetic alloys of 3*d* metals). According to this estimate, the grain size of a CoNi(P) alloy in the synthesized magnetic filaments does not exceed a few hundred Ångströms and, hence, the material occurs in a nanocrystalline state. Since the filament diameter equals the pore diameter and amounts to 1–2 μm, which is much greater than the grain size, the $M(H)$ data suggest that ferromagnetic filaments in the linear cylindrical pores of por-Si represent a three-dimensional package of nanodimensional grains. Indeed, according to [10], the magnetization of a three-dimensional system of exchange-coupled grains must approach saturation according to the law $M \sim H^{-1/2}$, precisely as is observed in experiment.

Describing the magnetization approach to saturation by the formula [6]

$$\frac{M(H) - M_s}{M_s} = \left(\frac{D^{1/2}\langle H_a\rangle}{H}\right)^{1/2}, \qquad (3)$$

we can determine $D^{1/2}\langle H_a\rangle$, the rms fluctuation of the local anisotropic magnetic field of a block of exchange-coupled nanocrystalline grains. For the CoNi(P) alloy studied, this value has proved to be $D^{1/2}\langle H_a\rangle = 64$ Oe. This parameter characterizes the magnetic microstructure of the nanocrystalline alloy and can be used to estimate the size of a homogeneous magnetization region (magnetic block) in the filaments by the formula $R_f = (2A/D^{1/2}\langle H_a\rangle M)^{1/2}$ [6]. Using the exchange coupling constant of the CoNi(P) alloy determined from the SWR data, we obtain an estimate of $R_f = 460$ Å. This vale is much smaller than the magnetic filament diameter (~1 μm).

Thus, we have established that the synthesized CoNi(PO) alloys of the same composition but different morphologies (a film on single crystal silicon substrate versus filaments in por-Si) are characterized by different laws of the magnetization approach to saturation. This is explained by different degrees of defectness of the material structure. The ferromagnetic filaments in por-Si are characterized by a correlation radius of local anisotropy (R_c) on the order of a few hundred Ångströms, while the film material has R_c at least one order of magnitude greater and, hence, occurs in a state characterized by a much lower degree of nonequilibrium.

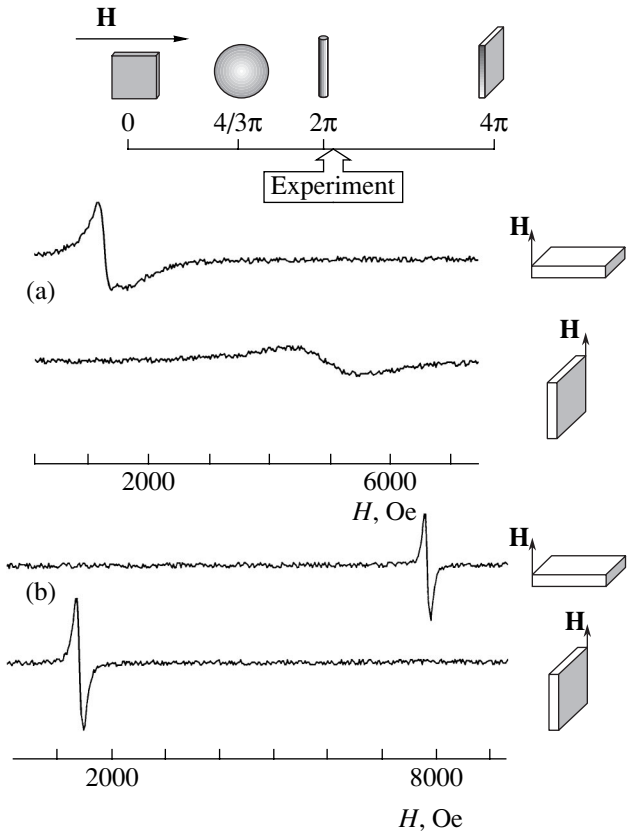

Fig. 3. FMR spectra of a CoNi(P) alloy in the form of (a) ferromagnetic filaments in a por-Si matrix and (b) a film on single crystal silicon substrate measured in various geometries.

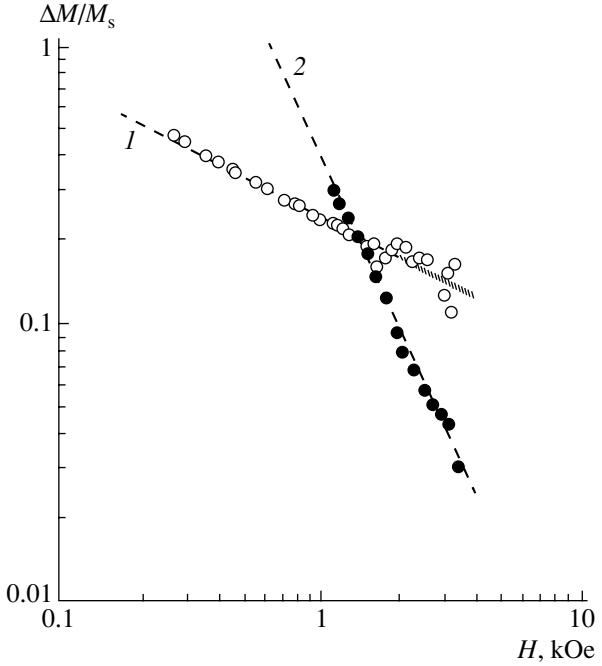

Fig. 4. The curves of magnetization to saturation (plotted in logarithmic coordinates) for a CoNi(P) alloy in the form of (*1*) ferromagnetic filaments in a por-Si matrix and (*2*) a film on single crystal silicon substrate.

In conclusion, note that the obtained composite medium, comprising ferromagnetic filaments in the linear pores of por-Si, must exhibit a specific response to the applied magnetic field. The results of investigation of the electrical and galvanomagnetic properties of this material is the subject for a separate publication.

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Translated by P. Pozdeev