

Atomic force microscopy measurements of magnetostriction of soft-magnetic films

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Abstract. A method for direct measuring the magnetostriction of ferromagnetic films (deposited on nonmagnetic substrates) in using an atomic force microscope was suggested. In measuring the magnetostriction for films ≥ 10 [mm] in length and $\geq 0,2$ [μm] in thickness, which were deposited on substrates ≤ 200 [μm] thick, the minimum measured magnetostriction magnitude is $\sim 10^{-7}$. The procedure was tested for Ni and Fe films. The magnetostriction magnitudes measured for the films are comparable with those obtained by other magnetostriction-measuring methods. The effect of alloying with zirconium and nitrogen on the magnetostriction of nanocrystalline Fe films was studied.

Introduction

As is known, static magnetic properties of ferromagnets are determined by the effective magnetic anisotropy. To reach the minimum coercive force of a material, its magnetic anisotropy must be decreased to a minimally possible magnitude. The more substantial contribution in the magnetic anisotropy is related to the magnetocrystalline anisotropy whose decrease in nanocrystalline materials is considered in terms of a random anisotropy model [1]. In the case of maximally possible decrease in the magnetocrystalline anisotropy, the principal contribution in effective magnetic anisotropy is related to the magnetoelastic anisotropy, which is the interaction between the magnetostrictions of ferromagnetic phases and mechanical stresses in the material. To control the magnetoelastic anisotropy and determine its contribution to the effective magnetic anisotropy, the magnetostriction magnitude should be measured by direct methods.

During magnetization of a ferromagnetic film located on a nonferromagnetic substrate, elastic stresses induced by magnetostriction are formed in the film. Depending on the magnetostriction sign, they may be tensile or compressive. In this case, since no magnetostriction stresses are induced in the substrate, its deformation is realized as a deflection in the system “film-substrate”. The relation between the substrate deflection and stresses induced in a film is described by a Stoney equation [2]. Conditions satisfied to the Stoney equation are analyzed in many works. Among them are in particular [3], in which the analysis was performed for magnetostrictive stresses of a ferromagnetic film located on a nonmagnetic substrate.

The simplest case of strained state of the system “film-substrate” is realized in satisfying the following conditions: (1) the film thickness is less substantially than the substrate thickness ($h_f/h_s \leq 0.01$) and (2) the film and substrate have equal dimensions and are shaped in the form of elongated rectangle (with length l and width w ; $w/l \leq 0.5$) whose one of short sides is mounted as a cantilever (Fig. 1). In this case, in changing the film magnetization, the deflection of free end of substrate is related to the magnetostriction along applied field direction (λ) by Eq. 1 [3]:

$$\lambda = \frac{D}{3} \frac{h_s^2}{h_f l^2} \frac{E_s / (1 + \frac{l}{l+w} \nu_s)}{E_f / (1 + \nu_f)}, \quad (1)$$

here D is the deflection at a distance of l from the fixed end of substrate with width w ; h_f and h_s are the thickness of film and substrate, respectively; E_f and E_s are the Young's modulus of the film and substrate, respectively; and ν_f and ν_s are Poisson's ratios of the film and substrate, respectively.

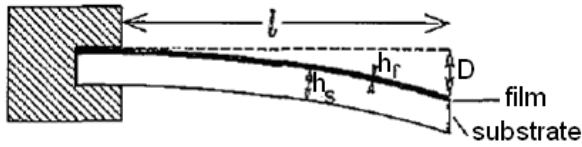


Fig. 1. Cantilever geometry in measuring the magnetostriction of films on substrates

To ignore the magnetic prehistory and magnetic texture of the sample, the magnetostriction magnitude measured for film samples along the magnetization direction is determined by Eq. 2:

$$\lambda = \frac{2}{3}(\lambda_{\parallel} - \lambda_{\perp}) \quad (2)$$

here $\lambda_{\parallel}$ and $\lambda_{\perp}$ are magnetostrictions calculated by Eq. 1 for the film subjected to in-plane magnetization in parallel and perpendicular to the long side of film, respectively.

The deflection D of substrate can be measured by different methods that are generated under the title cantilever technique. The deflection is determined, in particular, from changes in the capacity of air capacitor comprising a film under study as one of capacitor plates, from changes in the angle of reflection of laser beam directed to the free end of substrate, by interference probe or nanoindentation probe [4]. To perform measurements for bulk materials (free from substrate), such as ribbons and wires, an atomic force microscope is used [5].

It is necessary to note that surface magnetostriction is not taking into account for films with thickness more than 50 [nm] [6]. For nanocrystalline ferromagnetic films the grain boundary area magnetostriction could be regarded as the surface magnetostriction [7]. In this case the surface magnetostriction is determined by extension of grain boundary area that is to say by grain size.

The present study is aimed at the development of cantilever technique which is based on the use of atomic force microscope for measuring the magnetostriction of soft-magnetic films located on a nonferromagnetic substrate and application of the developed technique for the study of the effect of alloying of nanocrystalline Fe-based films with zirconium and nitrogen on their magnetostriction.

Experimental

For the investigation the films of different compositions were prepared. Nickel and iron films (used for the testing of the technique) and Fe-N films were prepared by dc reactive magnetron sputtering of Ni and Fe targets. Films Fe-Zr and Fe-Zr-N were prepared by rf reactive magnetron sputtering of the Fe-5 [at.%] Zr target. The following preparation conditions were used all the films: the preliminary vacuum in the chamber is $P_0 = 2 \cdot 10^{-6}$ [Torr] and argon pressure is $P_{Ar} = 3.5 \cdot 10^{-3}$ [Torr]. The nitrogen-containing films were prepared at a nitrogen pressure $P_{N_2} = 2.5 \cdot 10^{-4}$ [Torr]; the nitrogen content in the gas mixture is 6.5%. All the films were deposited on glass substrates 200 [μm] thick (h_s) 14x4 [mm^2] in size. The nitrogen content in the films was analysed by means EDX; the films thicknesses were determined using an interference-contrast microscope (Table 1). The phase composition and structure of the films were studied by X-ray diffraction analysis using a DRON-3 diffractometer and $\text{CuK}\alpha$ radiation. X-ray diffraction patterns were analyzed using specific program software. The grain size of phases formed was determined from the physical broadening of X-ray reflections (Table 1). Roughness of the film surface was measured by means a NT-MDT Smena atomic force microscope: average height of the relief is about 13 [nm], maximum one is up to 30 [nm].

Table 1. Chemical composition, thickness, phase composition and structure of films under study

Chemical composition [at.%]	Film thickness h_f [μm]	Phase composition [vol.%]	Lattice parameters [$\text{\AA}$]	Grain size [nm]
Ni	0.18	fcc-Ni – 100	$a = 3.5163 \pm 0.0002$	10 ± 1
Fe	0.37	bcc-Fe – 100	$a = 2.824 \pm 0.003$	39 ± 11
Fe – 90 N – 10	0.3	bcc-Fe(N) ~ 95 Fe ₂ N ~ 5	$a = 2.8732$ $a = 2.75877; c = 4.42911$	15 78.2
Fe – 95; Zr – 5	0.37	bcc-Fe(Zr) – 100	$a = 2.857 \pm 0.008$	34 ± 3
Fe – 85; Zr – 5 N – 10	0.5	bcc-Fe(N,Zr) ~ 93 Fe ₂ N ~ 7	$a = 2.899 \pm 0.007$ $a = 2.73752; c = 4.56028$	11 ± 1 35

The deflection of substrates during the film magnetization was measured using a NT-MDT Smena atomic force microscope. The films were magnetized in fields up to 80 [Oe] using a specially designed attachment produced the magnetic field. The deflection of substrate D was determined by analyzing topography images. During scanning the film surface, the magnetic field was applied periodically. In this case, decrease in the relief height corresponds to the positive magnetostriction of the film; increase in the relief height corresponds to the negative magnetostriction of the film.

Results and discussion

The substrate deflection magnitudes D measured for each of the films under study were used to calculate $\lambda_{\parallel}$ and $\lambda_{\perp}$ by Eq. 1. The Young's modulus of the Ni film was taken to be $E_f = 200$ [GPa]; the Young's modulus for Fe, $\text{Fe}_{95}\text{Zr}_5$, $\text{Fe}_{90}\text{N}_{10}$, and $\text{Fe}_{85}\text{Zr}_5\text{N}_{10}$ films is $E_f = 142$ [GPa] (determined by nanoindentation [8]). The Young's modulus of the substrate is $E_s = 60$ [GPa] [8]. The Poisson's ratios of the film and substrate are $\nu_f = 0.3$ and $\nu_s = 0.22$, respectively. The $\lambda_{\parallel}$ and $\lambda_{\perp}$ were used to calculate the magnetostriction λ (by Eq. 2) in different magnetic fields applied values. Errors of measurements were calculated statistically using experimental data array. Fig. 2 shows results of the measurements of $\lambda_{\parallel}$, $\lambda_{\perp}$ and λ magnitudes for the Ni film. The magnitude $\lambda \approx -10 \cdot 10^{-6}$ obtained in an applied field of 80 [Oe] agrees with data of [9], in which a magnitude of $-4 \cdot 10^{-6}$ was obtained for Ni films in an applied field of 80 [Oe] that was not saturation field as well as in our case.

It should be noted that in measuring by cantilever techniques, the accuracy of the measurement of the Young's modulus, Poisson's ratios and film and substrate thicknesses (i.e., magnitudes entering into Eq. 1) affects additionally the direct measured magnetostriction. In particular, the Young's modulus of glass is from 50 to 80 [GPa] and its Poisson's ratio is from 0.16 to 0.25.

Fig. 3 shows the magnetostriction λ of the Fe, $\text{Fe}_{95}\text{Zr}_5$, $\text{Fe}_{90}\text{N}_{10}$ и $\text{Fe}_{85}\text{Zr}_5\text{N}_{10}$ films, which was measured in fields (up to 80 [Oe]) below the saturation field. As is seen, the nonalloyed Fe film exhibits the greatest λ . The alloying with Zr decreases substantially the magnetostriction of Fe film. This result agrees with literature data [10]. The alloying with nitrogen leads to the sign alternation (from plus to minus) of λ and a decrease in its value. These results agree with data of [11]. The authors of [11] showed that, as the nitrogen content in a Fe film increases, the magnetostriction of the film alternates the sign; zero magnetostriction corresponds to a nitrogen content of ~ 6 [at.%] [11]. Simultaneous alloying by Zr and N results in decrease of the Fe film magnetostriction as well, but smaller extent than each element individually (Fig. 3). The observed effect from the alloying arises due to formation of proper ferromagnetic solid solution, $\alpha\text{-Fe}(\text{Zr},\text{N})$, $\alpha\text{-Fe}(\text{N})$ or $\alpha\text{-Fe}(\text{Zr})$ higher strength than $\alpha\text{-Fe}$, and diminution of their grain size (Table 1).

Remarkable that a separate contribution from magnetostriction of the grain boundary area and the grain body [7] in the films magnetostriction was not examined in this investigation.

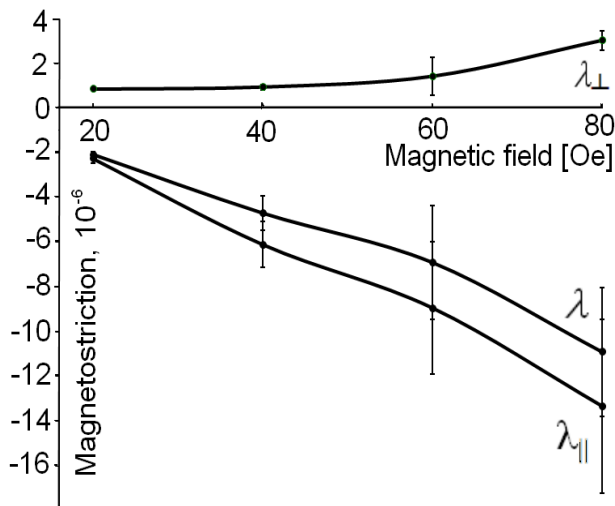


Fig. 2. Magnetostriction of Ni films

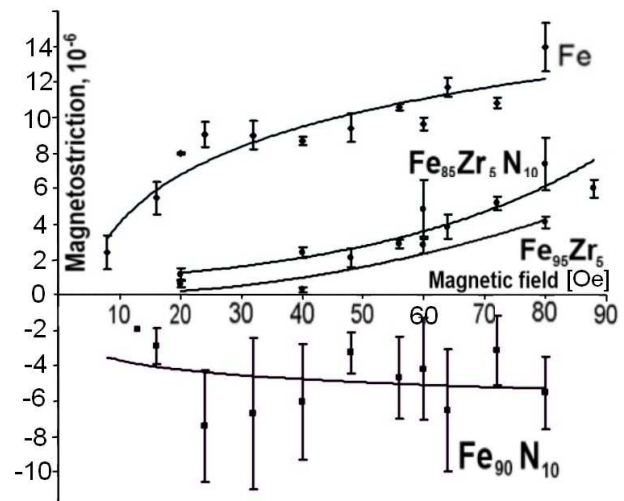


Fig. 3. Magnetostriction of the Fe, $\text{Fe}_{95}\text{Zr}_5$, $\text{Fe}_{90}\text{N}_{10}$ and $\text{Fe}_{85}\text{Zr}_5\text{N}_{10}$ films

The results discussed above illustrate, that the alloying of nanocrystalline Fe films with zirconium and nitrogen result in the magnetostriction decrease. This effect side by side with the decrease of the nanograin size (Table 1), promoting in it's turn of the magnetocrystalline anisotropy diminishing [1], should improve soft magnetic properties of Fe-Zr-N films.

Conclusions

A new cantilever method based on the use of atomic force microscope for measuring the magnetostriction of ferromagnetic films deposited on nonferromagnetic substrates was developed; up to our best knowledge this method is suggested at the first time. The minimum deflection D of the substrate recorded with atomic force microscope used is ~ 1 [Å]; for the samples under study, the magnitude corresponds to an accuracy of the magnetostriction measurement of $\sim 10^{-7}$.

The magnetostriction of Ni, Fe, Fe₉₅Zr₅, Fe₉₀N₁₀ and Fe₈₅Zr₅N₁₀ films deposited by magnetron sputtering on nonferromagnetic substrates was estimated in fields of up to 80 [Oe] by this method. It was shown that the alloying of nanocrystalline Fe films with zirconium and nitrogen results in the magnetostriction decrease.

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