# UHV Cantilever Beam Technique for Quantitative Measurements of Magnetization, Magnetostriction, and Intrinsic Stress of Ultrathin Magnetic Films

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A new method—based on the cantilever beam principle—is presented, by means of which quantitative values of the magnetization, magnetostriction, and intrinsic stress of magnetic thin films can be determined. Moreover investigations of magnetic anisotropies and Curie temperature are possible. The high sensitivity achievable enables measurements even on films approaching monolayer thickness. The method is fully compatible with UHV and—via the intrinsic stress—additionally provides important information on growth mode and microstructure of the films under investigation. First results on polycrystalline Fe films demonstrate impressively the performance of the technique.

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Magnetic thin films have attracted tremendous attention in the past two decades mainly due to their hitherto unrivaled relevance for storage technology [1]. More recently, since with refined molecular-beam epitaxy (MBE) techniques the preparation of well-defined low-dimensional epitaxial thin films has become possible, there is also growing fundamental interest [2] as ultrathin magnetic films may exhibit peculiar unexpected properties, e.g., a perpendicular magnetic anisotropy at film thicknesses up to a few monolayers [3]. For the study of magnetic properties such as magnetization, Curie temperature, or anisotropies of thin and ultrathin films several bulk techniques with enhanced surface sensitivity have been employed, e.g., torque magnetometers [4], Mössbauer spectroscopy [5], ferromagnetic resonance [6], or SMOKE (surface magneto-optic Kerr effect) [7]. Electronic configuration and magnetic domain structure, on the other hand, have been investigated with spin-polarized (SP) versions of standard surface science techniques, such as SP photoeletron spectroscopy [8], SP Auger electron spectroscopy [9], or SP scanning electron microscopy [10]. Undoubtedly there is also a strong demand for measurements of the magnetostrictive film properties [1]—i.e., the change of their equilibrium dimensions upon variation of magnetization—which in the case of thin films give rise to magnetostrictive stress, since adaptation to the new film dimensions is inhibited by adhesion to the substrate. There is an increasing number of examples where the magnetoelastic energy due to magnetostrictive and growth inherent intrinsic stress components is presumed to have exceeded the anisotropy energy of the films resulting in a change of the easy magnetization direction [11]; so far, however, experimental verification is lacking. Certainly such effects have crucial impact for technological applications of magnetic thin films. Magnetostriction measurements of thin films in the thickness range of 10–100 nm have been performed so far by means of the cantilever beam [12] and plate [13] techniques or indirectly by the effect of a known stress on the anisotropy field [14].

Here we describe a new experimental approach which enables us to determine *quantitatively* (i) the magnetization, (ii) the magnetostriction, and (iii) the intrinsic stress of thin films of thicknesses approaching even the monolayer; (iv) from the film stress, in addition, valuable information on microstructure and growth mode of the films can be deduced, which is of special importance for magnetic studies as thin film magnetism is strongly influenced by structural parameters. The proposed method is based on the cantilever beam principle as illustrated in Fig. 1(a), which has been successfully employed in the past to investigate the relation between intrinsic stress and the microstructure and growth mode of numerous polycrystalline [15] and epitaxial thin films [16]. The decisive point of the method is the accurate detection of the displacements of the free end of the cantilever beam substrate, from which quantitative values of the involved forces and torques can be derived. In our case the deflection is determined by a highly sensitive capacitance technique combined with lock-in-assisted signal detection that has been described in details in Refs. [17] and [18]. The smallest detectable substrate defection of our improved apparatus is approximately 1 nm, which—as demonstrated previously for the intrinsic stress measurements [19]—ultimately guarantees monolayer sensitivity also for the magnetic film properties. By mounting the cantilever beam device in the center of three orthogonal pairs of Helmholtz-like coils [Fig. 1(a)] homogeneous magnetic fields can be applied in any direction of space. By this means—in addition to the intrinsic film stress—also the magnetostriction constant of the films can be determined from the substrate bending due to magnetostrictive stress as well as the film magnetization (in special cases at least the $x$ component). In order to determine the film magnetization $\mathbf{M}$, we make use of the fact that magnetic dipoles $\mathbf{m} = V_F\mathbf{M}$ with $V_F$ being the film volume



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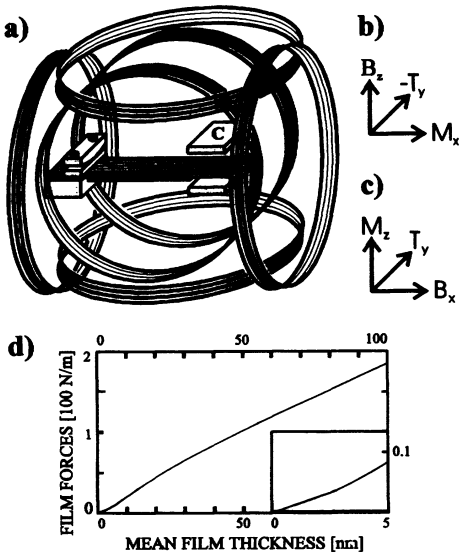


FIG. 1. (a) Schematic illustration of the cantilever beam device mounted in the center of three orthogonal pairs of Helmholtz-like coils for measurements of the magnetization, magnetostriction, and intrinsic stress of magnetic thin films; the displacement of the substrate's ($S$) free end is detected with high sensitivity by a differential capacitance technique ($C$). (b) and (c) Schematic illustration of the measurement of film magnetization $M$ (see text). (d) Film forces per unit width vs mean film thickness of polycrystalline Fe films UHV deposited at 300 K onto glass substrates.

experience a torque $\mathbf{T}_m$,

$$\mathbf{T}_m = \mathbf{m} \times \mathbf{B} = V_F \mathbf{M} \times \mathbf{B}, \tag{1}$$

in homogeneous magnetic fields $\mathbf{B}$, the $y$ component $T_y$ of which gives rise to a respective bending of the substrate. Because of the magnetic anisotropy of most magnetic thin films two simple situations frequently can be arranged, where the substrate deflection directly reflects the total film magnetization: (i) in the very common case of a strong in-plane anistropy (due to the demagnetizing field), by magnetizing the films in the $x$ direction (i.e., along the length of the cantilever beam) and applying a small vertical field $B_z$ [Fig. 1(b)] and (ii) in the case of an out-of-plane anisotropy by magnetizing the films in the $z$ direction and applying a magnetic field in the $x$ direction [Fig. 1(c)]. Last but not least two further advantages of the method have to be emphasized, namely, its compatibility with UHV and its applicability over a wide temperature range which also enables measurements of the Curie temperature of thin films with the same method.

Before we present first results measured with the new technique, we derive the formulas that are essential for evaluating the magnetic and mechanical film properties from the deflection $\Delta$ of the cantilever beam. For reasons of limited space, however, we concentrate our discussions on polycrystalline films deposited onto isotropic substrates; for epitaxial films grown on single-crystalline substrates the appropriate directional dependences of the elastic and magnetic constants have to be included. In equilibrium both the sum of all forces $F_i$ and the sum of all torques $T_i$ acting in the system film/substrate (distinguished in the following by indices $F$ and $S$, respectively) are zero. Therefore in the case of magnetostrictive and/or intrinsic stress within the films the substrate is strained until the resulting forces in the substrate $F_S$ balance the respective film forces $F_F$:

$$F_F = \int_{A_F} \sigma_F dA = -\int_{A_S} \sigma_S dA = -F_S . \tag{2}$$

The forces are obtained by integrating the stress $\sigma$ over the cross-sectional areas $A = tw$ of film and substrate with $t$ and $w$ being thickness and width, respectively. For uniform perpendicular stress distribution and films, which are completely covering one side of the substrate ($w_F = w_S = w$ and $l_F = l_S = l$), integration yields $\sigma_S = -(t_F/t_S)\sigma_F$. It follows that the fraction of film stress, which is relieved by straining of the substrate, is of the order of the ratio between the thicknesses of film $t_F$ and substrate $t_S$ and therefore negligibly small for thin films (about 1 promille for 100 nm thick films deposited onto 100 $\mu$m thick substrate).

Equilibrium furthermore requires that the torque $T_\sigma$, due to the forces $F_F$ and $F_S$,

$$T_\sigma = \tfrac{1}{2}\sigma_F w t_F (t_S + t_F), \tag{3}$$

is compensated by the bending moment $T_B$ of the cantilever substrate plate:

$$T_B = \frac{w E_S (t_S + t_F)^3}{12(1 - \nu_S^2)} \left( \frac{1}{R_x} + \nu_S \frac{1}{R_y} \right). \tag{4}$$

$E_S$ and $\nu_S$ denote Young's modulus and Poisson's ratio of the substrate, and $R_x$ and $R_y$ are the radii of curvature in the direction of its length and width [compare Fig. 1(a)]. For polycrystalline films exhibiting isotropic biaxial stress $R_x$ is equal to $R_y$; thus combination of Eqs. (3) and (4) straightforwardly leads to Stoney's well known formula [20], modified by using the bending moment of a plate [Eq. (5)] rather than that of a beam [21]:

$$\sigma_F = -\frac{E_S (t_S + t_F)^2}{6(1 - \nu_S) R t_F} \cong -\frac{E_S t_S^2}{3(1 - \nu_S) l^2 t_F} \Delta. \tag{5}$$

To arrive at the right side of Eq. (5) $t_F$ was neglected against $t_S$ and the approximation $1/R \cong 2\Delta/l^2$ was made.

In order to determine quantitatively the magnetostrictive constant $\lambda$ it is necessary to measure the relative substrate deflection between two well-defined states of film magnetization, in our case along the length ($\Delta^L$) and width ($\Delta^W$) of the substrate. The difference between the corresponding $x$ components of the magnetostrictive stress then is calculated to

$$\sigma_x^L - \sigma_x^W \cong -\frac{t_S^2 E_S}{3 l^2 t_F (1 + \nu_S)} (\Delta^L - \Delta^W) \tag{6}$$

by using $(1/R_y)^L = (1/R_x)^W$ and $(1/R_y)^W = (1/R_x)^L$. $\sigma_x^L - \sigma_x^W$ is linked to $\lambda$ via the stress-strain ($\sigma$-$\varepsilon$) relations for isotropic materials $\varepsilon_x = (\sigma_x - \nu\sigma_y)/E$ and $\varepsilon_y = (\sigma_y - \nu\sigma_x)/E$. Assuming that the volume change upon magnetization is small, the magnetostrictive strain $\lambda$ in the direction of magnetization is accompanied by a respective strain of $-\lambda/2$ in the two perpendicular directions and one obtains

$$\sigma_x^L - \sigma_x^W = -\frac{3E_F}{2(1 + \nu_F)}\lambda. \quad (7)$$

Combination of Eqs. (6) and (7) leads to

$$\lambda = \frac{2t_S^2}{9l^2t_F}\frac{E_S(1 + \nu_F)}{E_F(1 + \nu_S)}(\Delta^L - \Delta^W). \quad (8)$$

Notice the term $1 + \nu_S$ in the denominator of Eq. (8), which is contrary to the $1 - \nu_S$ of the erroneous formula used previously [12]. We remark that magnetostriction depends only on the magnetization of the films and usually a remnant component is observed even when the magnetizing fields have been turned off.

As stated above, the film magnetization can be calculated from the torque which magnetic dipoles experience in homogeneous magnetic fields. The corresponding $y$ component $T_{y,M} = V_F(M_zB_x - M_xB_z)$ in equilibrium is compensated by the bending moment $T_B$ of the substrate. In the very common case of a strong in-plane anisotropy ($M_z = 0$), the film magnetization is not changed by perpendicular fields $B_z$ smaller than the respective coercitive field and $T_{y,M} = -V_FM_xB_z$. For films magnetized in the $x$ direction, taking into account that $1/R_y = -\nu_S/R_x$, because there are no forces in the $y$ direction involved, combination with Eq. (4) yields

$$M = M_x = \frac{E_S w t_S^3}{6V_FB_zl^2}\Delta. \quad (9)$$

An analogous equation is easily derived for films with perpendicular anisotropy.

The results presented in the following concern polycrystalline Fe films electron beam evaporated at 300 K from Knudsen-type tungsten crucibles onto $30 \times 5 \times 0.14$ mm$^3$ cantilever beam glass substrates, which have been carefully outgassed at 600 K prior to the film deposition. Thin film preparation as well as the cantilever beam measurements were performed in a UHV system with a base pressure better than $1 \times 10^{-10}$ mbar (see Ref. [18]). The mean film thickness was determined with a quartz crystal microbalance calibrated by a second quartz in substrate position. The deposition rate was 0.005 nm/s and the residual gas pressure during deposition was better than $2 \times 10^{-9}$ mbar. The intrinsic film stress was determined continuously during the film deposition; the magnetic measurements were started about 15 min later. To calculate quantitative values for the mechanical and magnetic properties a defined substrate deflection was produced as reference by applying a known weight.

As stated above the intrinsic film stress provides detailed information on film growth and microstructure. The film forces of our Fe films, shown in Fig. 1(d), are tensile and increase nearly linearly with film thickness. The corresponding film stress of $1.9 \times 10^9$ N/m$^2$ is in good agreement with previous studies on polycrystalline Fe films growing on amorphous substrates [22]. As discussed in detail in Ref. [15] or [16], the dependence of the film forces on film thickness reveals that Fe has grown by columnar grain growth, a modification of Volmer-Weber or island growth for metals with low mobility. The film stress is dominated by the tensile stress at the small angle grain boundaries, which form the major defects of columnarly grained films. The first appearance of tensile stress is indicative of the beginning coalescence of individual islands and due to the low mobility of Fe at 300 K lies close to percolation ($\approx 2-3$ nm) as confirmed by simultaneous electrical conductance measurements. From the film thickness at percolation an average grain size of about 6 nm can be estimated (see Ref. [18]) in accordance with a scanning tunneling microscopy investigation of analogous Fe film deposited onto mica substrates [23].

Figures 2(a)–2(d) show magnetic hysteresis loops of various Fe films obtained by varying the magnetic field in the $x$ direction [compare Fig. 1(a)] and measuring the substrate deflection due to short pulses of a vertical magnetic field $B_z$ of 3400 A/m. The Fe films exhibit mean thicknesses ranging from 100 nm down to 3 nm, at which the films are already electrically conducting (percolation).

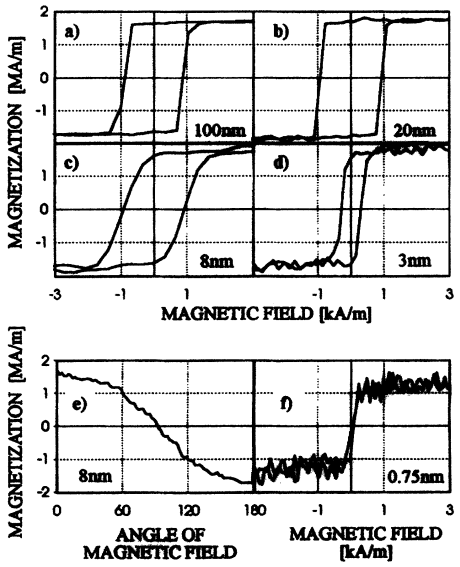


FIG. 2. Hysteresis loops of polycrystalline Fe films UHV deposited onto glass substrates (a) 100 nm, (b) 20 nm, (c) 8 nm, and (d) 3 nm; for comparison, the saturation magnetization of bulk iron is 1.76 MA/m. (e) Respective $x$ component of the remnant film magnetization of the 8 nm Fe film as a function of the angle of rotation of the magnetizing field applied within the film plane; the corresponding cosine dependence indicates isotropic magnetic behavior. (f) Hysteresis loop of a continuous 0.75 nm thick Fe film ($\approx 5$ monolayers) sandwiched between two Ag films on MgO(001).

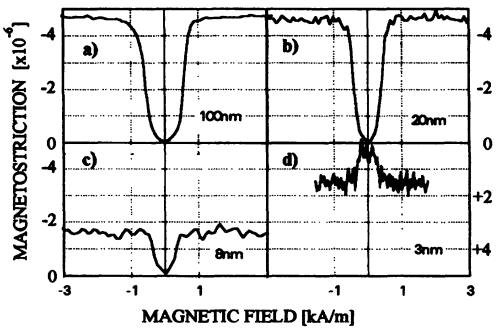


FIG. 3. Remnant magnetostrictive properties of polycrystalline Fe films UHV deposited onto glass substrates: (a) 100 nm, (b) 20 nm, (c) 8 nm, and (d) 3 nm; for comparison, the saturation magnetostriction of bulk iron is $-4.6 \times 10^{-6}$.

The experimental saturation magnetization of all films is $1.78 \pm 0.08$ MA/m, in excellent agreement with the respective bulk value of 1.76 MA/m. All hysteresis loops are well resolved; from the signal to noise ratio of 3 nm film corresponding to about 20 monolayers a sensitivity down to film thicknesses of 1–2 monolayers can be extrapolated. We mention that at mean thicknesses below 3 nm our Fe films exhibit (super)paramagnetic behavior, which is typical of thin films in the island stage of film growth. It is interesting to note that the coercive field of the films in the channel state (3–5 nm) is significantly smaller than that of the continuous films [Figs. 2(a)–2(c)]. In order to further illustrate the capacity of the technique the hysteresis of a continuous 0.75 nm thick Fe film ($\approx$ 5 monolayers) sandwiched between two Ag films on MgO(001) is plotted in Fig. 2(f). Even at this low film thickness the two branches of the hysteresis are clearly distinguished. The saturation magnetization of $1.25 \pm 0.08$ MA/m lies significantly below the bulk value probably due to a considerable reduction of the Curie temperature. Rotation of the magnetizing field within the film plane and measurement of the corresponding remnant $x$ component of film magnetization enables us to detect possible magnetic anisotropies. An example is shown in Fig. 2(e), where $M_x$, depending on the angle of the applied field, follows a cosine function, indicating isotropic magnetic behavior as should be expected for polycrystalline Fe films (on glass substrates).

Figure 3 summarizes the *remnant* magnetostrictive properties of the system Fe/glass, calculated from the relative substrate deflection after saturating the films in the $y$ direction and subsequently applying a respective field in the $x$ direction. The experimental saturation value therefore corresponds to the saturation magnetostriction $\lambda_S$, while the onset of magnetostriction depends on the respective in-plane coercivity. At 100 nm and 20 nm $\lambda_S$ is $-4.6 \times 10^{-6}$, in good agreement with the respective bulk value ($-4.4 \times 10^{-6}$). At mean film thicknesses below 10 nm considerable deviations from the bulk value are observed: $\lambda_S$ is $-1.7 \times 10^{-6}$ at 8 nm, and at 3 nm even the sign has changed ($\lambda_S = +1.5 \times 10^{-6}$).

In conclusion, the presented results convincingly demonstrate that the cantilever beam technique—used so far mainly to study mechanical film properties—constitutes a powerful method to investigate the magnetic properties of thin and ultrathin films as well.

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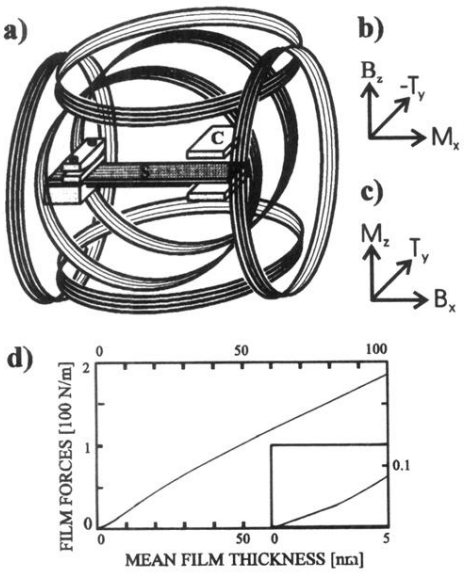


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