Determination of the properties of iron films during evaporation

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Ferromagnetic iron films were evaporated in $\sim 10^{-6}$Torr vacuum on lithium fluoride, glass, and magnesium oxide. The reflection coefficient in the range $\lambda = 0.8$–1.1μ, the amplitude of the magneto-optic equatorial effect, and the coercive force were monitored during evaporation. The film thickness was measured by a radiofrequency quartz balance method (sensitivity 24.5 Hz/Å) and the substrate temperature varied from one experiment to another between 100 and 850°K. An analysis of the measured quantities made it possible to identify continuous films, beginning from the lowest thicknesses. The dependence of the Curie point of iron on the sample thicknesses was obtained.

1. INTRODUCTION

Investigations of the properties of ferromagnetic films during their preparation have two important advantages over with the conventional studies after preparation. Firstly, this approach makes it possible to determine most clearly the dependence of the magnetic properties on the thickness of a film. Dynamic studies of this dependence provide new experimental means for the determination and interpretation of the properties of films of different thicknesses. Moreover, this avoids the question of the constancy, during an investigation, of the properties of films several atomic layers thick. Secondly, studies of the magnetic properties during the preparation of films make it possible to prepare samples with a known distribution of the properties across the thickness. This makes it much easier to use such films in other experiments.

It is desirable that a study of the properties of ferromagnetic films during preparation be accompanied by monitoring of the structure of the samples. Electron microscopy and electron diffraction are among the most popular methods suitable for structure monitoring. However, the use of electron microscopy during film evaporation restricts the range of materials that can be employed as substrates and this is a serious difficulty in the preparation of high-quality samples of interesting materials.[1] The accumulated experience in electron diffraction during film evaporation shows that, at least in some cases, the electron bombardment results in the deterioration of the film structure.[2] Therefore, the question of monitoring of a film structure during its evaporation requires special consideration.

The present paper reports measurements of the reflection coefficient and magnetooptic properties in the range $\lambda = 0.8$—$1.1\ \mu$ during the evaporation of films on substrates kept at temperatures from 100 to 850°K.

2. EXPERIMENTAL METHOD

The equatorial Kerr effect was used as the main investigation method.[3,4] A schematic diagram of the apparatus is shown in Fig. 1. A light spot falling on a sample was $\sim 1 \times 1$ mm. A silicon photodiode was used as a detector. The photodiode circuit[5] produced a voltage proportional to the light flux and this made it possible to monitor directly the reflection coefficient of a film. Cyclic reversal of the magnetization produced a proportional (to the magnetization) alternating voltage at the photodiode output and this voltage represented the alternating component of the light flux due to the equatorial Kerr effect.

The coercive force was measured with a circuit described earlier.[6] The amplitude of the field in different experiments ranged from 60 to 380 Oe. The apparatus enabled us to determine reliably the magnetooptic signal of $\sim 10^{-5}$ relative amplitude. The circuit for measuring the coercive force recorded the width of a hysteresis loop when the relative magnetooptic signal was $\sim 10^{-6}$.

The mass deposited on 1 cm^2 of a sample was measured during evaporation by means of a radiofrequency (rf) quartz balance.[7] We employed quartz resonators of the AT cut with a natural frequency of 16 MHz. The drift of the rf balance frequency under the influence of heating gave rise to an error in the measurement of the specific mass of the film which did not exceed 5%. Control experiments showed that the film and quartz resonator were in a homogeneous flux of the substance being evaporated, which emerged from a point source. According to Mueller and White,[8] the calibration constant for 16 MHz was 580 Hz/(μg/cm^2), to within 1%. A check showed that the constant of our microbalance was very close to this value. In the geometry employed in our study, the calibration constant of the rf quartz microbalance was 315 Hz/(μg/cm^2).

An additional error in the measurement of the mass of sample could arise because of the difference between the sticking coefficients of the quartz resonator and the substrate. The influence of this effect was reduced by the following procedure. The substrate was covered by a screen and a film of the investigated substance up to 200 Å thick was evaporated on the quartz resonator. The

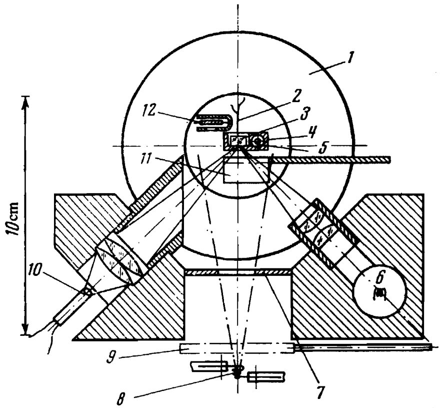

FIG. 1. Schematic diagram of the apparatus: 1) solenoid; 2) thermocouple; 3) substrate; 4) holder; 5) heater; 6) incandescent lamp; 7) screen with aperture; 8) evaporator; 9), 11) movable screens; 10) photodiode; 12) quartz resonator.

screen was then removed and the evaporation was continued both on the resonator and substrate. This procedure could only give an overestimate of the sample mass since the sticking coefficient of a metal reaching a freshly deposited metal was unity.

The additional rise of the substrate temperature during evaporation[9] was determined in control experiments in which a copper-constantan thermocouple was bonded to the substrate on the evaporator side. Illumination provided by a lamp raised the temperature of the substrate by ~30 deg in 10 min and the initial heating rate was 10 deg/min. When iron was evaporated at a rate of 30 Å/sec, the heating rate was 0.5 deg/sec. Thus, the absolute rise of the substrate temperature due to evaporation was small. Nevertheless, accidental variations of the substrate temperature were probably responsible for the scatter of the properties from sample to sample and they were manifested more strongly for thicker samples.

Tungsten evaporators were raised in temperature to white heat in $\sim 10^{-5}$ Torr vacuum before the experiments. The contamination of the quartz resonator and substrate by the evaporators without charge was estimated from the frequency shift which occurred when the evaporators alone were heated. This frequency shift did not exceed 0.025 Hz/sec. During the evaporation of the films, the frequency shift was 15–400 Hz/sec. The results relating to the contamination by the evaporators and to a possible overheating of the substrate and quartz resonator were obtained under the least favorable conditions. In all the experiments, the stray optical signal produced by the evaporators and reaching the photodiode was negligible.

The lowest vacuum reached in the system was 3×10^{-7} Torr but it deteriorated to $\lesssim 10^{-5}$ Torr at the end of an evaporation run.

3. EXPERIMENTAL RESULTS

3.1. Film and Substrate Materials

The film material was iron of purity better than 99.9%. Very thin (<20 Å) polycrystalline iron films had been investigated by several workers[10–12] but some of these investigations[10,11] were carried out only at room temperature. Detailed calculations of the dependence of the Curie temperature and magnetic moment on the film thickness were reported in[13,14]. In most of our experiments, the substrates were LiF single crystals. The mismatch of the characteristic parameters of the iron and lithium fluoride lattices was only 0.5%.[15] Hence, we expected that the structure of very thin iron films on these substrates would be closest to a model single crystal of the same thickness. Some of the samples were evaporated on glass and MgO single crystals.

3.2 Preparation of Samples

Crystals of LiF and MgO were cleaved in air. A cleaved lithium fluoride surface was usually a plane of the (100) type. In all cases when iron films were epitaxial, they grew in accordance with the diagonal scheme.[16,17] Thus, most of the results were obtained for the switching of the sample magnetization along the [110] direction in a (100) plane.

The substrate was first heated to 200° C for several minutes in those cases when the evaporation was carried out with the substrate temperature below 200° C.

The whole system was tested by evaporating copper and manganese on lithium fluoride and gold on sodium chloride. These experiments showed that the relative error due to cracking and strays in the measurement of the magnetooptic signal did not exceed 10%. In the absence of the magnetization signal, the circuit for measuring the coercive force produced a voltage which was almost proportional to the switching field amplitude and it did not react to the strays.

3.3 Experimental Results

Figure 2 gives the results obtained in one of the evaporations of iron on lithium fluoride. In the presence of magnetization, the signal produced by the circuit for measuring the coercive force fell to a value corresponding to the real width of the hysteresis loop.

Figures 3–5 show typical records obtained for different samples. It is clear from Fig. 3 that there were three main types of dependence of the reflection coefficient on the thickness. It was found most frequently that, irrespective of the rate of evaporation or temperature, the reflection coefficient varied with thickness in the way shown for samples I, II, and III in Fig. 3. The arrow in Fig. 3 shows the rise of the reflection coefficient of sample V, which occurred on heating to 200° C.

The dependence of the magnetooptic signal on the thickness (Fig. 4) was similar to the corresponding dependence of the reflection coefficient. In the case of samples of type I–III, the magnetooptic signal appeared for very small thicknesses and differed little from sample to sample. In the case of samples of type IV, this signal appeared for larger thicknesses and, in the case of a sample of type V, the signal appeared even much later than for samples I–III. After heating, the magnetooptic signal of sample V rose strongly by a factor of 8 (see curve V in Fig. 7).

Figure 5 shows the dependence of the coercive force on the film thickness. The time constant of the measur-

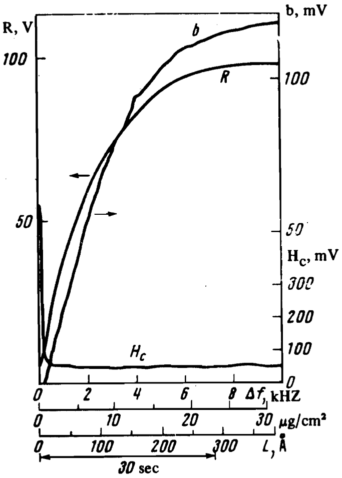

FIG. 2

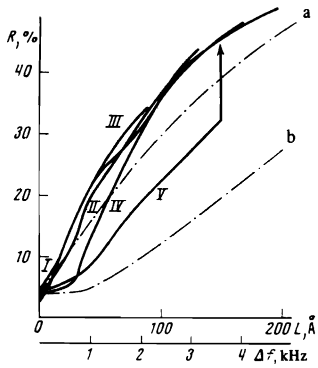

FIG. 3

FIG. 2. Variations of the reflected light flux R, magnetooptic signal b, and coercive force H_C in the course of evaporation of iron on lithium fluoride. The amplitude of the switching field was $H_0 = 250$ Oe.

FIG. 3. Dependences of the reflection coefficient of light on the thickness of iron films on lithium fluoride. Substrate temperature during evaporation (°K): I) 140; II) 500, III) 720; IV) 700; V) 300. a), b) Dependences of the reflection coefficient of continuous and island iron films on lithium fluoride ($\gamma = 1\mu, \varphi = 45°$).

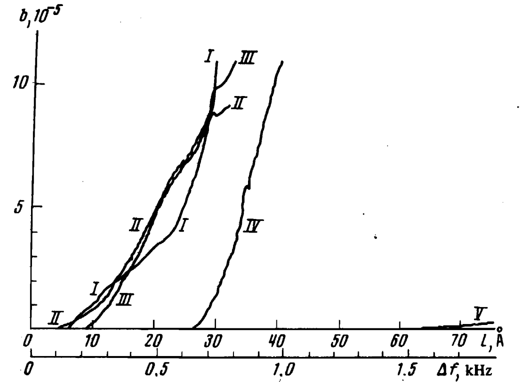

FIG. 4. Dependences of the amplitude of the magnetooptic signal b on the film thickness. The samples are the same as in Fig. 3.

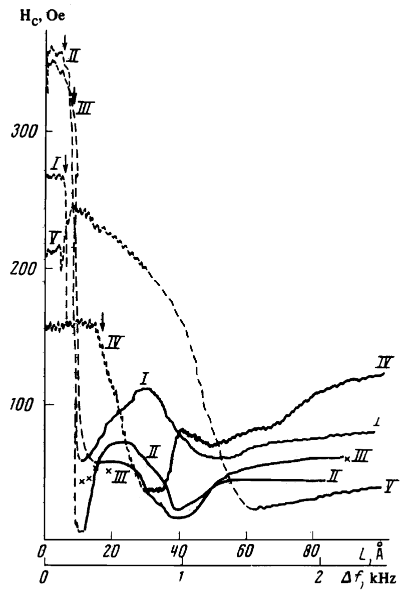

FIG. 5. Dependences of the coercive force on the thickness of a sample. The samples are the same as in Figs. 3 and 4. Crosses are used to denote the values obtained from photographs of sample III.

ing circuit did not allow us to follow accurately the sudden changes in the coercive force, particularly those associated with the appearance of the magnetooptic signal. However, a study of the evaporation of nonferromagnetic metals indicated that the fall of the signal by 5% at the output of the coercive force measuring circuit could only be due to the appearance of the magnetooptic signal. The moments at which these changes occurred are identified by arrows in Fig. 5. The continuous curves in this figure are shown only in those cases when the coercive force measuring circuit followed reliably the true width of the hysteresis loop.

A coercive force minimum corresponding to the film thickness ~10 Å was typical of samples I–III. The minimum at 40–60 Å was exhibited only by some samples of type I–III. No singularities in the coercive force were found for greater thicknesses, which was in agreement with Hoffmann's results.[18] When samples were cooled after evaporation, the coercive force sometimes varied in a very surprising manner, which showed that the structure of a film could change even after evaporation.

When the evaporation was completed, we investigated the anisotropy of the magnetooptic effect in weak fields, in the same way as was done earlier.[17] These measurements were carried out in air using separate apparatus.[4] When samples were exposed to air, the magnetooptic signal, coercive force, and reflection coefficient remained constant for films $\gtrsim 60$ Å thick. All the samples prepared by evaporation on substrates kept at temperatures of 150–300°C exhibited a 90° anisotropy of the magnetooptic signal. The other samples exhibited a 180° anisotropy or were isotropic.

On the whole, the adopted investigation method was found to be suitable for determination of the magnetooptic properties of ferromagnetic films right down to the smallest thicknesses and the investigations could be carried out in the range from 100 to 850°K.

4. DISCUSSION OF RESULTS

4.1 Density of Samples

The films evaporated on substrates kept at temperatures of 200–250°C were investigated by the magnetic resonance method. The results were reported in[19] and could be summarized as follows. All the investigated samples, right down to a thickness of 140 Å, had magnetic properties very similar to those of iron single crystals. The resonance measurements indicated the absence of spin pinning on the surface. The only quantity which varied significantly from sample to sample was the width of the resonance absorption line. For most of the samples, this width was ~100 Oe in the 8-mm range, i.e., the resonance lines were exceptionally narrow for samples of this kind.

The results of the resonance measurements suggested that the density of the films was equal to the density of iron single crystals (7.8 g/cm^3). It is clear from Fig. 3 that the reflection coefficient of type I–III samples was practically independent of the substrate temperature during evaporation. Hence, we concluded that the structure of these samples was the same throughout the investigated range of substrate temperatures. We then assumed that the density of all the films was equal to the density of single-crystal iron and we calculated the thickness using the readings of the rf quartz microbalance. The sensitivity of the balance, reduced to the thickness was 24.5 Hz/Å. All the thicknesses given in the present paper were found using this calibration constant.

4.2 Models of Continuous and Island Films

In order to obtain information on the physics of magnetism from experiments on thin films, we have to know whether these films are continuous. In the absence of direct structure monitoring, the continuity may be judged by comparing the dependences of the measured quantities on the thickness of a sample with the corresponding dependences expected from the models of continuous and island films. Let us consider the reflection of light from a continuous film on a substrate.[20] When light is incident from vacuum (medium 1) on a film (medium 2), located on a substrate (medium 3), the amplitude of the reflection of the s- and p-polarized light is given by

$$r(s,p)=\frac{r_{12}-r_{23}e^{2i\psi}}{1-r_{12}r_{23}e^{2i\psi}}, \tag{1}$$

$$\psi=\omega L(\varepsilon_2-\sin^2\varphi)^{1/2}/c, \tag{2}$$

$$r_{23}=(r_{12}-r_{13})/(1-r_{12}r_{13}). \tag{3}$$

Here, r_{12} and r_{13} are the amplitudes of light reflection at interfaces between media 1 and 2 and 1 and 3, respectively. The reflection coefficient is $R = |r|^2$; L is the film thickness; ϵ_2 is the film permittivity; ω is the incident light frequency; c is the velocity of light; φ is the angle of incidence of light.

The permittivity tensor of a magnetic medium is of the form

$$(\hat{\varepsilon}_i)=\begin{pmatrix} \varepsilon_2 & -i\varepsilon' I/I_s & 0 \\ i\varepsilon' I/I_s & \varepsilon_2 & 0 \\ 0 & 0 & \varepsilon_2 \end{pmatrix}.$$

In the case of the equatorial Kerr effect, i.e., when the direction of magnetization is perpendicular to the plane of incidence of light, we have

$$r_{12}(p)=r_{12}^0(p)+\Delta r_{12}(p) I/I_s, \tag{4}$$

where

$$\Delta r_{12}(p)=i\varepsilon' \frac{2\sin\varphi\cos\varphi}{[\varepsilon_2\cos\varphi+(\varepsilon_2-\sin^2\varphi)^{1/2}]^2}$$

and I/I_S is the relative magnetization which is assumed to be unity.

Using the above formulas, we find that the magnetooptic change in the intensity of light reflected by a film on a substrate is

$$b=\Delta R=\left|r(p)+\Delta r_{12}\frac{dr(p)}{dr_{12}(p)}\right|^2-|r(p)|^2=2\,\mathrm{Re}\left(\frac{\Delta r_{12}(p)}{r(p)}\frac{dr(p)}{dr_{12}(p)}\right)|r(p)|^2; \tag{5}$$

where

$$\frac{dr(p)}{dr_{12}(p)}=\frac{[1-r^2(p)][1-r_0^2]}{1-r_{12}^2(p)},$$

$$r_0^2=\frac{(1-r_{23}^2)e^{2i\psi}}{1-r_{23}^2e^{4i\psi}}.$$

We used Eqs. (1) and (5) to calculate, for an angle of incidence of 45°, the reflection coefficient of unpolarized light of $\lambda = 1\ \mu$ wavelength and the magnetooptic effect in a film of iron on a lithium fluoride substate. We used the values of ϵ and ϵ' of iron taken from the paper of Krinchik and Artem'ev.[3] It was assumed that the magnetization remained constant right down to zero thickness.

The reflection coefficient of an island film was calculated in the following way. Doremus[21] calculated the permittivity of a system of separate islands on a substrate with a known permittivity for the case when $\lambda \gg 2r$, where 2r is the island diameter. In these calculations, it is necessary to know the surface coverage, i.e., the ratio of the area occupied by islands to the total area of the film. Published information was available on island films of nickel and silver on quartz.[22,23] The surface coverage given in these papers for the average film thickness agreed with one another and the conditions used in the preparation of these films differed only slightly from those employed in our experiments. We used these values of the surface coverage in a calculation of the permittivity and then of the reflection coefficient at $\lambda = 1\ \mu$ for an angle of incidence of 45° in the case of iron islands on lithium fluoride, as a function of the average film thickness. The results of these calculations are plotted in Fig. 3. We can see that the reflection coefficient of a continuous film rises much faster with thickness than the corresponding coefficient of an island film.

The following comments can be made on the magnetooptics of island films. In the case of a superparamagnetic system of iron islands up to 100 Å in diameter, i.e., up to an average film thickness of 10–50 Å, the relaxation time at 500–700°K was less than 1/50 sec, so that measurements at 50 Hz would be impossible in weak fields.[24] Estimates obtained using a method suggested by Neugebauer[25] indicated that the magnetization of an island system changed only severalfold when the field was increased from 100 to 400 Oe. This should result in a considerable difference between the dependences of the magnetooptic signal on the thickness for samples subjected to different magnetization switching fields. In general, the magnetooptics signal produced by an island film should be weaker than that due to a continuous film.

The dependence of the coercive force on the thickness of continuous films will require further study. We shall point out only that, in the case of thin films, the coercive force should depend strongly on the quality of the substrate and, in the case of thick homogeneous films, the coercive force should not vary greatly with thickness. A coercive force maximum of island iron films was reported to occur for an average film thickness of ~60–100 Å and the coercive force at this maximum was 200–500 Oe.[18,26]

4.3 Comparison with Experimental Results

Since we did not determine the optical and magnetooptic constants, a comparison with the calculations could only be qualitative. The reflection coefficient was calibrated as follows. It was assumed that the reflection coefficient of a 300 Å thick film was in agreement with the calculated value and that it amounted to 64%. The reflection coefficient of films whose final thickness was less than 300 Å was fitted to the curve obtained in this way. The reflection coefficients of samples of type I–III of all thicknesses differed from one another by not more than 3%. The average value of the reflection coefficient of LiF was 3.46 ± 0.13%, which was close to the value calculated for $\lambda = 1\ \mu$ (3.47%). A considerable difference between the continuous and island films (curves a and b in Fig. 3) appeared in the derivative of the reflection coefficient with respect to the thickness, obtained for very small thicknesses. Hence, the results plotted in Fig. 3 could best be interpreted as follows. Samples of type I–III were continuous, beginning from the smallest thicknesses. Sample V remained of the island type after evaporation. The islands merged during annealing to form a continuous film. Thin samples of type IV were of the island type but thicker films merged during evaporation.

The dependence of the magnetooptic signal on the film thickness confirmed that type I–III films were continuous, beginning from the smallest thicknesses. In fact, the magnetooptic signal appeared beginning from thicknesses <10 Å and its dependence on thickness was close to the calculated curve.

Figure 6 shows photographs of hysteresis loops obtained for samples of type I–III. The presence of a normal hysteresis loop in the case of samples <30 Å thick indicated, as shown by Neugebauer in [25], that these were continuous films. Figure 7 shows the dependence of the relative change in the intensity of the reflected light $\delta = b/R$ on the film thickness. It is clear from this figure that the dependence of δ on the thickness of type I–III films was in qualitative agreement with the curve calculated for a continuous film.

Further support for these conclusions was provided by measurements of the coercive force. In the case of type I–III samples, the coercive force was close to 50 Oe and did not exhibit the 200–500 Oe maximum re-

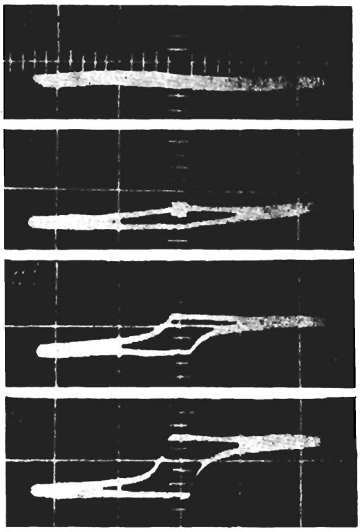

FIG. 6. Hysteresis loops obtained for iron films of type VI which had different thicknesses and were deposited on lithium fluoride substrates kept at a temperature $T_{sub} = 480°K$; $H_0 = 380$ Oe.

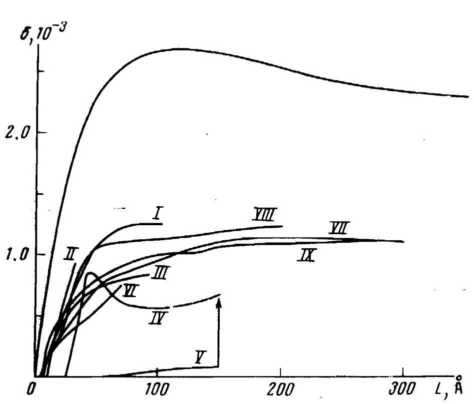

FIG. 7. Dependences of the relative change in the intensity of the reflected light $\delta = b/R$ on the thickness of a sample. The topmost curve represents the results of calculation for a continuous iron film on lithium fluoride. The samples are the same as in Figs. 4–6 and 8. Here, VII denotes iron on lithium fluoride, $T_{sub} = 530°K$, $H_0 = 120$ Oe; VIII denotes iron on glass, $T_{sub} = 500°K$; IX denotes iron on magnesium oxide, $T_{sub} = 540°K$, $H_0 = 380$ Oe.

ported for island films. The coercive force depended weakly on the substrate temperature during evaporation. In the case of sample V, the coercive force measuring circuit recorded a maximum. Type IV samples exhibited behavior intermediate between that of samples I—III, on the one hand, and sample V, on the other. Thus, the results obtained indicated that iron films prepared in our study could be continuous, beginning from thicknesses of ~5 Å.

It was reported in[27] that iron films evaporated on LiF remained of the island type right up to 200 Å. This discrepancy between our results and those reported in[27] was due to different conditions during the evaporation process.

4.4 Dependence of Curie Point on Film Thickness

Since the method employed enabled us to evaporate continuous iron films and monitor their properties during evaporation, we were able to determine the dependence of the thickness at which ferromagnetic properties first appeared on the substrate temperature. This was equivalent to an investigation of the thickness dependence of the Curie temperature of the ferromagnet because, as long as the thickness was below the value for which the substrate temperature was equal to the Curie temperature, the magnetooptic effect could not be observed. It is clear from Figs. 3—5 that the reflection coefficient of samples of type I—III increased, beginning from zero thickness, and its dependence on thickness was close to that observed for bulk crystals. However, the magnetooptic signal and the coercive force appeared only from a certain finite thickness, whose value depended on the substrate temperature. This thickness was practically independent of the magnetic field used to switch the magnetization of the sample. For each sample, the thickness corresponding to the onset of ferromagnetic properties was determined from appearance of the magnetooptic signal and from the sudden fall of the signal produced by the coercive force measuring circuit.

The influence of the substrate on the minimum thickness corresponding to the appearance of ferromagnetic properties was determined by control experiments in which the substrates were MgO single crystals or glass. Evaporation on magnesium oxide took place at 250°C and on glass at 230 and 25°C. The behavior of the films deposited on magnesium oxide and on heated glass substrates did not differ from the behavior of the films deposited on the LiF at the corresponding temperature (Fig. 7). In the case of films evaporated on glass at room temperature, the dependence of the reflection coefficient on the thickness was qualitatively similar to the corresponding dependence predicted by the model of island films and the thickness corresponding to the onset of ferromagnetic properties rose to 20 Å. The coercive force was found to have a maximum of 100 Oe in the thickness range 50—70 Å.

Figure 8 shows a summary of our measurements of the thickness at which the ferromagnetic properties were first observed in continuous iron films deposited on substrates kept at different temperatures. This figure also includes the theoretical dependences of the Curie point of iron on the thickness of a sample calculated by the molecular field method (I)[13] and by the Green's function method (II).[14] It is clear from Fig. 8 that the minimum thickness at which ferromagnetism was observed in our samples was ~5 Å. This could be due to several factors. Firstly, it could be the thickness at which the film became continuous. In the range of these thicknesses, the monitoring of the film continuity based on the reflection coefficient was still ineffective. Secondly, our method was sensitive only to the component of

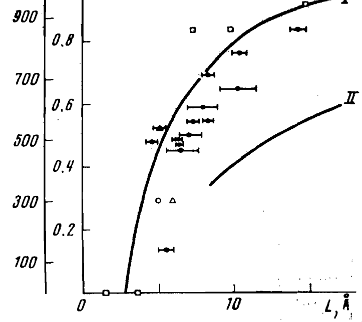

FIG. 8. Dependences of the Curie point of iron on the film thickness. Our results for Fe on LiF (●), Fe on MgO (▲), and Fe on glass (X), and the results taken from [11] (○), [12] (△), and [13] (□).

the magnetization lying in the plane of the film and, therefore, it was not possible to use this method to record the magnetization in the case of a strong anisotropy perpendicular to the plane of the film, whose existence in films thinner than 5 Å was reported by Gradmann and Müller.[28] Measurements of the magnetization of thin iron films prepared by electrolytic precipitation indicated that the room-temperature ferromagnetism also appeared at ~6 Å,[11] but the method employed in that case gave only the magnetization in the plane of the film. On the other hand, the Mössbauer method used by Lee et al.[12] was sensitive to the absolute value of the magnetization. Therefore, the absence of the magnetic moment in the case of samples 4.6 Å thick, reported in[12], was more likely an indication of the existence of two "dead" iron monolayers than the presence of an anisotropy perpendicular to the surface of the film.

It is clear from Fig. 8 that the experimental points were close to the curve calculated by the molecular field method. It was reported in[28] that the best agreement for Permalloy was obtained by the Green's function method. The agreement between the results obtained and the calculations based on various models of "ideal" samples indicated that our films were of a fairly high quality and suggested that measurements on films of better quality with a carefully controlled structure could only refine the dependence obtained and would not alter it qualitatively.

5. CONCLUSIONS

Thus, in the present investigation: 1) we developed a magnetooptic method for monitoring the optical and magnetooptic properties of thin ferromagnetic films during their evaporation; 2) we demonstrated the possibility of depositing, on a wide range of single-crystal LiF substrates, iron films which exhibited thickness dependences of the properties similar to those of bulk crystals; 3) we determined the dependence of the Curie temperature of iron on the thickness of a sample.

Further development of this investigation method should include measurement of the permittivity of the samples during evaporation, improvement of the substrate quality, determination of the influence of substrates on the film properties, and development of techniques for monitoring the structure of films during evaporation.

This approach should make it possible to determine the dependence of the magnetic anisotropy on the film thickness. Magnetooptic investigations of very thin films at different wavelengths should also make it possible to determine the dependence of the density of electron states in a ferromagnet on its thickness.

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