

INVESTIGATION OF THE MAGNETIC STRUCTURE OF FERROMAGNETIC SUBSTANCES
BY A MAGNETOOPTICAL APPARATUS WITH MICRON RESOLUTION

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A new magneto-optical method for local measurement of magnetic characteristics is employed for studying the magnetic structure of ferromagnetic substances. The thickness of a 180° domain boundary in nickel and iron is measured. The orientation of the magnetization vector in surface domains 0.65 to 1.8μ thick in permalloy films with a "supercritical" hysteresis loop is determined.

1. INTRODUCTION

APPARATUS for investigating the magnetic properties of a ferromagnetic substance in micropor-tions of a surface of about one square micron in area was described in^[1]. As follows from the nature of the dynamic method used in this work, the apparatus developed allows one to determine the change of magnetization of the investigated local portion of the surface of the ferromagnet during a periodic change, within certain limits, of an external magnetic field or of an elastic stress. The lower limit of the dimensions of the investigated local portion of the surface corresponds to the limit of optical resolution, i.e., to approximately $0.2-0.3 \mu$. The volume of the investigated portion is determined by the depth of penetration of the light into the ferromagnet. For metals this depth is several hundredths of a micron, which needs to be taken into account in interpreting the results obtained on bulky samples, or ferromagnetic films that are relatively thick. The method may be used with equal success to study changes in the magnetization of the portion due both to displacement of domain boundaries and to rotation of the magnetization vector. The most interesting applications of the method, obviously, are those cases when the magnetic characteristics or the magnetic state of the portion under investigation differ from the average characteristics of the whole sample. Thus, the investigated local object may be an individual domain of the ferromagnet, a domain boundary, a finely dispersed inclusion of a different phase, a region of the ferromagnetic substance surrounding a discrete dislocation, a nucleus of magnetic reversal, a microscopically small ferromagnetic sample, etc.

From the above it follows that the local method of measuring magnetization can find diverse physical and practical applications. The present paper illustrates some possibilities of using this method for physical investigations of the magnetic structure of ferromagnets.

The measurements were performed in an apparatus similar to the one described in^[1] with certain changes in individual units. To focus the light, an MIM-8 reflecting metallographic microscope was used. The light detector was an FÉU-19M photomultiplier with an adjustable slit, permitting investigation of the light reflected from microportions of the ferromagnetic surface of about one square micron in area. A microfeed permitted moving the photomultiplier in the focal plane of the microscope. From the photomultiplier, the signal went to the input of a selective amplifier having an amplification coefficient of 5×10^6 and tuned to a frequency of 72 Hz. A vacuum tube voltmeter served to register the signal amplitude, and an oscillograph was used for visual observation of the signal as well as for phase measurements and for monitoring the shape of the magnetizing pulses. All power supplies were carefully stabilized. To reduce fluctuations, the working voltage of the photomultiplier was lowered to 600 V.

2. MEASUREMENT OF THE THICKNESS OF THE TRANSITION LAYER BETWEEN DOMAINS IN NICKEL AND IRON

As is known, two competing factors—the exchange energy and the effective anisotropy energy—establish a definite thickness of the transition layer between domains.^[2] There are various methods for experimentally checking the structure

of domain boundaries, e.g., the method of powder figures and the electron microscope. A magneto-optical method for investigating domain boundaries, based on the use of the Kerr polar effect, was proposed in^[3] and realized in^[4,5]. An important advantage of this method is that the effect observed in this way is due not to the magnetic field existing at the boundary, but to the magnetization of the substance of the interdomain layer itself. To increase the precision and reliability of the measurements, we used the dynamic method described in the Introduction. The signal source was the change in magnetization of the surface due to movement of the boundary itself.

The spontaneous magnetization vector I_s is rotated by 180° in passing from domain to another over a 180° domain boundary. Because of this, in portions where domain boundaries emerge, there is a normal component of the magnetization on the surface of the ferromagnet. Polarized light reflected from these portions will undergo a rotation of the plane of polarization (the Kerr polar effect), and a pattern of nonuniform illumination will appear in the focal plane of the microscope. If the sample is placed in a small ac magnetic field, the boundary will oscillate about an equilibrium position as a whole within the limits of reversible displacement.

The oscillating magnetic field is produced by two coils supplied from a GZ-34 audio oscillator. The sample was a platelet about 1 mm thick cut from a single crystal of nickel¹⁾ in the (110) plane. The sample was carefully polished mechanically, then heated in vacuum to relieve mechanical strains, and finally polished electrolytically.

As the boundary oscillated relative to the slit of the photomultiplier placed in the focal plane of the microscope, current pulses appeared in the anode circuit. These pulses were fed to the amplifier, which registered a harmonic of 72 Hz. For a fixed slit width, the signal is maximum if the vibration extends from one edge of the slit to the other (Fig. 1, a, b, c). Then if the slit width d_s is equal to the thickness of the image of the boundary d_b , the signal attains its absolute maximum (Fig. 1e). The signal is reduced because of the decrease in the light intensity (background) when $d_s < d_b$ (Fig. 1c), and because of the change in the pulse shape when $d_s > d_b$ (Fig. 1d). When $d_s > d_b$ the signal should be observed only when the field builds up to some threshold magnitude (Fig. 1f). Thus, by varying the

amplitude of the modulating field and the slit width, it is possible to realize the case illustrated in Fig. 1e, and, knowing the magnification of the microscope, one can evaluate the thickness of the transition layer between domains.

Besides the aforementioned reason, reduction of the signal with decreasing slit width may be due to the effect of the resolving power of the microscope. Then the width at which the absolute maximum is attained corresponds to the upper limit of the thickness of the domain boundary.

The regularities enumerated above were actually observed in the investigation of 180° boundaries in nickel. For the condition $d_s > d_b$ a threshold field was observed, the amplitude of which corresponded to the dc field that shifts the boundary to the edge of the slit. Maximum signal was observed for a slit width of about 0.35 mm and a microscope magnification of $1000\times$. Thus the upper limit of the thickness of a 180° boundary in nickel is 0.35μ . A theoretical estimate of the 180° boundary in nickel gives the value 0.206μ .^[6] For the corresponding thickness a value of 6μ was obtained in^[5]; we believe this to be the result of an experimental error.

Analogous measurements were performed on 180° domain boundaries in silicon iron (crystallographic plane (110)). The absolute maximum of the signal was obtained at a slit width of 0.45 to 0.55 mm (magnification $1000\times$) for various boundaries. In order of magnitudes, these values agree with the data of^[4,5]. However, they are markedly greater than the theoretical boundary thickness.^[6] The reasons for this discrepancy between experimental and theoretical results in nickel and iron are not yet clear.

As was mentioned above, a premature decrease in signal with decrease in slit width, i.e., an apparent overestimate of the boundary thickness, may

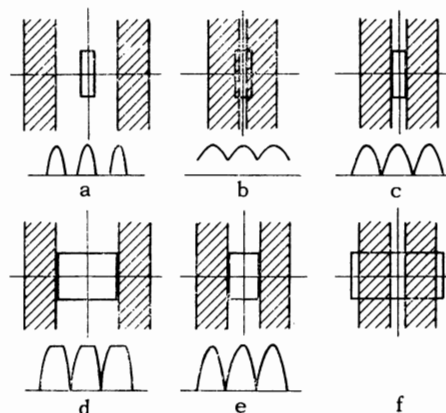


FIG. 1. Change of signal with change of amplitude of modulating field and slit width of the photomultiplier. The extreme positions of the boundary with respect to the slit are shown.

¹⁾The nickel single crystal was kindly placed at our disposal by I. M. Puzey.

be due to the effect of the resolving power of the microscope. However, it seems to us unlikely that the discrepancy between theory and experiment is due to this. In the text are presented data for iron obtained with an objective having an aperture $A = 0.95$ (magnification $1000\times$, optimal slit width $d_s = 0.5$ mm, apparent boundary thickness $d_s = 0.5 \mu$). By using an objective with $A = 0.30$ (magnification $300\times$), one obtains $d_s = 0.3$ mm and $d_b = 1.0 \mu$. For an immersion objective with $A = 1.25$ (magnification $2000\times$), we have $d_s = 1.0$ mm and $d_b = 0.5 \mu$. Thus, use of an objective with a definitely lower resolving power leads to an overestimate of the boundary thickness, but further increase in the resolving power no longer affects the measured value.

3. INVESTIGATION OF THE DOMAIN STRUCTURE OF PERMALLOY FILMS WITH A "SUPERCRITICAL" HYSTERESIS LOOP

Permalloy films that have a thickness not less than a certain critical value h'_{CR} and not greater than another critical value h''_{CR} may have a characteristic "supercritical" hysteresis loop and a banded domain structure.^[7,8] The properties of such films are associated with the appearance of an easy axis of magnetization perpendicular to the plane of the film. Figure 2 shows two models of the domain structure of films with "supercritical" hysteresis loops.

Model a, proposed by Saito, Fujiwara, and Sugita,^[7] assumed the film to be one domain thick. The magnetization vector smoothly changes in direction along the x axis within the limits $\pm \theta$. These authors estimate the lower limit of the thickness of "supercritical" films h'_{CR} to be 1000 to 5000 Å. The theory of this domain structure gives the following dependence of the width of the domains d on the film thickness h : $d \sim h^{1/3}$, whereas experimentally a dependence of the form $d \sim h^{1/2}$ is found. It also follows from this model that the residual magnetization is $I_r > 0.5 I_s$.

Palatnik, Lukashenko, and Ravlik^[8] suggested that the "supercritical" films have a structure of the type b (Fig. 2), corresponding to the Kittel model.^[9] The authors of^[8] also estimate the upper limit of the thickness of "supercritical" films h''_{CR} to be 12μ .

Up until now domain structures have been investigated principally by means of the powder method, which does not allow determination of the direction of magnetization in the domains. We give below the results of an investigation of the domain structure of "supercritical" films by the magneto-optical method described in the introduction.

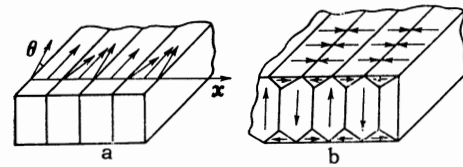


FIG. 2. Models for the domain structure of films with "supercritical" hysteresis loops.

A reversing field of several hundred Oe was provided by a magnetic probe with a gap of about 5 mm fed by an audio oscillator. The oblique incidence of the light beam necessary for observation of the Kerr equatorial effect was obtained by introducing an oblique illumination prism and displacing the field diaphragm relative to the optical axis of the microscope. The measurements showed that in the investigated "supercritical" films (thickness 10μ , domain width 1.8μ) the domain structure on the surface of the sample corresponded to Fig. 2b. This is confirmed by the following group of experiments.

1. The ac magnetic field supplying the magnetic probe is sinusoidal, perpendicular to the plane of incidence (Fig. 3a), and has a frequency $f = 72$ Hz. In this case, an equatorial effect $\delta = 18$ V is observed (the magnitude of the effect is given in terms of the output voltage of the amplifier, since in the calculations we used only the ratios of the magnitudes of the effects for identical photomultiplier background), and for unipolar sinusoidal pulses of the biasing field the effect is $\delta = 9$ V.

2. The ac magnetic field is sinusoidal and parallel to the plane of incidence (Fig. 3b). Then we get $\delta = 0$ for $f = 72$ Hz, and $\delta = 7.5$ V for a unipolar biasing field.

When the input aperture of the photomultiplier is shifted from domain to domain, the phase of the signal at the amplifier output changes, and when the polarity of the pulses of the biasing field is changed, the phase remains unchanged.

When the frequency is halved ($f = 36$ Hz), the signal in the case of a sinusoidal biasing field is $\delta = 8$ V; for unipolar biasing pulses at the same frequency, $\delta = 2$ V.

These experimental results can be explained by the presence of a domain structure of the type shown in Fig. 2b. The inclination angle α of the magnetization vector in contacting domains to the domain boundary can be found from two independent measurements corresponding to the two referred-to directions of the external biasing field relative to the plane of incidence.

In the first case, reversal of the magnetization of the sample by a sinusoidal field makes it possible to obtain the total equatorial effect, in which the

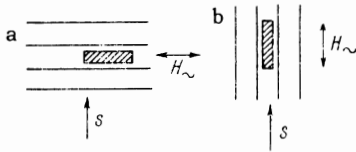


FIG. 3. Orientation of the slit, the biasing field, and the plane of incidence of light relative to the domain boundaries. In case a the equatorial effect is due to the longitudinal component of the magnetization, in case b to the transverse component.

component of the magnetization vector perpendicular to the plane of incidence varies within the limits $\pm I_S$. The magnitude of the effect X_1 in volts is then proportional to the radius of a circle OA (Fig. 4). When unipolar pulses are used, an equatorial effect is observed corresponding to the first harmonic of a pulse having a cutoff angle of 90° , and the amplitude of the effect X_2 is proportional to the segment BC. Thus, $\cos \alpha = (X_1 - X_2)/X_1$. Substituting the experimental data into this formula, we obtain

$$X_1 = 18\epsilon, \quad X_2 = \frac{9}{0.5} = 18\epsilon;$$

$$\cos \alpha = \frac{18 - 18}{18} = 0, \quad \alpha = \frac{\pi}{2}$$

(0.5 is the expansion coefficient for the first harmonic of a pulse with a cutoff angle of 90°).

We now consider the case when the biasing field is parallel to the plane of incidence of the light. For reversal by a sinusoidal field, the signal will have the form sketched in Fig. 5a. If it is assumed that the shape of the pulses is sinusoidal, the corresponding Fourier expansion contains only even harmonics:

$$y = \frac{2}{\pi} - \frac{4}{\pi} \left(\frac{\cos 2x}{1.3} + \frac{\cos 4x}{3.5} + \dots \right) \quad (y = |\sin x|).$$

This explains the absence of signal in the case when the frequency of the reversing field equals the frequency to which the amplifier is tuned. Knowing the magnitude of the second harmonic a , it is possible to determine the amplitude of the pulse

$$X_1 = \frac{3}{4}\pi a = 2.355 \cdot 8 = 18.8 \epsilon,$$

which is proportional to the radius of the circle OA (Fig. 4).

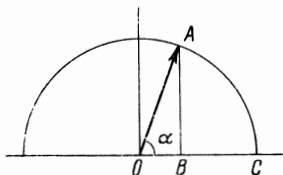


FIG. 4. Diagram for determining the angle of inclination of the magnetization vector of the surface domains to the domain boundary.

For unipolar pulses the effect observed corresponds to the harmonic of pulses having the shape shown in Fig. 5b, and the amplitude X_2 is proportional to the segment AB. Clearly, if the frequency of the magnetizing pulses f_{mag} equals the frequency of the tuned amplifier f_{amp} , the signal at the output of the amplifier corresponds to the second harmonic, and if $f_{\text{mag}} = f_{\text{amp}}/2$, to the fourth harmonic of the input pulses. Assuming that the pulses shown in Fig. 5b are approximately sinusoidal, we find

$$X_2 = 2.355 \cdot 7.5 = 17.6 \epsilon;$$

$$\sin \alpha = \frac{X_2}{X_1} = \frac{17.6}{18.8} \approx 1, \quad \alpha = \frac{\pi}{2}.$$

In this case some error is introduced, since by assuming that these flat-topped pulses are sinusoidal, we have underestimated the true value of X_2 somewhat.

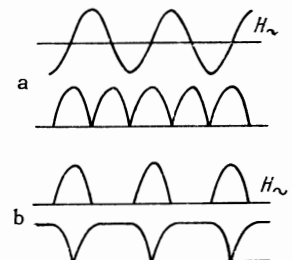
Within the limits of experimental accuracy, no polar Kerr effect was observed. Absence of a polar Kerr effect means that when the magnetization vector reverses it remains in the plane of the film. There is no component of the magnetization normal to the surface of the film, within the limits of accuracy of the measurements.

Similar measurements on films 2.5μ thick (domain width 0.65μ) showed that the domain structure is of the type of Fig. 2b in this case also. The presence in the "supercritical" films that we investigated of a structure of this type does not preclude the realization in certain cases of a structure of the type of Fig. 2a, which is possible as an intermediate in the transition from films with rectangular hysteresis loops to films with "supercritical" loops as the thickness is increased.

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¹G. S. Krinchik, G. M. Nurmukhamedov, and V. P. Zolotarev, PTÉ, No. 4, 171 (1964), Instr. and Exptl. Tech., 890 (1964).

FIG. 5. Shape of the signal upon magnetization of the sample by a sinusoidal field (a) and by unipolar pulses (b) for the case when the magnetic field is parallel to the plane of incidence of the light.



²S. V. Vonsovskii and Ya. S. Shur, *Ferromagnetizm* (Ferromagnetism), Gostekhizdat, 1948.

³G. S. Krinchik, *FMM* **3**, 549 (1956).

⁴L. V. Kirenskiĭ and V. V. Veter, *JETP* **35**, 819 (1958), *Soviet Phys. JETP* **8**, 568 (1959).

⁵M. K. Savchenko and V. I. Sinegubov, *JETP* **44**, 781 (1963), *Soviet Phys. JETP* **17**, 528 (1963).

⁶B. A. Lilley, *Phil. Mag.* **41**, 792 (1950).

⁷N. Saito, H. Fujiwara, and Y. Sugita, *J. Phys. Soc. Japan* **19**, 1116 (1964).

⁸L. S. Palatnik, L. I. Lukashenko, and A. G. Ravlik, *FTT* **7**, 2829 (1965), *Soviet Phys. Solid State* **7**, 2285 (1966).

⁹C. Kittel, *Phys. Rev.* **70**, 965 (1946) (Russ. Transl. in the collection *Fizika ferromagnitnykh oblastei* (Physics of Ferromagnetic Domains), IIL, 1951).

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157