INFLUENCE OF THE THICKNESS OF "SUPERCRITICAL" PERMALLOY FILMS ON THEIR DOMAIN STRUCTURE

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Submitted May 21, 1970

Zh. Eksp. Teor. Fiz. 59, 1177–1187 (October, 1970)

The domain structure of "supercritical" Permalloy films was investigated in the range of thicknesses h from 1 to 170 μ. The domain structure was found to change when h was increased. Rectilinear powder patterns, corresponding to a stripe domain structure, were observed in the $h < 3\ \mu$ range. In the range of thicknesses h from ~3 to ~150 μ, the domain boundaries were curved and the thickest films ($h > 150\ \mu$) had a complex structure with inverse magnetization regions within the principal domains.

ONE of the pressing problems in the modern theory of ferromagnetism is the dependence of the domain structure on the dimensions of a sample, particularly on the thickness h of a ferromagnetic film. It is known[1] that single-domain structure across the thickness is exhibited by ferromagnetic films ($h \lesssim 2000$ Å). The magnetization vector I_S lies in the plane of the film because of the strong geometrical anisotropy of all films. The dimensions of the spontaneous magnetization regions (domains) depend on the conditions during the preparation of a sample and on its preliminary magnetic treatment: these dimensions are in the range 10^2–$10^4\ \mu$.

If, in addition to geometrical anisotropy, a film exhibits transverse anisotropy (the easy magnetization axis is rotated by an angle $\theta \neq \pi/2$ from the normal to the film), the magnetization vector of a film whose thickness exceeds a certain critical value h_c lies outside the plane of the film. This gives rise to what is known as a stripe domain structure in which the domain width D is ~1 μ. Such a structure has been observed in thinned single crystals of cobalt,[2,3] magnetoplumbite,[4] barium ferrite,[5] and other materials. Stripe domain structure has been reported also for "supercritical films" of polycrystalline samples.[6,8] The transverse anisotropy of such polycrystalline films is the result of various structural effects: macrostresses; shape anisotropy of the grains, pores, inclusions, etc.

The stripe domain structure of a magnetic crystal undergoes changes when the thickness h is increased; the domain width D increases and basically new structural elements appear at certain critical values of h. Transition domain structures have been investigated most fully for uniaxial single crystals.[2-5,9] There have been theoretical investigations[3,10-12] in which certain magnetization distribution models have been proposed. Thus, Wyslocki[3] has shown that simple Goodenough and honeycomb structures transform into more complex configurations above a certain critical thickness h_0. In both cases, inverse magnetization domains form in thick films. The dependence $D \propto h^{1/2}$ is obeyed by films of thickness $h < h_0$, whereas thicker films ($h > h_0$) obey $D \propto h^{2/3}$. However, it must be mentioned that the results of investigations of thin single crystals are highly contradictory, and apply only to open domain structures, i.e., to that class of ferromagnets for which $\kappa = 2\pi I_S^2/K_\perp \ll 1$ ($K_\perp$ is the transverse anisotropy constant).

Uniaxial ferromagnets, which have small anisotropy constants (i.e., $\kappa \gg 1$), may have domain configurations with closed magnetic flux paths.[13] According to Lifshitz' calculations,[14] a domain structure of this type in a film whose thickness is above a certain critical value h_c' should also become more complex because of the formation of wedge-shaped regions of inverse magnetization.

There is no published information on the domain structure of ferromagnetic single crystals with $\kappa \gg 1$. In view of this, the "supercritical" films, whose room-temperature anisotropy constant is $K_\perp \approx 10^5$ erg/cm^3 and $I_S \approx 800$ G (i.e., $\kappa \gg 1$), are very interesting to investigate.

We studied earlier the domain structure of "supercritical" Permalloy films of thicknesses h = 1–40 μ.[15] We showed that the domain boundaries in films with $h > 3\ \mu$ become twisted, forming zigzag powder figures on the surface of a sample. However, the wedge-shaped inverse magnetization domains, predicted by E. Lifshitz,[14] have not been observed. In the present investigation, the range of thicknesses is extended to 170 μ. According to Lifshitz,[14] in this range of thicknesses we should observe not only primary but also secondary complications of the domain structure.

SAMPLES AND INVESTIGATION METHODS

Ni–Fe films (initial composition 83 wt. % Ni and 17% Fe) were condensed on polished D16-T Duralumin plates, which were heated to 290°C. The rate of condensation was 20 Å/sec and the vacuum was 10^{-4} torr. This was the optimum vacuum for the formation of the "supercritical" state.[16]

An x-ray diffraction investigation, carried out using a DRON-1 diffractometer, yielded the following results. The structure and composition of the films were different on the two sides of the condensate. The concentration of Ni was 83.6% on the side facing the crucible and 82.4% on the substrate side (these results were obtained for a film 170 μ thick). The dimensions L of the grains were ~2000 and 800 Å, respectively. The level of dis-

orienting microstresses was approximately the same on both sides: about 60 kg/mm^2.

The influence of macrostresses on the magnetic properties of the films was eliminated by separating them from the substrates. The transverse anisotropy in the investigated condensates was due to the microscopic shape anisotropy. The value of $K_\perp$, estimated from the hysteresis loops,[17] was $\sim 5 \times 10^4$ erg/cm^3. Since the saturation magnetization for this type of Permalloy was $I_S \approx 800$ G, we concluded that $\kappa \approx 80 \gg 1$.

The much larger size of the grains on the side facing the crucible was attributed to the relatively high rate of condensation, which favored the coarsening of the structure with increasing h.[15] This structural inhomogeneity of the films resulted in some reduction of the transverse anisotropy constant on the crucible side. The slight difference in the composition of the film on each side had little effect on the energy of the grain shape anisotropy and, consequently, on $K_\perp$.

The domain structure was investigated by the Akulov-Bitter method. A magnetic suspension was prepared in accordance with Elmore's prescription[18] but with some modifications. This suspension was highly sensitive and it enabled us to reveal the fine details of the domain boundaries. The powder patterns were "sharpened" by applying a magnetic field $H_\perp = 200$ Oe at right-angles to the plane of the film. In some cases, the sample was subjected to a preliminary electrolytic polishing. No mechanical treatment of the films (grinding or polishing) was carried out.

Our task was to determine the influence of the thickness on the domain structure of "supercritical" films. This required a new method for preparing films of variable thickness. Usually, samples are prepared from thin single crystals by mechanical treatment, which is followed by annealing. This method is unsuitable for "supercritical" films because the supercritical state is due to a nonequilibrium structure, which is destroyed by annealing. One can also vary the condensate thickness by altering the duration of evaporation of a film. This gives a discrete set of values of h and the results may be nonreproducible because of the differences in the physical and technological conditions which exist during condensation.

In view of these difficulties, we prepared wedge-shaped samples from a plane-parallel film, 170 μ thick. The electrolytic thinning of a film on the side facing the crucible produced a sample whose thickness ranged from ~ 1 to 170 μ in a distance of ~ 1 cm. The opposite surface was polished electrolytically for 30 sec and then coated with a layer of lacquer. The film was held vertically and immersed repeatedly in an electrolyte. Powder figures were observed on both sides of the sample but the observations were carried out on the smoother side facing the substrate because the polishing produced better results on this side. The lacquer layer was removed before the deposition of the magnetic suspension.

Local measurements of the thickness h at various places along the wedge surface presented considerable difficulty. We used the following method in the determination of h. A sample was covered by a thin layer of magnetic suspension, which was then dried. The surface was then painted with a colored ink. The thickness of the film was measured with a mechanical gauge. At the point where the gauge ball came in contact with the film, the ink cracked and we were able to observe the domain structure corresponding to the local value of h. The error in these measurements of h was ± 5 μ.

EXPERIMENTAL RESULTS AND DISCUSSION

1. Figure 1 shows the powder patterns on the surface of a wedge-shaped film after it had been demagnetized parallel to the thin edge of the wedge. The maximum thickness of the sample was 170 μ and the minimum thickness ~ 1 μ. The film was subjected to a magnetic field $H_\perp = 200$ Oe at right-angles to the plane of the film. Therefore, the distance between the stripes represents twice the domain width. We can see that the domain structure of the wedge, demagnetized in a direction parallel to its thin edge, is highly regular—particularly at the thin end. In view of the high regularity of the stripes, a sample of this type can be used successfully to study the dependence of the domain width D on h. Since it is difficult to determine the local thickness, the quantitative relationship between D and h is worth a separate study covering a wide range of film thicknesses.

Figure 1a shows that part of the sample (maximum distance from the edge ~ 100 μ) which extends from h ~ 1 μ to h ~ 10 μ. Zigzag lines appear prominently in the region h > 3 μ. Simple zigzags (Fig. 1a and 1b) are observed in the range of h from ~ 3 to 40—50 μ, in good agreement with the results obtained earlier.[15]

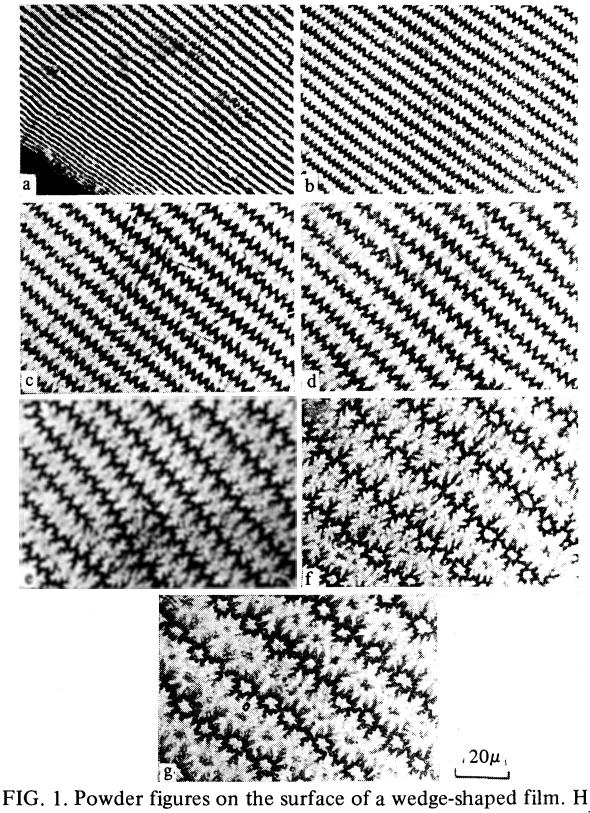

FIG. 1. Powder figures on the surface of a wedge-shaped film. $H_\perp$ = 200 Oe. Film thickness h (μ): a) 1-10; b) 35-40; c) 50; d) 60-70; e) 80; f) 130-150; g) 170.

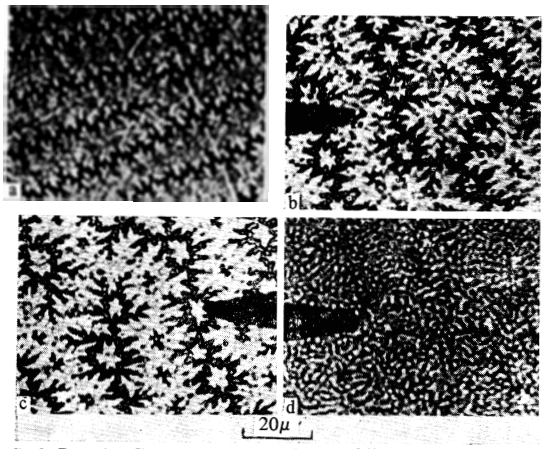

FIG. 2. Powder figures on the surfaces of "supercritical" Permalloy films having a thickness of 80μ (a) and 170μ (c-d), observed in fields: a) $H_\perp$ = +200 Oe; b) $H_\perp$ = +200 Oe; c) $H_\perp$ = −200 Oe; d) $H_\perp$ = 0.

The amplitude of these zigzags C is comparable with the domain width D, and it increases with increasing h.

An additional twist of the amplitude, $C_1 \ll C$, appears in the domain boundaries when the thickness exceeds ~50 μ (Fig. 1c). Figures 1d–1e show the powder figures corresponding to the film thicknesses 60–70 and 80 μ, respectively. We can see that the basic nature of the domain structure remains the same in this range of h, but that the domain width increases and the amplitudes of the macro- and micro-zigzags (C and C_1) become larger.

In the thickness range h = 130–150 μ (Fig. 1f), we can see some rings which are located along the domain boundaries. When h is increased to 170 μ (Fig. 1g), the parts of the domain walls with macro-zigzags, located between the rings, disappear from the pattern.

2. We can use Eq. (44.8) in[19] to estimate the change in the domain width ΔD/D due to the nonparallel orientation of the surfaces of a wedge-shaped sample. For the wedges used in our investigation, we found that ΔD/D < 5%. Nevertheless, we checked the influence of the nonparallelism by investigating plane-parallel films, 1 cm in diameter and 80 or 170 μ thick. Figure 2a shows the powder pattern obtained for a film 80 μ thick. As expected, the pattern is in good agreement with that obtained for a wedge-shaped sample (compare Figs. 1e and 2a). In both cases, macro- and micro-zigzags are observed on the condensate surface and the values of D agree to within ~10%, which is quite satisfactory in view of the errors in the measurement of h and D.

Figures 2b–2d show the powder figures obtained for a film 170 μ thick. The black shadow in these figures is an arrow indicator, which helps to identify a particular part of the layer surface. The patterns shown in Fig. 2b and 2c correspond to opposite polarities of the transverse magnetic field $\pm H_\perp$ = 200 Oe. Figure 2d corresponds to $H_\perp$ = 0. We can see that there are cross-shaped figures between the rings. The arrow in Fig. 2b points to one such cross. When the field $+H_\perp$ is reversed in polarity (Fig. 2c), the point of the arrow is found to lie within a ring and the cross facing the arrow is no longer visible. The centers of the rings observed in $+H_\perp$ are replaced by the cross-shaped figures in $-H_\perp$.

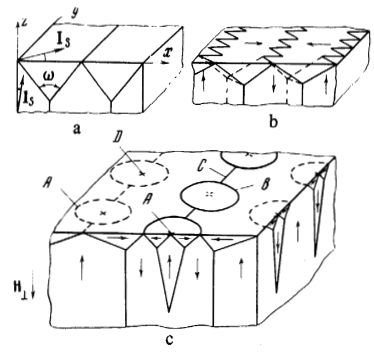

FIG. 3. Models of domain structures of "supercritical" films of various thicknesses h (μ): a) <3 [21]; b) 3–50 [15]; c) 150–170.

A complex pattern is observed in $H_\perp$ = 0 (Fig. 2d).

3. The experimental results presented in Figs. 1 and 2 and those given in[15] can be used to describe the evolution of the domain structure in "supercritical" films when their thickness is increased (Fig. 3). Since $\kappa \gg 1$ for our films, we shall assume that the domain structure is closed. This is in agreement with the fact that the contrast of the powder figures of the investigated films is very poor in the absence of an external field although the magnetization vector of the principal domains is nearly perpendicular to the plane of the film (for films of thickness h > 30 μ the relative remanent magnetization is $I_r/I_S < 0.05$). In the range of thicknesses h from ~1 to 170 μ, the following four types of domain configuration are observed:

a) stripe structure, h < 3 μ;
b) macro-zigzag structure, h = 3–50 μ;
c) macro- and micro-zigzag structure, h = 50–150 μ;
d) complex structure, h > 150 μ.

a) The range h < 3 μ is characterized by a stripe domain structure with planar boundaries between the domains. One of the models described in[20,21] applies to this case. The powder figures on the surface of a film are straight lines parallel to the direction of the preliminary demagnetization. Figure 3a shows the domain structure suggested by Krinchik and Chepurova.[21]

b) When h is increased, the volume V of the closure domains increases and, consequently, the anisotropy energy of the sample becomes greater. We have suggested in[15] that the increase in V is impeded by the change in the angle ω, which becomes greater than 90°. In this case, the condition div I_S = 0 is not satisfied at the 90° boundaries. The energy associated with the resultant volume magnetic "charges" may be reduced by a twist in the domain walls.[22] This produces the domain configuration shown in Fig. 3b. The twisting of the walls gives rise to zigzag powder figures on the surface of the film. A domain structure of this type has been considered by us in detail in[15]. We shall call it the "macro-zigzag structure."

c) When the thickness is h $\gtrsim$ 50 μ, an additional (secondary) twisting of the walls is observed. In addition to the macro-zigzags, whose amplitude is comparable with the domain width, we also observe small-amplitude micro-zigzags. We shall call this domain configuration the "macro- and micro-zigzag structure."

d) A basically new configuration, which we shall call the "complex domain structure," appears in films

whose thickness is 150—170 μ. We shall use the following model to represent this complex structure (Fig. 3c). Since $\kappa \gg 1$, the magnetic flux path is closed. The volume of the closure prisms (and, consequently, the anisotropy energy) may be reduced by the formation of conical inverse-magnetization regions within the principal domains.

In the complex structure, the domain boundaries are twisted (there are micro-zigzags in the boundaries). For the sake of simplicity, these twisted boundaries are shown as straight lines in Fig. 3c. Since the boundaries are "charged," the configuration of leakage fields on the surface is complex if $H_\perp = 0$.

It must be stressed that the models of domain structures of "supercritical" films cannot be regarded as fully established. Additional experiments and theoretical calculations are needed to obtain more information on the distribution of magnetization in uniaxial ferromagnets with $\kappa \gg 1$. This applies to thin films ($h < 3\ \mu$), as well as to thick films, in which the domain structure is more complex.

4. We shall now consider the possible influence of static magnetic fields on the domain configurations of "supercritical" films whose thickness is 150—170 μ.

A. If we apply a field $\mathbf{H}_\perp$ at right-angles to the plane of the film, the vectors $\mathbf{I}_S$ of the closure domains should be deflected from the plane of the film by an angle $\theta = H_\perp/4\pi I_S$.[19] For $H_\perp = 200$ Oe, this angle is $\theta \approx 0.02$ and, therefore, the domain width should change only very slightly (0.04%). Thus, the application of a field $H_\perp = 200$ Oe has practically no effect on the domain configuration of the "supercritical" films. These estimates are in agreement with the observation that the powder pattern configurations remain unchanged when the field $H_\perp$ is increased from ~50 to 200 Oe: only the contrast of the powder figures is increased. The question now arises what should be the distribution of the magnetic suspension when a field $\pm\mathbf{H}_\perp$ is applied.

We shall assume that a field $+\mathbf{H}_\perp$ is directed downward as shown in Fig. 3c. This field weakens the leakage fields at the points denoted by A and enhances the fields above the lines and curves denoted by B and C, as well as above the points D. Thus, we should observe the figures represented by the continuous lines in Fig. 3c: the configuration should include a system of rings B, parts of the boundaries C between these rings, and crosses D. The dashed curves A indicate the changes which should occur when the vertical field is reversed to $-\mathbf{H}_\perp$. Under the influence of fields $\pm\mathbf{H}_\perp$, the changes in the powder patterns predicted by this model are in agreement with the experimental observations (Figs. 2b and 2c).

B. If a thick "supercritical" film has the domain configuration shown in Fig. 3c, a static field **H** of sufficient amplitude and oriented in the plane of the film should alter the distribution of the magnetization near the film's surface. Figure 4a shows schematically the distribution of the magnetization in a surface layer when this longitudinal field is **H** = 0. For simplicity, the centers of the rings in neighboring stripes are shown at the same level (compare with Fig. 3c). The application of a field **H** at right-angles to the stripes (Fig. 4b) increases the volume of those domains whose $\mathbf{I}_S$ vectors are parallel to **H**. If, in addition to the field **H**, we apply a transverse field $+\mathbf{H}_\perp$ and direct this field upward, we should observe the powder figures represented by the continuous lines in Fig. 4b. The rings become elongated toward the left, i.e., opposite to the vector **H**. When the transverse field is reversed from $+\mathbf{H}_\perp$ to $-\mathbf{H}_\perp$ (leaving the amplitude and direction of **H** unaltered), the suspension should become concentrated at the points represented by the dashed curve in Fig. 4b. Thus, the rings should become elongated along **H** if the transverse field is $-\mathbf{H}_\perp$.

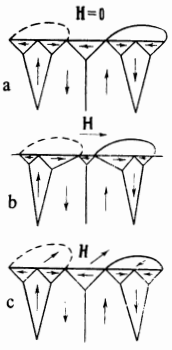

FIG. 4. Influence of a field **H**, lying in the plane of the film, on the domain structure shown in Fig. 3c.

The powder patterns should be affected also by a field **H** oriented parallel to the domains (Fig. 4c). Depending on the mutual orientations of **H** and $\mathbf{I}_S$, the rings in the closure domains should become elongated either along **H** or opposite to this field. The application of a field $+\mathbf{H}_\perp$, oriented upward, should produce the boundaries shown as the continuous lines in Fig. 4c. When this field is reversed to $-\mathbf{H}_\perp$, we should observe the rings elongated along **H** and represented by the dashed curve in Fig. 4c.

The experimental results presented in Fig. 5 are in full agreement with the theoretical model of Fig. 4. The original domain structure (H = 0, $H_\perp = +200$ Oe) is shown in Fig. 5a. The next figure (Fig. 5b) shows the results obtained by applying H = 200 Oe, oriented at right-angles to the stripes, in addition to $+\mathbf{H}_\perp$. The direction of **H** is indicated by the arrow in Fig. 5. We can see that the rings become elongated along the field **H**. When the transverse field is reversed from $+\mathbf{H}_\perp$ to $-\mathbf{H}_\perp$ and **H** is kept the same, we can see rings elongated in the opposite direction (Fig. 5c).

Figures 5d—5f show the influence of a field **H**, parallel to the stripes, on the ring-shaped structure elements. Figure 5d corresponds to the case H = 0 and $H_\perp = +200$ Oe. The next stage (Fig. 5e) represents the influence of $+\mathbf{H}_\perp$ and H = 200 Oe oriented along the domains. In this case, the rings are elongated in the direction opposite to **H**. When the field $+\mathbf{H}_\perp$ is reversed to $-\mathbf{H}_\perp$ (Fig. 5f), we can see the neighboring boundaries where the rings are elongated along **H**.

5. We shall now consider the influence of the edge of a sample on the domain structure of "supercritical" films. If a wedge-shaped sample is demagnetized in a direction perpendicular to the thin edge, we obtain a very irregular domain structure (Fig. 6a). The irregularity of the structure is due to the fact that the domain width D changes with increasing h because of the formation of irregularities known as "magnetic dislocations."[15] This complex structure is not an equilibrium configuration and a study of the dependence of D on h carried out on wedge-shaped films, demagnetized in a

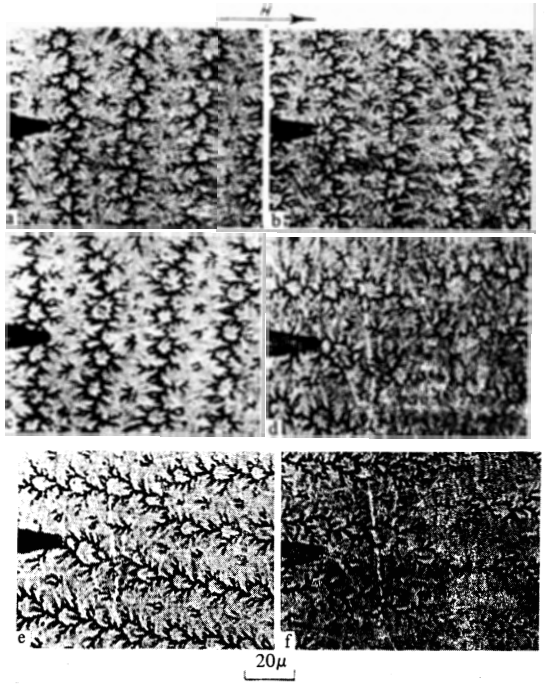

FIG. 5. Powder figures on the surface of a film, 170 μ thick, observed using various combinations of fields **H** (parallel to the plane of the film) and $\mathbf{H}_\perp$ (perpendicular to the film): a) H = 0, $H_\perp$ = +200 Oe; b) H = +200 Oe (oriented at right-angles to the stripes), $H_\perp$ = +200 Oe; c) H = +200 Oe, $H_\perp$ = −200 Oe; d) H = 0, $H_\perp$ = −200 Oe; e) H = +200 Oe (oriented along the stripes), $H_\perp$ = −200 Oe; f) H = +200 Oe, $H_\perp$ = +200 Oe.

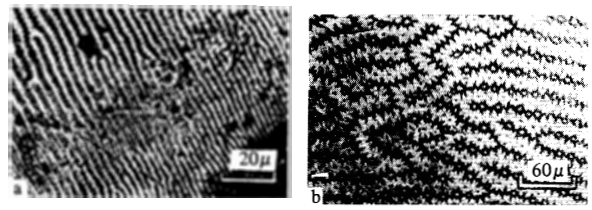

FIG. 6. Departures from the regularity of the domain structure at the edge of a wedge (a) and in the region of h = 170 μ (b); $H_\perp$ = 200 Oe.

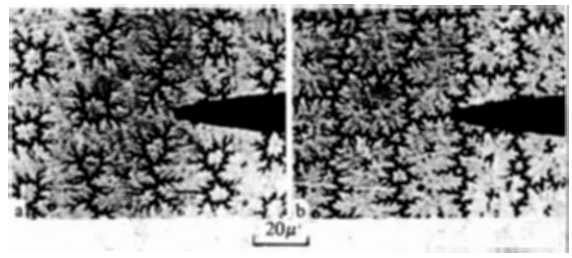

FIG. 7. Departures from the regularity of the domain structure in a film 170 μ thick: a) $H_\perp$ = +200 Oe; b) $H_\perp$ = −200 Oe.

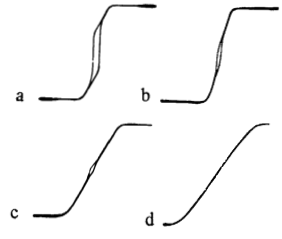

FIG. 8. Hysteresis loops of "supercritical" Permalloy films of various thicknesses h (μ): a) 0.5; b) 5; c) 30; d) 170.

direction perpendicular to the thin edge, may give rise to considerable errors.

The domain structure may be irregular also when the wedge is demagnetized in a direction parallel to the thin edge. In this case, the irregularity is due to the demagnetizing fields at those edges of the film which are perpendicular to the stripes. This is particularly important for films with large values of h (Fig. 6b). We can see that the stripes are now oriented parallel to the edge and this can be explained as follows. The magnetostatic energy of the leakage fields decreases if the vector $\mathbf{I}_s$ is directed parallel to the edge of the sample, i.e., if the magnetic flux path is closed. Since the orientation of the boundaries parallel to $\mathbf{I}_s$ is energetically favorable in the case of stripe domains,[6] the domain boundaries become oriented along the film edge.

The powder patterns near the edge may differ considerably from those near the center of the sample. Figure 7 shows some figures which form a net. Figures 7a and 7b were obtained by applying oppositely directed transverse fields $H_\perp$ = ±200 Oe. The arrow indicator points to the same place on the surface of a film. In a field $+\mathbf{H}_\perp$ (Fig. 7a), we can see some rings. When the field is reversed to $-\mathbf{H}_\perp$ (Fig. 7b), these rings transform to zigzag lines and crosses at the centers of "cells".

It follows from Figs. 6 and 7 that departures from the regularity of the stripes under the influence of the leakage fields at the edge of a film do not give rise to new types of domain boundary. On the other hand, these leakage fields may considerably increase the domain width (by a factor of 1.5 in Fig. 6b) because of the irregularity. This must be allowed for in investigations of the dependence of the domain width on the film thickness.

6. It seemed interesting to determine the switching curves of the films with different domain structures. Figure 8 shows the hysteresis loops of films ranging in thickness from 0.5 to 170 μ. We can see that these loops are basically the same throughout the whole investigated range of h and only the parameters of the hysteresis loop change with increasing h (the remanent magnetization and the coercive force decrease gradually).

The saturation field H_s was determined from Fig. 8 for each of the investigated films. The amplitude of the switching field was the same for all the films: it was equal to 350 Oe. An allowance had to be made for the demagnetization factor $N \approx \pi^2 h/d$, where d is the diameter of the film. For h = 150 μ and d = 1 cm, this factor was N = 0.17, i.e., the demagnetizing field was ~140 Oe. The values of H_s for films of different thicknesses are given in the table below:

h, мк:	0,5	5	35	170
H_s, э:	90	90	110	110

We can see that the saturation field H_s increases slightly when the film thickness is increased from 0.5 to 170 μ.

No sudden change in the parameters of the hysteresis loop were observed at those critical values of h at which the nature of the domain boundaries changed or new structures appeared.

CONCLUSIONS

An investigation of the powder figures on "supercritical" Permalloy films indicated that four domain configurations existed in films whose thickness h ranged from 1 to 170 μ. The stripe domain structure was observed in films of thickness $h < 3\ \mu$. In the range of h from ~3 to ~50 μ, the domains had twisted boundaries ("macro-zigzag structure"). An additional (secondary) twisting of the boundaries was observed in films of thickness $h > 50\ \mu$; this was called the "micro- and macro-zigzag structure". Finally, in films of thickness $h > 150\ \mu$, a new "complex" domain structure was observed. A model explaining the complex structure has been put forward. This model is based on the assumption that the magnetic flux path is closed and that conical inverse-magnetization regions form within the principal domains.

The authors are grateful to V. V. Belozerov for carrying out the x-ray diffraction measurements.

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Translated by A. Tybulewicz
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