Structure of the Intermediate State in Superconductors

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A niobium powder method has been used to display the arrangement of normal and superconducting domains in the intermediate state of a superconductor. Patterns have been observed on samples of tin, indium, lead, vanadium, and tantalum, and have been studied in some detail for tin. It has been found possible to produce patterns which are plane parallel lamina in a flat plate. From their spacing the surface energy at a superconducting-normal interface was evaluated. For tin, the surface energy parameter, Δ, is of the order of 3×10^{-5} cm. Both the magnitude of Δ and its variation with temperature are consistent with theories of its origin advanced by Ginsburg and Landau, Bardeen, and Lewis. Similar results were obtained for indium and lead, but hard superconductors like tantalum, and especially vanadium, give coarser patterns, indicating a very much larger surface energy.

I. INTRODUCTION

WHEN a superconducting metal is cooled below its transition temperature (e.g., 3.7°K for tin), its electrical resistance vanishes. Small magnetic fields are excluded from the body of the sample in this superconducting state. If, however, a sufficiently large magnetic field is applied, superconductivity is destroyed. Then the electrical resistance of the sample reappears, and magnetic flux penetrates it. The value of the critical field varies ordinarily from zero at the transition temperature to a few hundred oersteds at 0°K.

The transition in a magnetic field occurs abruptly only if the effective field is constant at all parts of the surface of the sample. In practice, an abrupt transition requires that the sample be a long rod or wire with its axis parallel to the field direction. For any other shape or orientation of sample, the effective field will reach the critical value at some places while the applied field is still somewhat lower.

Consider, for instance, the short cylinder shown in cross section in Fig. 1. At very low fields all the lines of force go around the outside of the sample as in Fig. 1(A). The greatest flux concentration is at the corners, i.e., the circumference of the ends, and penetration begins there when the local field reaches the critical value. This leads to a situation like Fig. 1(B), where the edges of the sample are normal and have flux passing through them while the central region remains superconducting.

This condition is, however, not stable. For stability, the local field would have just the critical value on the boundary, and be somewhat larger in the normal region. But in a configuration like that of Fig. 1(B), the largest fields must occur right on the boundary, while farther from the center they must be smaller. This follows from Maxwell's equations, because the field lines are concave inward.[1]

What must happen is that the sample breaks into a number of regions, some of which are normal and contain flux while others are superconducting and exclude flux. The smaller these domains, the less will be the field energy, so that field energy tends to make the domains small. But every time domain size is decreased by the formation of new domains, there is an increase in the area of surface between normal and superconducting regions. Thus a finite surface energy limits the smallness of the domains and, in combination with the field, determines domain size and configuration. Conversely, in favorable situations, the surface energy can be deduced if the domain pattern is known.

The intermediate state of a superconductor is, then, composed of some mixture of superconducting and normal regions.[2] The size, shape, and arrangement of these domains have for the most part been inferred from indirect evidence such as the shape of magnetization curves. The only direct investigation was that of Meshkovsky and Shalnikov,[3–5] who used a bismuth microprobe to map the distribution of magnetic field in the equatorial plane gap between tin hemispheres and on the surface of a sphere. In both places domains

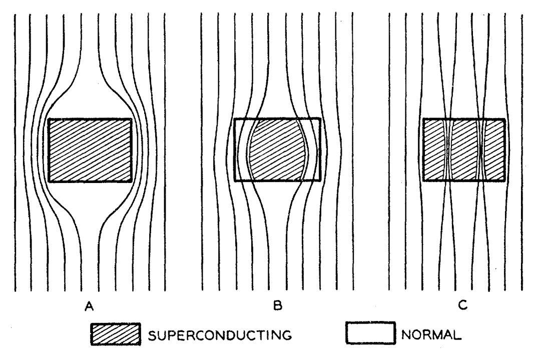


FIG. 1. Flux distribution around a short cylinder. (A) Low field, no penetration; (B) larger field, penetration at edges; (C) intermediate state with field passing through normal regions.

[1] See, for instance, D. Shoenberg, *Superconductivity* (Cambridge University Press, Cambridge, 1952), pp. 24–25.

[2] See D. Shoenberg, reference 1, Chap. IV.

[3] A. G. Meshkovsky, J. Exptl. Theoret. Phys. (U.S.S.R.) **19**, 54 (1949).

[4] A. G. Meshkovsky and A. I. Shalnikov, J. Phys. U.S.S.R. **11**, 1 (1947).

[5] A. G. Meshkovsky and A. I. Shalnikov, J. Exptl. Theoret. Phys. (U.S.S.R.) **17**, 851 (1947).

of macroscopic size were observed, indicating that not much branching took place along the field direction. This technique is clear and direct, but extremely laborious, so that only a very few maps were obtained. It was not possible to explore a wide range of samples, temperatures, and fields.

In the present investigation, a magnetic powder has been used to display the structure of normal and superconducting domains on the surface of various samples.[6] The powder is spread on the surface of the specimen. When a magnetic field is applied to produce the intermediate state, the powder is disturbed at normal regions which contain field. In this way the entire field pattern is observed at once. It is therefore practical to study the distribution of normal and superconducting domains in a variety of materials and shapes under different temperature and field conditions.

As will be seen below, in some circumstances the domain patterns are fairly simple. From these the surface energy at the boundary between the normal and superconducting phases can be determined in a direct manner.

II. EXPERIMENTAL TECHNIQUE

The first candidate for use as a magnetic powder is obviously a ferromagnetic material such as iron or permalloy. While such materials can provide useful information, they have serious objections. There are large local fields around individual grains, which might disturb the distribution of superconducting domains. Moreover, the large forces between the particles cause them to form chains and other patterns even in a fairly simple field, as around a bar magnet.

We have therefore used a diamagnetic powder in these experiments. A diamagnetic powder is expelled from normal regions of the surface, through which flux passes. It remains in the superconducting regions. Thus as one looks at the sample, its shiny surface shows up the normal regions, while powder covering the superconducting areas leaves them relatively dark.

Probably the only materials with sufficiently strong diamagnetism are the superconductors themselves. In the superconducting state, they are perfectly diamagnetic, having essentially zero permeability. We have therefore used niobium powder, as this material has a transition temperature of around 8.5°K and a critical field of several thousand oersteds at 0°K. Thus at all temperatures and fields needed for most soft superconductors, the niobium powder remains perfectly diamagnetic.

For high resolution the powder should be as fine as possible, but very fine powder shows some tendency for the grains to stick together and form clumps. In most of this work 200-mesh niobium powder was used. The grains had dimensions of the order of 0.06 mm, although they were by no means spherical. This powder was fine enough so that the grain sizes were negligible in comparison with the dimensions of most of the patterns observed. The grains were also a negligible fraction of the thickness of most of the samples.

As compared with a ferromagnetic material, the niobium powder has a lower susceptibility, with consequently reduced stray fields and interparticle forces. It is possible to make the niobium particles form chains of force along magnetic field lines, but it is quite difficult. Ordinarily the particles respond quite independently to the field. The diamagnetic powder has a further characteristic advantage, in that its equilibrium position is in regions of zero field, where it is subjected to no force. It therefore should cause negligible disturbance of the superconductor on which it is spread.

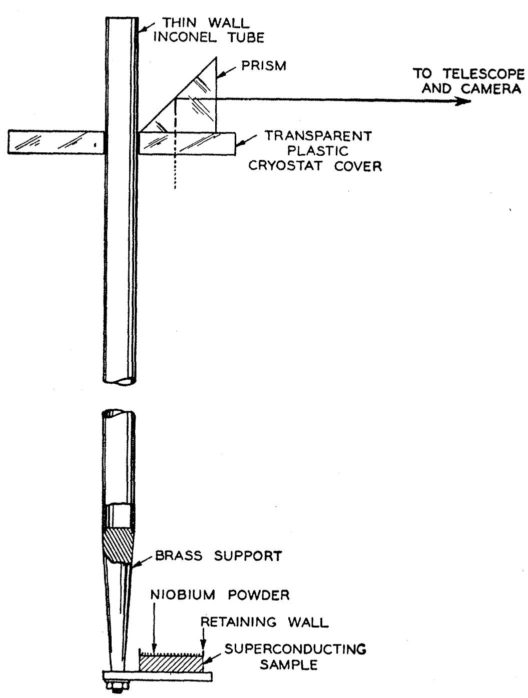


FIG. 2. Apparatus for observing intermediate state domain patterns.

Apparatus

The experiments were performed in a cryostat which was largely designed by E. Corenzwit. The helium Dewar has an inside diameter of 2 inches, so that samples up to this size can be accommodated. Immersed in the liquid nitrogen in the outer Dewar, there is a solenoid which gives a vertical field of about 80 oersteds per ampere. A field up to 3000 oersteds is obtainable. This field is uniform within about 2% over a radius of 2 cm from the axis of the solenoid.

On the top of the cryostat there is a cover plate of transparent plastic. Through an *O*-ring seal in the cover plate passes a thin-walled $\frac{1}{2}$-in. metal tube which carries the specimen platform at its lower end (Fig. 2). The

[6] Schawlow, Matthias, Lewis, and Devlin, Phys. Rev. **95**, 1344 (1954).

sample is attached lightly to this platform (usually by rubber cement). It carries around it a nonsuperconducting retaining wall to confine the magnetic powder to the specimen area.

Usually a thin layer of powder is spread over the surface of the specimen before putting the top onto the cryostat. If the powder is not uniformly distributed, it is sometimes possible to spread it magnetically. This is accomplished by warming the specimen above its transition temperature and applying a field of several hundred oersteds. Then the powder grains become polarized by the field and repel each other, so that they spread over the available area. This operation is helped by lowering the solenoid (which is arranged so that it can be moved vertically). Then the sample is near the top of the coil, and the diamagnetic powder is in a region of field which decreases upward. The powder experiences some upward force which aids in overcoming friction, so that it can move over the surface.

After spreading, the field is removed and the system is cooled to the working temperature. If this is below the helium λ temperature, the sample can be observed clearly and photographed under steady conditions. Above the λ temperature, bubbling has to be discouraged by momentarily applying an overpressure of helium gas to permit observation. The overpressure must not be left on for a long time, or the temperature will rise by an uncertain amount.

On the transparent cryostat top, a right-angle prism reflects light from the specimen to a telescope. The eyepiece of this telescope gives a virtual image which is focussed by the camera lens. Most of the photographs were taken with a Speed Graphic camera, using a Polaroid back.

III. EXPERIMENTS ON STRUCTURE

(1) Tin Disk (Short Cylinders with Field Parallel to Axis)

The first sample investigated was a disk or short cylinder of polycrystalline tin, 2 cm in diameter and 1.4 cm thick. This is a sort of flat-faced approximation to the sphere investigated by Meshkovsky and Shalnikov, and corresponds to the cylinder described in the introduction. From etch patterns, the grain size in this sample was of the order of 1 mm.

Figure 3 shows the surface of this cylinder when a field of 0.45 H_c is applied. The dark niobium powder, which had originally covered the entire surface of the shiny disk, has moved in around the circumference. Magnetic field has penetrated the edges of the disk, and we have the unstable configuration of Fig. 1(b). Thus if the disk is now tapped, powder moves freely in the inner region, but will not move out near the edge.

As the field is increased, it breaks into the disk in irregular regions, as shown in Fig. 4. Bare regions, which are normal and contain flux, are surrounded by darker superconducting regions on which the powder remains. With a further increase, the pattern becomes more regular (see Fig. 5), and the superconducting domains are generally radial with a fairly regular spacing. The relative simplicity and regularity of this pattern indicates that it should be possible to obtain surface energies quantitatively from domain patterns.

Above the critical magnetic field, the disk is once more covered nearly uniformly by the powder (Fig. 6). Its general appearance is brighter, however, as now the nonspherical grains of powder orient themselves along the field direction. Thus the fraction of the disk area covered by powder is less than in zero field, and so more light is reflected. In some cases, also, several grains may form short chains along the field direction, further reducing the covered area.

When the field is switched off, a radial pattern appears which develops over an interval of a half-minute or so (Fig. 7). Its formation is marked by sudden movements of powder grains in one part of the disk, followed later by movement somewhere else. It appears that flux escapes first from the edge of the disk, leaving a superconducting ring around the periphery. The flux remaining in the interior breaks out from different places at different times. The escape process seems to be quite different from that postulated by Faber and Pippard[7] for a long single-crystal cylinder, where all the flux is thought to escape through one gap.

Unlike all the other patterns shown, that of Fig. 7 is not stable, but represents the effect of transient events during the escape period. That is, if the disk is agitated by tapping, some of the bare spaces are covered by powder. Since this cylinder was polycrystalline and possibly also because it was short,[8] a little of the flux remained in it even when the applied field was removed (Fig. 8).

Radial patterns resembling those of Fig. 7 were obtained with lead powder in the gap between two tin hemispheres by Meshkovsky and Shalnikov.[4] In their arrangement, the magnetic field and the top hemisphere had to be removed before the pattern could be observed. Thus it was not possible for them to use powder to examine the domain pattern at a particular field. They could observe only the transient escape pattern, although the distinction does not seem to have been made between this and the bismuth microprobe patterns.

(2) Long Bar

Flux and resistance measurements on wires in a transverse magnetic field[9] have shown that resistance begins to reappear when flux begins to penetrate the wire. It was therefore postulated that, for a wire in a transverse field, the domains are also transverse so as to leave no longitudinal superconducting paths.

[7] T. E. Faber and A. B. Pippard in *Progress in Low Temperature Physics* (North-Holland Publishing Company, Amsterdam, 1955), Vol. 1, p. 159.
[8] D. Shoenberg, reference 1, p. 35.
[9] D. Shoenberg, reference 1, p. 111.

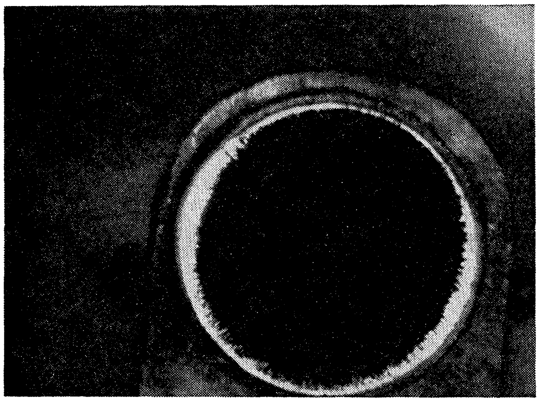


FIG. 3. Niobium powder pattern on the surface of a short tin cylinder. $T=1.82°K$, $H=0.45\ H_c$. Flux has penetrated the edge of the sample, displacing the niobium powder and exposing the bright tin surface.

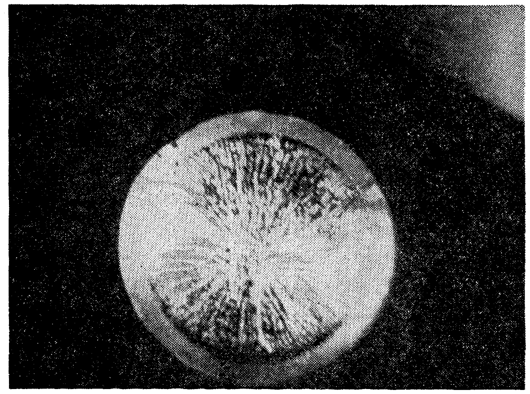


FIG. 5. Powder pattern on a short tin cylinder. $T=1.83°K$, $H/H_c=0.72$.

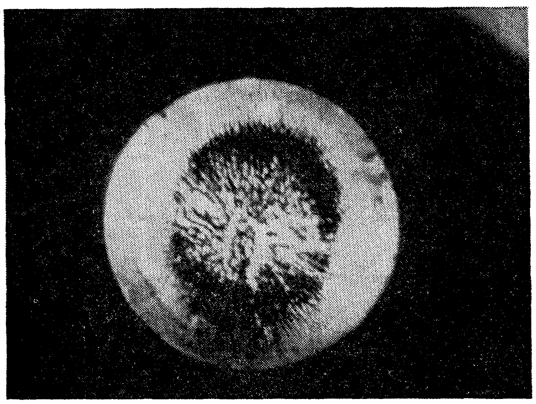


FIG. 4. Powder pattern on a short tin cylinder. $T=1.83°K$, $H/H_c=0.67$.

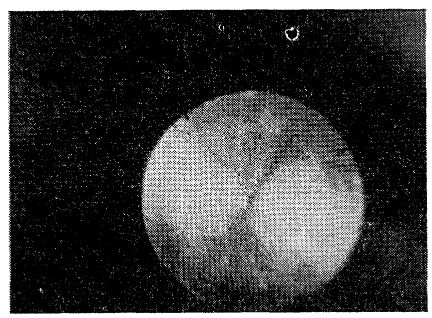


FIG. 6. Powder pattern on a short tin cylinder with field greater than critical. $T=1.83°K$, $H/H_c=1.06$.

As an approximation to a long wire, powder patterns were observed on a tin rod whose length, 45 mm, was much greater than its diameter, 9 mm. As seen in Fig. 9, the pattern consists of transverse domains. Thus we have direct confirmation of the domain structure previously inferred from resistance measurements. The central longitudinal line represents the junction of penetration regions originating at the two edges.

It was feared at first that patterns could not be observed on other than flat surfaces, because the powder would slide off. However, when a superconducting rod with its axis horizontal is subjected to a vertical field, the rod's demagnetization produces a larger field at the edge than on top of the rod. Thus the diamagnetic powder experiences a small force toward the center of the rod's upper surface which opposes gravity.

IV. FLAT PLATE: MEASUREMENT OF SURFACE ENERGY

To evaluate the surface energy, one needs a quantitative relation between it and domain size. For a particular sample geometry and magnetic field strength, the domain size is determined by balancing magnetic field energy against surface energy. The smaller the domains, the less distorted is the magnetic field, and the less its energy. But if the domains are small, the interfacial surface area between normal and superconducting regions is large.

The field energy is difficult to calculate for even the simplest configurations. The domain shape determines the demagnetizing factors and through them the field energy. Even the rounding of the domains near the surface is important for this calculation. Landau[10] has shown how this can be done if the domains are straight, parallel laminas in an infinite flat plate. Lifshitz and Sharvin[11] have calculated numerical values for the spacing, for all values of field between 0 and H_c. They show that

$$a=(d\Delta/\Phi)^{\frac{1}{2}}, \tag{1}$$

where a is the spacing between domains, d is the thickness of the plate, Δ is the surface energy parameter,

[10] L. Landau, Physik. Z. Sowjetunion **11**, 129 (1937).

[11] E. M. Lifshitz and Yu. V. Sharvin, Doklady Akad. Nauk S.S.S.R. **79**, 783 (1951).

TABLE I. $\Phi=n^2d\Delta$, where n is the number of domains per centimeter, d is the plate thickness, and Δ is the surface energy parameter (from Lifshitz and Sharvin[11]).

H/H_c	Φ	H/H_c	Φ
0	0.0000	0.6	0.0182
0.1	0.0055	0.7	0.0128
0.2	0.0136	0.8	0.0065
0.3	0.0195	0.9	0.0020
0.4	0.0224	1.0	0.0000
0.5	0.0221		

Φ is a function of H/H_c calculated numerically and tabulated in Table I. The surface energy, in ergs per cm², is $\Delta\cdot 8\pi H_c^2$. Thus, Δ has the dimensions of length and is measured in cm or angstroms.

We can approximate to the geometry of this model by using a plate whose thickness is much less than its length and width. To help ensure that the domains be parallel straight lines, the plate should also have a width less than its length.

These conditions were realized experimentally with plates $\frac{1}{2}$ in. wide, $1\frac{3}{4}$ in. long, and $\frac{1}{8}$ in. thick. When such a plate was placed in a field normal to its surface, the field first broke in at the edges, as with the disk. When the applied field was increased beyond about $\frac{1}{2}$ H_c, the flux broke into the central section of the plate, forming the expected laminar pattern (Fig. 10).

Under these circumstances, the intermediate state does consist of parallel, nearly straight domains.

According to Eq. (1), n^2, the square of the number of domains per cm, should be proportional to Φ, and so should decrease as H approaches H_c. This dependence of domain spacing on field is confirmed by the experiments both qualitatively and quantitatively. Figure 11 shows the results of several series of measurements on polycrystalline tin samples at temperatures near 1.85°K. The solid curve is drawn from Eq. (1) assuming a value of $\Delta=3150$ A. It is to be noted that there is only one adjustable parameter, and the goodness of the fit obtained is very satisfactory.

From the dotted curves in Fig. 11 it is evident that a change of 10% in the value assumed for Δ would give a markedly poorer fit. One might be justified in ascribing an error of $\pm 10\%$ to the value of Δ derived in this way. However, the patterns are not ideally perfect parallel straight lines, so that there is still some sub-

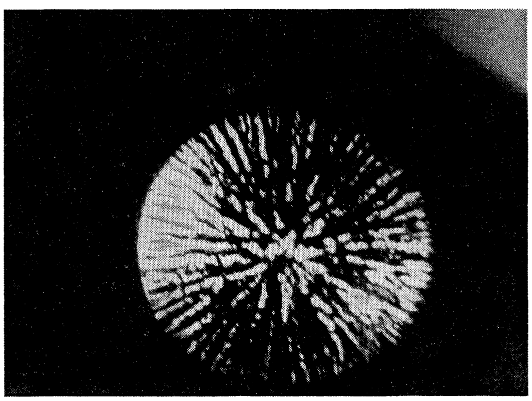


FIG. 7. Powder pattern on a short tin cylinder. $T=1.83$°K; field reduced to zero from above critical. This is the unstable pattern produced as flux escapes.

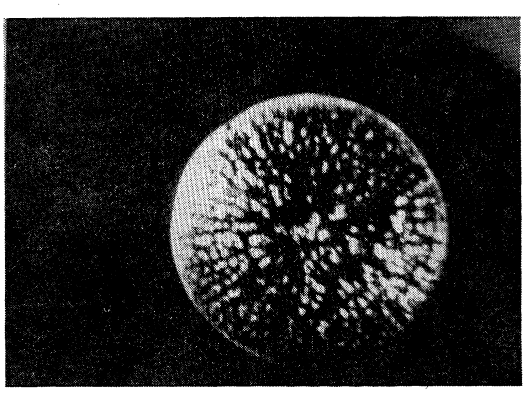


FIG. 8. Powder pattern on a short tin cylinder. Same conditions as Fig. 7, except that disk has been tapped to overcome friction. This pattern shows flux trapped by the polycrystalline cylinder.

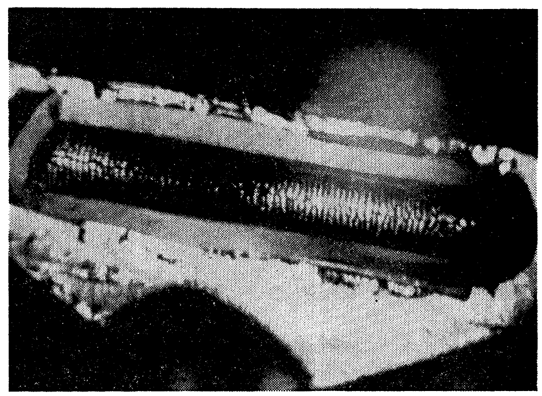


FIG. 9. Transverse domains on a tin rod. 1.77°K, $H/H_c=0.72$.

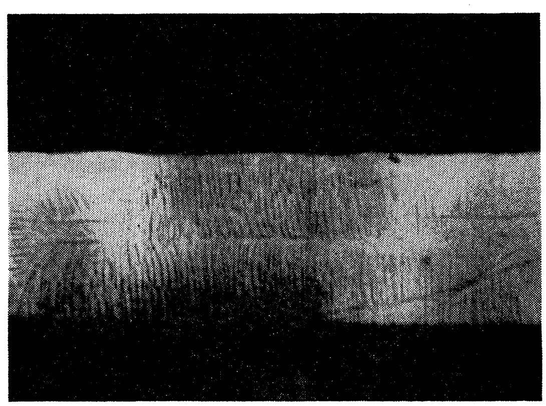


FIG. 10. Powder pattern on a flat tin plate showing laminar domains. $T=1.93$°K, $H/H_c=0.82$.

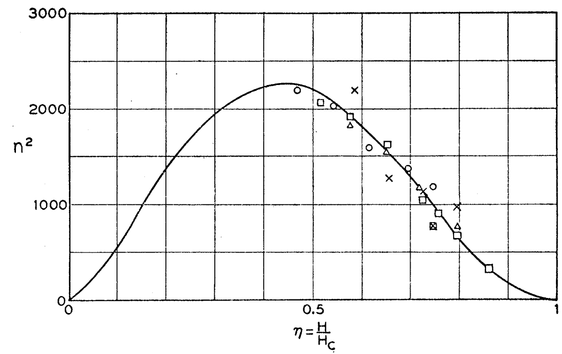


FIG. 11. (Number of domains per cm²) as a function of H/H_c for flat tin plates 0.32 cm thick. $T=1.86°$K.

jective element in counting the number of domains per cm. It therefore seems best to assume for the present an error of $\pm 10\%$ in n, with a resulting error of $\pm 20\%$ in Δ.

Some observations were made on plates of different thicknesses. The thickness cannot be increased much without increasing the other dimensions if the sample is to approximate a thin plate. Since the inner diameter of the cryostat is only 5 cm, it is not possible to increase the size of the sample much. Samples as thin as $\frac{1}{16}$ in. were tested, and it was confirmed that the domain spacing was less than for the $\frac{1}{8}$-in. plate. However, the pattern spacing was now comparable with the limit of resolution imposed by the size of the powder grains. Thus the $\frac{1}{8}$ in. thickness was used for all quantitative work.

Figure 12 shows the surface energy of tin at various temperatures as determined in this way. The solid curve is calculated by Lewis[12] taking into account the kinetic energy arising from localizing electron wave functions at the boundary.[13,14] Agreement is good, particularly at the higher temperatures. Thus surface energy measurements are consistent with the assumption that this localization is the real source of the surface energy. At lower temperatures ($T \ll T_c$), the measured surface energy is somewhat less than that calculated. This discrepancy may be cause by an energy gap between the superelectron levels and the nearest available states into which they can be excited. The observed values of Δ are not very different from those determined from phase boundary propagation velocity by Faber.[15]

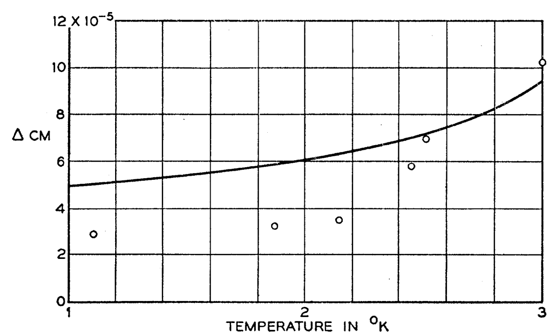


FIG. 12. Surface energy parameter for a superconducting-normal interface in tin as a function of temperature. The solid curve was given by the theory of H. W. Lewis.

Surface Energy of Vanadium

Domain patterns were also observed on samples of vanadium, which is a typical hard superconductor. The theory of surface energy, which applies fairly well to tin, indicates that $\Delta H_c \lambda \sim$ a constant.[16] It might be expected that vanadium, having a higher value of critical field, would have a lower surface energy parameter than tin. Instead of this, the experiments show that vandium has a surface energy very much larger than that of tin.

Figure 13 shows typical powder patterns on a 0.24 cm thick sample of polycrystalline ductile vanadium. The material used was 99.7% pure vanadium (impurities 0.07% carbon, 0.12% oxygen, 0.003% hydrogen, and 0.09% nitrogen), and was quite ductile and machineable.

In a tin sample of this thickness, only a little penetration at the edges occurred before the break-through into a lamina pattern began. In vanadium, as seen in Fig. 13(a), the field penetration at the edges goes almost to the center before anything approaching transverse domains appears. However, when the field is raised slowly, large domains appear very abruptly, as in Fig. 13(b). Each of the indentations of Fig. 13(b) appeared practically instantaneously, with a very small increase in field.

It is apparent from these observations, that the domains in vanadium are considerably larger than in tin for the same sample thickness. The domain size is reduced in thinner samples, but even in a foil 0.0025 cm thick (Fig. 14) it is still much larger than in the original tin plate. Equation (1) is not really applicable here, as the domains of Fig. 14 are by no means narrow in comparison with the plate thickness. Rough estimates of the normal-superconducting surface energy in vanadium from these patterns show that it is at least a thousand times greater than in tin.

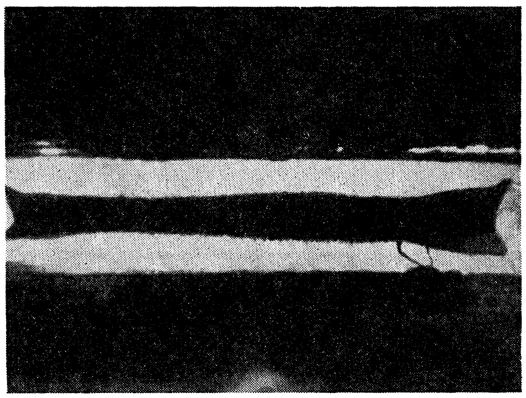
Such a large surface energy is entirely outside the range of values which might come from localizing electron wave functions in the way considered by Ginsberg and Landau, Bardeen and Lewis. There is, as yet, no explanation for this additional source of surface energy. However, a very large surface energy would explain much of the characteristic behavior of hard superconductors.

[12] H. W. Lewis (to be published).
[13] V. L. Ginsburg and L. D. Landau, J. Exptl. Theoret. Phys. (U.S.S.R.) 21, 979 (1950).
[14] J. Bardeen, Phys. Rev. 94, 554 (1954).
[15] T. E. Faber, Proc. Roy. Soc. (London) A233, 174 (1954).
[16] J. Bardeen, Eqs. (4.22) and (4.23).

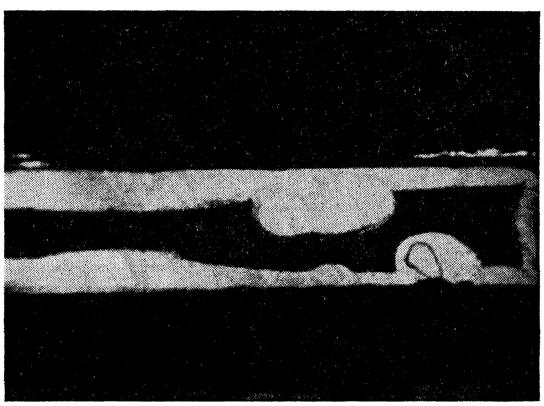
Domain Patterns and Surface Energy of Other Materials

A few preliminary observations have been made on samples of indium, lead, niobium, and tantalum. It is found that the surface energy in indium is comparable with that of tin and in lead is somewhat less. Tantalum and niobium also have a much larger surface energy than these soft superconductors.

All of the specimens described in this paper were polycrystalline. They had, therefore, plenty of nuclei for the formation of superconducting domains. Thus the number of domains was not limited by the lack of nuclei. Some preliminary studies of single tin crystals have been made. Patterns resembling those on polycrystalline tin have been observed, but other complications due to anisotropy in the normal resistance were found. Further studies using oriented single crystals are in progress.



(a)



(b)

FIG. 13. Powder patterns of the intermediate state in vanadium. (a) 0.24 cm thick, 0.95 cm wide, 4.44 cm long, $T=2.15°K$, $H/H_c=0.61$; (b) same sample as (a), $T=2.15°K$, $H/H_c=0.81$.

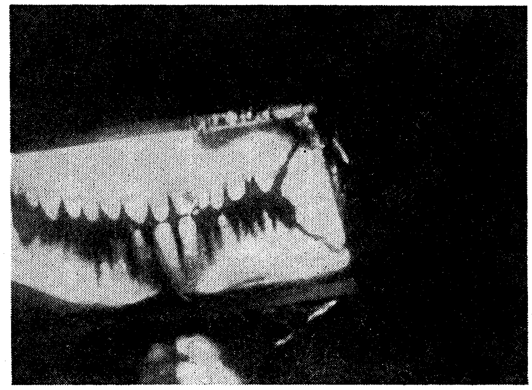


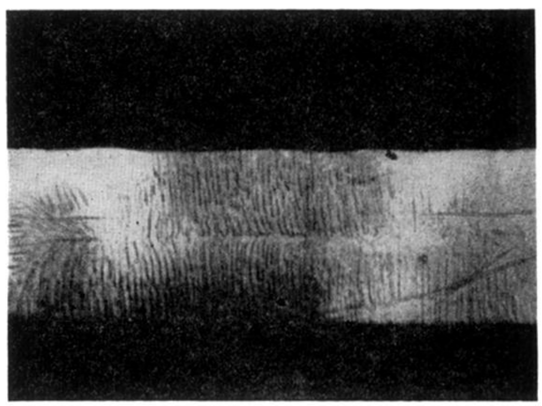
FIG. 14. Powder pattern of the intermediate state in vanadium. Sample 0.0025 cm thick, other dimensions similar to those of Fig. 13, $T=1.80°K$, $H/H_c=0.45$.

V. CONCLUSIONS

The magnetic powder method has shown the arrangement of normal and superconducting domains in various samples. The method can also be used for quantitative measurements of surface energy. Further investigations of surface energy in various materials are in progress and conclusions must be only qualitative at this stage. It is evident, however, that the Ginsburg-Landau-Bardeen-Lewis extension of the phenomenological theory to take into account electronic wave function localization, is at least qualitatively correct for soft superconductors. For the hard superconductors, there must be some different, and much larger, source of surface energy.

ACKNOWLEDGMENTS

This research has benefitted greatly from numerous helpful discussions with Dr. H. W. Lewis. E. Corenzwit assisted in the cryostat design. Many of the experimental details and measurements are due to G. E. Devlin's skill and ingenuity.



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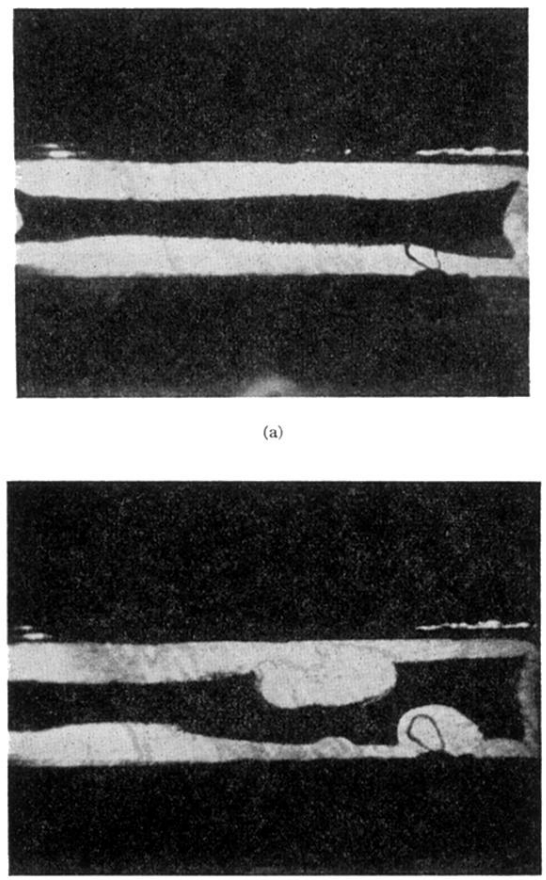

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(b)

FIG. 13. Powder patterns of the intermediate state in vanadium. (a) 0.24 cm thick, 0.95 cm wide, 4.44 cm long, $T=2.15°K$, $H/H_c=0.61$; (b) same sample as (a), $T=2.15°K$, $H/H_c=0.81$.

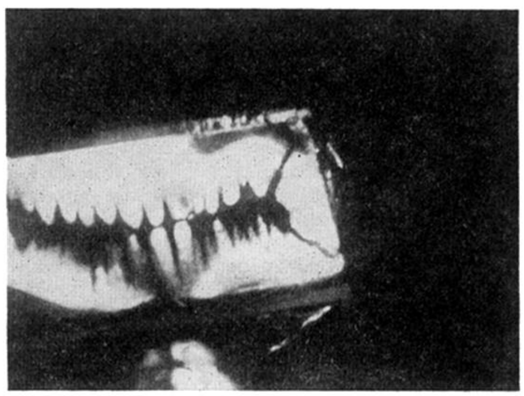

FIG. 14. Powder pattern of the intermediate state in vanadium. Sample 0.0025 cm thick, other dimensions similar to those of Fig. 13, $T=1.80°K$, $H/H_c=0.45$.

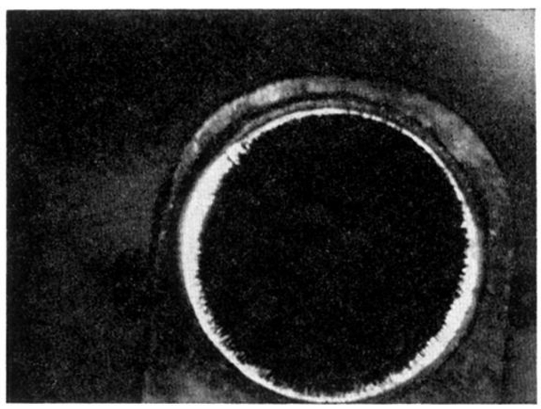

FIG. 3. Niobium powder pattern on the surface of a short tin cylinder. $T=1.82°K$, $H=0.45\ H_c$. Flux has penetrated the edge of the sample, displacing the niobium powder and exposing the bright tin surface.

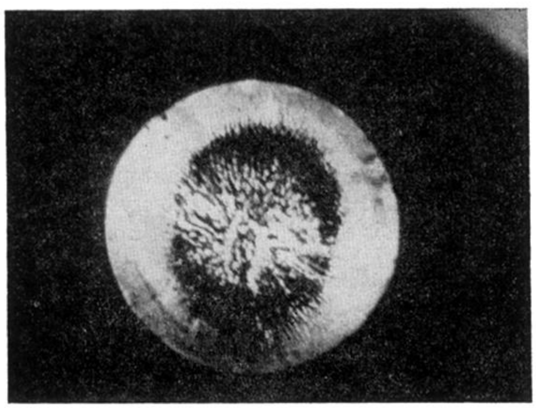

FIG. 4. Powder pattern on a short tin cylinder. $T=1.83°K$, $H/H_c=0.67$.

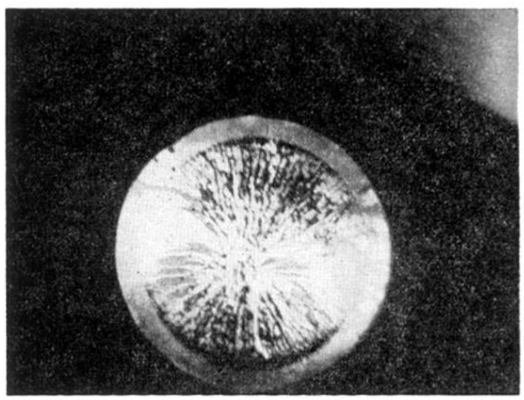

FIG. 5. Powder pattern on a short tin cylinder. $T = 1.83°K$, $H/H_c = 0.72$.

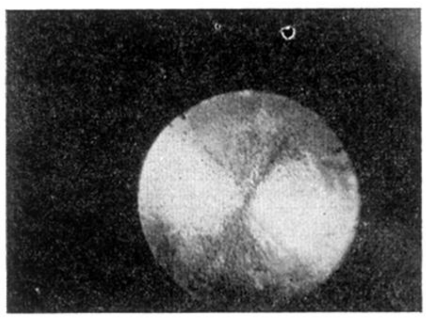

FIG. 6. Powder pattern on a short tin cylinder with field greater than critical. $T=1.83°K$, $H/H_c=1.06$.

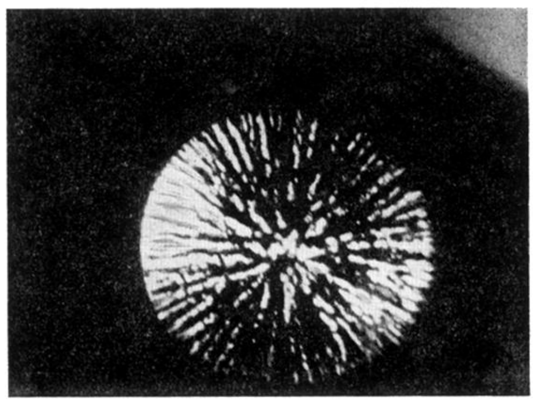

FIG. 7. Powder pattern on a short tin cylinder. $T=1.83°K$; field reduced to zero from above critical. This is the unstable pattern produced as flux escapes.

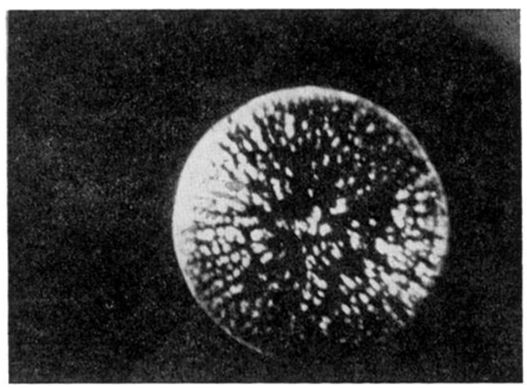

FIG. 8. Powder pattern on a short tin cylinder. Same conditions as Fig. 7, except that disk has been tapped to overcome friction. This pattern shows flux trapped by the polycrystalline cylinder.

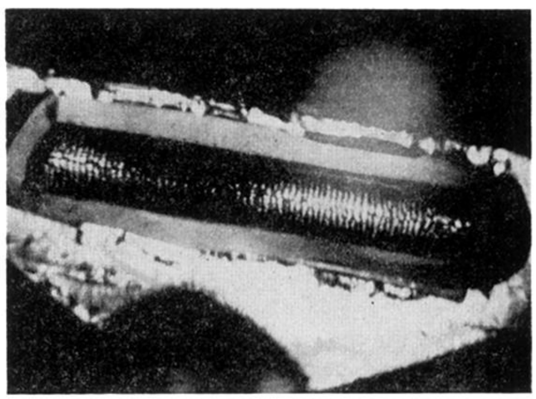

FIG. 9. Transverse domains on a tin rod. 1.77°K, $H/H_c=0.72$.