Superconductivity of a layer of microscopic twin crystals

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A macroscopic current has been observed to flow along the polished end of a tin sample at $T > T_c$. The superconductivity of a layer $\sim 4 \times 10^{-4}$ cm thick is attributed to a three-dimensional array of regions of $\langle 301 \rangle$ twinning planes, separated by distances $\sim 10^{-5}$ cm. A superconductivity of the "two-dimensional metal" near such plane has been observed previously {M. S. Khaĭkin and I. N. Khlyustikov, Pis'ma Zh. Eksp. Teor. Fiz. **33**, 167 (1981) [JETP Lett. **33**, 158 (1981)]}. The same method has now been used to observe a superconductivity of $\langle 101 \rangle$ twinning planes in indium twin crystals.

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In some previous experiments[1] we observed a superconductivity of a "two-dimensional metal," in this case a twinning plane in a metal crystal. A superconductivity arising in a thin layer of a bicrystal adjacent to the (301) twinning plane was detected through the appearance of a magnetic moment M_D of a tin sample at a temperature[1)] above the critical temperature for the crystal, $T_c = 3.722$ K. The moment M_D increases exponentially as T_c is approached. Furthermore, in some earlier experiments[2] we had observed an increase (of ~10%) in the electrical conductivity of a strained sample under the same conditions which resulted in the observation of the moment M_D. In Ref. 1 we argued that the familiar increase in the value of T_c of a metal caused by deformation might be a consequence not so much of mechanical stress (as in the usual interpretation) as of the appearance of twins in the sample.

In the present experiments we have attempted to arrange conditions for the flow of a macroscopic superconductivity current between regions of twinning planes which form a closed circuit. Such a current was not observed in Ref. 1. This negative result is not at all surprising, since the actual boundary between crystal twins is by no means a solid, coherent twinning plane but instead consists of distinct twinning-plane regions with dimensions of a few microns to a few tens of microns, separated by dislocated regions. This conclusion is supported by electron-diffraction measurements,[3] the comparatively slight (~40%) anisotropy of M_D, and the results of Ref. 4. Clearly, it is a very difficult matter to create a twin in which the coherent twinning plane is continuous. We have accordingly taken a different approach: Since tin can be twinned easily by mechanical means, we prepared some highly deformed samples in which the concentration of microscopic twins was large enough (as we will see below) to establish electrical contact between their boundaries. A superconducting current over the entire sample was inferred from the appearance of a hysteresis of the magnetic moment of the sample upon a change in the applied magnetic field.

The test samples were cylinders 1.7 mm in diameter consisting of 99.999%-pure tin single crystals in various orientations. The flat end of a sample was polished on glass with corundum powder with a grain size ~10 μm. As a result, a surface layer at the end was converted into a region of small crystals (~1 μm in size) to a depth ~50 μm (when this layer was dissolved away, the surface of the original crystal was revealed, with small embedded crystals covering roughly half the area). The moment M_D was measured with the magnetometer of Ref. 5; the polished end of the sample was placed at the center of the pickup coil of the magnetometer. When the sample was moved along the axis of the coil, it was found that the observed signal came entirely from the end of the sample.

Figure 1 shows one of the recordings of the magnetic moment $M(H)$ of the end of a sample (H is the magnetic field applied to the sample). At $\Delta T = T - T_c = 0.004$ K we observe only the diamagnetic moment M_D, similar to that observed in Ref. 1, ex-

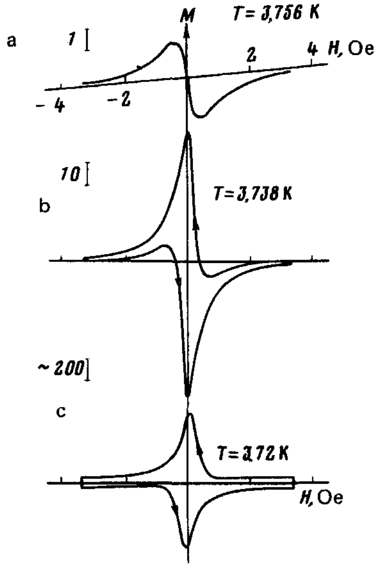

FIG. 1. Measurements of the magnetic moment M of the polished end of a tin sample, plotted as a a function of the magnetic field H. The bars at the left show the relative scales of the M axes; the temperatures T are shown at the right. Experimental results on a different sample with $\Delta T \approx 0.02$ K are shown in Fig. 2.

cept that there are no discontinuities on the curve of $M(H)$. Curve a is smooth and completely reversible. As the temperature is lowered, the $M(H)$ curve exhibits a hysteresis which implies the appearance of a stable, closed superconducting current in the sample. On curve b, the circuit for this current is closed at $H = 0.8$ Oe, at which the moment M_D is quite visible. With a further lowering of the temperature, the hysteresis loop increases in amplitude (curve c), concealing M_D and indicating an increase in the critical current J. (Partial cycles are observed within the hysteresis loop as the field H is restored.)

The current flows along the periphery at the end of the sample. This current distribution was established in an experiment with a sample from which a central region 0.8 mm in diameter of the polished end was removed by drilling. The conversion of the disk into a ring did not affect the value of M. Another control experiment was carried out with a sample consisting of a 5-μm thick film deposited by evaporation on the end of a quartz rod in place of the sample. The hysteresis curve of $M(H)$ obtained from this film at $T < T_c$ is similar to the curve in Fig. 1. We know that the current in a thin film in a magnetic field perpendicular to the plane of the film flows along the periphery.[6]

The current flowing in the sample was determined by a substitution method. A wire turn was placed in the pickup coil in place of the sample, and a calibrated current from an external circuit was passed through this turn.

The thickness of the superconducting layer was measured in the following way. A known amount of tin was dissolved from the end of the sample by an electrolytic method. After each such dissolution, we studied the superconductivity of the sample. Working in this manner, we obtained the results shown in Fig. 2. The maximum

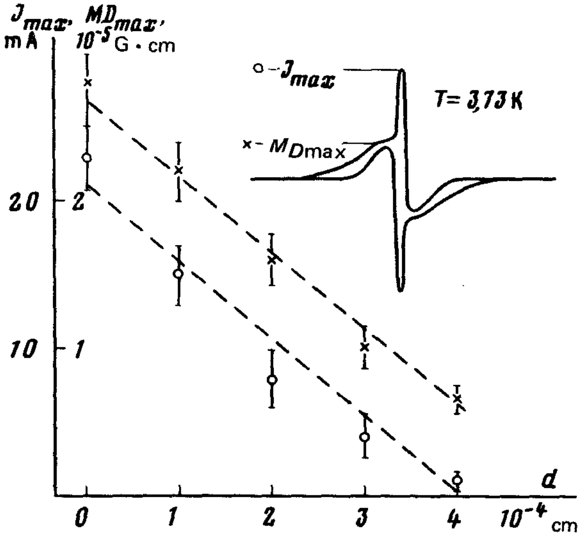

FIG. 2. The maximum magnetic moment $M_{D\max}$, referred to 1 cm^2 of the surface area, and the maximum critical current $J_{\max}$ (at $\Delta T \approx 0.02$ K), plotted as functions of the thickness d of the layer dissolved from the polished end of the sample. The inset at the upper right shows the regions on the curve to which the measured values correspond.

magnetic moment $M_{D\max}$ and the maximum critical current $J_{\max}$ fall off roughly in proportion to the thickness (d) of the dissolved layer, indicating that the distributions of M_D and J are homogeneous over the thickness of the superconducting layer $d_s \approx 4 \times 10^{-4}$ cm.

Turning to a discussion of the experimental results, we first note the two following circumstances:

1) We established in Ref. 1 that the moment M_D arises exclusively from a twinning plane; a boundary between crystals with random mutual orientations does not exhibit a moment M_D. A random boundary is a layer of dislocations, and stresses arise near it as the sample is cooled. There are no such stresses near a twinning plane.

2) The curves of M_D (H) and M_D (T) for the polished layer at temperatures above the region in which the hysteresis occurs are completely similar to the corresponding curves for a twinning plane.[1] The only difference between the two cases is in the absolute value of M_D, which is about two orders of magnitude higher (in addition, no metastable states are observed).

These results evidently mean that the moment M_D of the polished layer is produced by regions of ⟨301⟩ twinning planes, which separate microscopic twin crystals, and this moment is not affected by other structural elements in the layer.

Working from this conclusion, let us estimate the average distance between twin boundaries, δ. Knowing the magnetic moment of the superconducting layer, the thickness of the layer (Fig. 2), and the moment of an isolated twinning plane (5×10^{-7} G • cm at $\Delta T = 0.02$ K, according to the measurements of Ref. 1), we can find the density of twin boundaries in the layer: $n \approx 10^5$ cm^{-1}. We then find $\delta \approx 10^{-5}$ cm, i.e., a distance smaller than the coherence length $\xi_0 = 3.5 \times 10^{-5}$ cm.

This result ($\delta < \xi_0$) indicates that the superconducting current can flow along many twin boundaries forming random closed circuits in the interior of the layer. In fact, the experiments reveal (Fig. 2) that decreases in T and H are accompanied at first by a slight hysteresis, which implies the appearance of closed currents in small regions of the layer; later, a closed current arises around the entire sample. This occurs when the equivalent thickness w of the superconducting layer adjacent to the twinning plane reaches the value of δ; the measurements of Ref. 1 yielded $w = 0.6 \times 10^{-5}$ cm at $\Delta T = 0.02$ K.

We did not study the distribution of the density j_c of the critical current $J_{\max}$ (Fig. 2) over the superconducting layer. To estimate j_c, we assume that the current is concentrated in a peripheral ring whose width is equal to the layer thickness d_s. This estimate (which is clearly an overestimate) yields $j_c \approx 10^5$ A/cm^2. By way of comparison, the critical current density found for $\Delta T = -0.1$ K in Ref. 7 for tin films synthesized by evaporation was $j = 2.5 \times 10^4$ A/cm^2. Under the experimental conditions corresponding to Fig. 1c, the current density j_c increases by more than an order of magnitude, and as T_c is approached more closely, it increases by yet another order of magnitude.

To supplement the experiments of Ref. 1 on tin, we carried out some similar measurements with mechanical twins of indium, in which the twinning plane has the ⟨101⟩ orientation. The behavior of the diamagnetic moment M_D in indium is described by the equations in Ref. 2 with the constants $h = 0.1$ Oe and $\tau = 0.0008$ K.

In summary, we have studied the superconductivity of a fine-crystal metal at a temperature above the critical temperature for the single crystal. We have found convincing evidence that this superconductivity results from an effect discovered in Ref. 1: the superconductivity of a two-dimensional metal, in this case the twinning plane of a metal crystal.

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[1)] In the figures in this paper, as in Ref. 1, the temperature given is the temperature determined from the helium vapor pressure, which is 0.01 K lower than the temperature of the sample.[2]

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