

CRITICAL CURRENT ANISOTROPY IN SUPERCONDUCTING PbIn SINGLE CRYSTALS

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Anisotropy of the critical current in cylindrical single crystals of superconducting alloys Pb-24 at.% In and Pb-12 at.% In is investigated in a broad range of field strengths $H_{c1} < H < H_{c3}$ and for different treatments of the surface. The long axis along which the transport current is directed coincides with either the [100] or the [110] direction, and the magnetic field is perpendicular to the current. Measurements of critical current anisotropy in etched samples show that the character of the anisotropy reflects the crystallographic symmetry of the samples. Critical current peaks are observed when the magnetic field is oriented along $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ directions. The magnetic field dependence of the anisotropy is studied. Surface pinning of vortices is the dominant factor determining the magnitude of the critical current, and the observed anisotropy is associated with the condition of the surface layer.

THE passage of a transport current through a type-II mixed-state superconductor is accompanied by energy dissipation and an associated electric voltage, resulting from vortex motion induced by the Lorentz force. In any given magnetic field a so-called critical value of the current is linked with the voltage. The critical current differs from zero because various defects and inhomogeneities serve as vortex pinning sites. The defects can be located either on the surface or in the bulk of the superconductor. For type-II superconductors having small values of the Ginzburg-Landau parameter κ the critical current J_c depends largely on the condition of the surface layer. Different treatments of the surface (polishing, etching, coating with a normal metal) affect the amount of surface pinning.^[1-3] In addition, Swartz and Hart^[1] have shown that J_c is influenced not only by vortex pinning in the surface layer but also by the very existence of the surface, i.e., by a surface barrier. It is of interest to determine the relative roles of bulk and surface pinning, and also of the surface barrier, in generating the critical current for single crystals. Bulk defects, particularly dislocations, are distributed anisotropically in single crystals. Therefore for a sample with an isotropic surface we can expect to determine the anisotropy of J_c that is associated with bulk properties. On the other hand, we can observe how J_c is affected by chemically induced anisotropy of the surface.

1. EXPERIMENTAL RESULTS

We investigated the anisotropy of the critical current J_c in single crystals of Pb-24 at.% In and Pb-12 at.% In alloys having different orientations. High-purity Pb and In were used. Alloys of the given concentrations are solid solutions with face-centered cubic lattices. The single crystals were grown by single-pass zone melting from ingots of the homogenized single-phase alloy enclosed in cylindrical glass ampoules. The composition of the samples was checked by chemical analysis, and their orientations were determined by means of x ray diffraction. The surface condition was changed by chemical etching and polish-

ing. Since there was no qualitative difference in the results obtained with the two different alloys we here give only the results obtained for the alloy containing 24% In ($\kappa = 4.6$ ^[4]). Characteristics of the single crystals are given in the accompanying table.

In all the experiments the long, rotational, axis of the sample was perpendicular to the external magnetic field, and the transport current was directed along the axis. All measurements were obtained at 4.2°K. The conventional four-probe technique was used to measure the critical current, which was defined as that present when the voltage appearing on the sample was 0.5 μ V/cm. The current contacts were soldered on the samples in such a way as to make contact with practically the entire area of each end face; point contacts were used to measure the potential. The anisotropy of J_c was investigated as a function of the angle φ between the external magnetic field H and the selected crystallographic direction. Variation of the magnetic field orientation relative to the line between the potential contacts produced no effect on the shape of the angular diagrams for J_c .

Cylindrical samples in a magnetic field perpendicular to the cylinder axis exhibit surface superconductivity.^[5] Therefore the anisotropy of J_c was measured in a broad range of fields above and below H_{c2} .

Prior to the first measurements the samples were subjected to orientational etching in a 95%-5% solution of methanol and nitric acid. Figure 1 shows the dependence of J_c on φ for different fields in the case of sample I, which had a near two-fold axis (see the table). The figure shows that the anisotropy of J_c is

Characteristics of the single crystals

No. of sample	Sample diameter, 10^{-2} cm	$R(300^\circ)/R(4,2^\circ)$	Orientation of sample axes	
			θ , deg	ψ , deg
I	9	2.37	45	84
II	8.8	2.39	0	2
III	1.29	2.37	0	4

Note. The angle θ is measured from the (100) plane; ψ is the angle between the sample axis and the [100] direction.

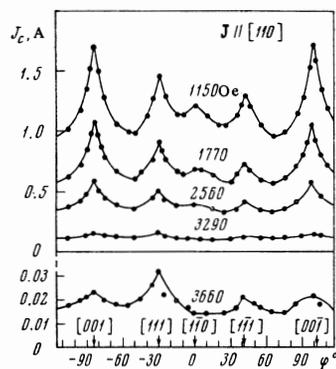


FIG. 1. Dependence of the critical current J_C on φ for sample I in different magnetic fields. $\varphi = 0$ for $H \parallel [1\bar{1}0]$.

associated with the crystallographic symmetry of the sample. Peaks of J_C are observed when the magnetic field is oriented along $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ directions. The peak heights differ. For $H \parallel \langle 100 \rangle$ the peak is highest and is distinct in all magnetic fields, while for $H \parallel \langle 110 \rangle$ the peak is much less pronounced and disappears in the highest fields. When the field exceeds $H_{C2} = 3400$ Oe the critical current falls off steeply for all orientations (Fig. 4), although the anisotropy of J_C is of the same character as in fields below H_{C2} .

Figures 2 and 3 show the dependence of J_C on φ for samples II and III, where the long axis is nearly a four-fold axis of rotation. In both samples a maximum of the critical current is observed for $H \parallel \langle 100 \rangle$. The peak in sample III for $H \parallel \langle 110 \rangle$ does not appear in sample II. However, when the critical current is determined by extrapolating the first linear segment of the current-voltage characteristic,^[5] a maximum of J_C in this direction can also be observed in sample II (Fig. 2a, curve 3).

The anisotropy of the critical current in a given field can be characterized by either the absolute anisotropy (the difference between the critical currents in

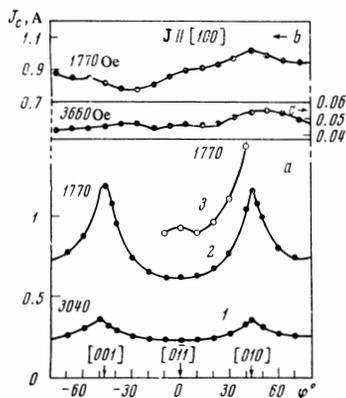


FIG. 2

FIG. 2. Dependence of J_C on φ for sample II: a – sample with etched surface; b and c – sample with polished surface. $\varphi = 0$ for $H \parallel [110]$. Curves 1 and 2 – J_C determined from voltage appearing on the sample; curve 3 – J_C determined by extrapolating the first linear segment of the current-voltage characteristic.

FIG. 3. Dependence of J_C on φ for sample III in different magnetic fields. $\varphi = 0$ for $H \parallel [0\bar{1}1]$.

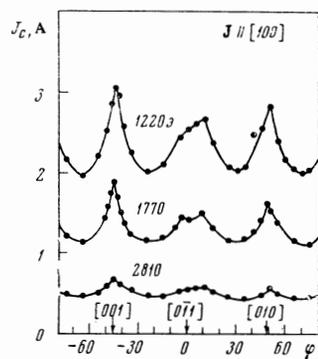


FIG. 3

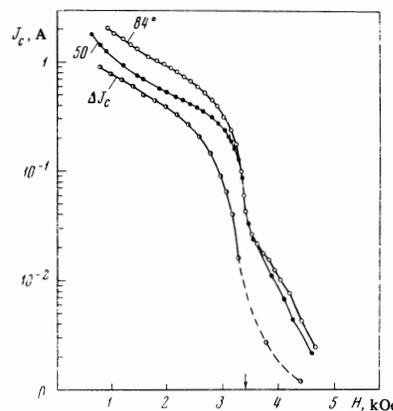


FIG. 4. Dependence of J_C on H for sample I, with two different field directions. The maximum of J_C is found at $\varphi = 84^\circ$ with $H \parallel [001]$, and the minimum at $\varphi = 50^\circ$. The magnetic field dependence of the absolute anisotropy ΔJ is also shown: $\Delta J = J_C(84^\circ) - J_C(50^\circ)$. The field H_{C2} is indicated by an arrow.

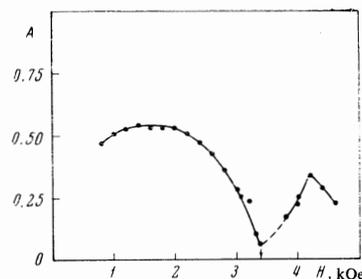


FIG. 5. Magnetic field dependence of the relative anisotropy A for sample I. $A = J_C(84^\circ) - J_C(50^\circ) / \bar{J}$. The field H_{C2} is indicated by an arrow.

the directions of the maximum and minimum: $\Delta J = J_{\max} - J_{\min}$), or by the relative anisotropy [ΔJ divided by the average critical current $\bar{J} = (J_{\max} + J_{\min})/2$]. The relative anisotropy will be denoted by the letter A . Figure 4 shows the dependence of ΔJ on the magnetic field for sample I, along with the dependence of J_C on H for $\varphi = 84^\circ$ (maximum) and $\varphi = 50^\circ$ (minimum). The absolute anisotropy ΔJ diminishes as the magnetic field increases up to H_{C3} . For the same sample the magnetic field dependence of the relative anisotropy is of different character (Fig. 5). We find that A is maximal near the field strength $\frac{1}{2}H_{C2}$ and falls off steeply as H_{C2} is approached. Above H_{C2} the relative anisotropy at first increases with the field, but diminishes again as the external magnetic field approaches H_{C3} . The dependence of A on H is similar for all the samples. For samples II and III the maximum of A is 0.64 and 0.50, respectively.

All the foregoing results pertain to samples that were etched to produce anisotropic surfaces. For the purpose of distinguishing the bulk and surface effects in the character and magnitude of the critical current anisotropy, after the given measurements samples II and III were polished chemically in a mixture of glacial acetic acid and hydrogen peroxide. The etched and polished surfaces have different appearances. On an etched surface highlights are seen, which are reflections from the principal crystallographic planes;

after being polished the same surface reflects light uniformly. Figure 2 shows that polishing changes both the character and the magnitude of J_c anisotropy. Here for sample II in a 1770-Oe field curve b represents $J_c(\varphi)$ following the polishing treatment, for comparison with the curve for the etched surface of the same sample. The maxima and minima observed for the polished surface cannot be associated with the crystallographic directions. In the 1770-Oe field the anisotropy A is smaller for the polished than for the etched surface by the factor 2.4. Above H_{c2} the anisotropy of J_c for the polished sample becomes randomly irregular (Fig. 2c). When a polished sample is re-etched the previous character and magnitude of critical current anisotropy are restored.

2. DISCUSSION OF RESULTS

The differences in the character and magnitude of critical current anisotropy following different surface treatments of the single crystals showed that J_c depends mainly on the surface condition of the superconducting material. The decisive role of the surface is manifested by the fact that in fields above H_{c2} , where superconductivity exists only in the surface layer, the anisotropy of J_c resembles that observed in the mixed state region. Polishing has an identical effect on the critical current in all fields up to H_{c3} . The anisotropy of J_c for polished samples is of random character, not reflecting crystallographic symmetry, and the anisotropy is of much smaller magnitude. We believe that the residual anisotropy resulted from imperfect polishing rather than from bulk pinning. Bulk defects appear to have little effect on the critical current. Annealing for 110 hours near the melting point produced practically no change in the density of the critical current. This is understandable since lead-based alloys have a low Debye temperature and most of the annealing effect takes place at room temperature. These alloys have very many more surface defects than bulk defects; consequently surface pinning is predominant.

Only two investigations of critical current anisotropy have been reported in the literature.^[6,7] In both instances the measurements were performed on Nb single crystals. Anisotropy of J_c was observed only following considerable deformation; the critical current was isotropic in undeformed single crystals. The authors attribute anisotropy in the deformed samples to the introduction of a large number of anisotropically distributed bulk dislocations. The critical current anisotropy that we observed in PbIn single crystals is associated with surface anisotropy. To reveal anisotropic bulk pinning in PbIn alloys we would have to deform the samples at low temperatures.

We obtained photomicrographs of different regions on the etched surface of sample I. In regions intercepted by the principal planes etch pits ($\sim 10^{-4}$ cm) are observed, whereas the surfaces of other regions are smooth. The distribution of the pits on the etched surface thus reflects the crystallographic symmetry of the sample. Figures 1–3 show that maxima of J_c appear when the magnetic field H is perpendicular to the principal planes $\{100\}$, $\{110\}$, and $\{111\}$ where pits are

etched. For samples with either a two-fold or a four-fold axis parallel to the long axis of the cylinder the highest peak of J_c results when the field is oriented along a $\langle 100 \rangle$ direction. In both cases H is perpendicular to the $\{100\}$ plane, and the vortices are moving in different directions relative to the crystal axes because of different transport current directions. When the magnetic field is perpendicular to the $\{110\}$ plane, independently of the transport current direction, a very much less pronounced peak of J_c is observed. For sample I (Fig. 1) a high J_c peak is observed when H is along $\langle 111 \rangle$, although the vortices in this case do not move in any specified crystallographic direction. Therefore the magnitude of the critical current is not governed by the direction of vortex motion relative to the crystal axes nor, consequently, by the planes through which the vortices enter and leave the sample, but rather by the pinning force in surface regions perpendicular to the magnetic field.

The anisotropy of pinning forces on the surface of a single crystal can result both from anisotropically distributed defects (such as the points where dislocations reach the surface) and from anisotropy of the surface topography (which appears after etching). Critical current anisotropy is observed only in etched samples. Polishing causes the disappearance of the anisotropy; this effect is observed when a single sample is alternately polished and etched several times. The etch pits appear to be additional very effective sites of surface pinning. The anisotropic distribution of these sites, combined with the crystallographic structure of the sample, is responsible for the anisotropy of the critical current.

Chemical polishing makes the surface isotropic; the etch pits are smoothed out and the critical current anisotropy disappears. Therefore anisotropy of the surface microrelief causes anisotropy of the critical current.

The magnetic field dependence of the anisotropy A in the interval $H_{c1} < H < H_{c2}$ shows that surface pinning is more effective for a relatively lower vortex density in the sample (both the critical current and its anisotropy are greater). As the field is increased the vortex density in the superconductor is enhanced. The interaction between vortices becomes more important, thus evidently reducing the role of surface pinning. Figure 5 shows that as H_{c2} is approached A is diminished.

The behavior of $A(H)$ and the character of the anisotropy are similar in the field interval $H_{c2} < H < H_{c3}$ and the mixed state region, for still unknown reasons. Above H_{c2} the existence of surface regions in an inclined magnetic field leads to a periodic vortex structure in the surface layer.^[8] The motion of this structure should also be affected by surface anisotropy. However, surface regions perpendicular to the magnetic field should go into the normal state in the field H_{c2} . Further investigations are needed to reveal the mechanism leading to critical current anisotropy in fields above H_{c2} .

As was mentioned in our Introduction, the magnitude of the critical current can depend both on the pinning force in the surface and on the surface barrier, i.e., on the surface regions that are crossed by vortices.

Our measurements of the critical current anisotropy showed that the nonuniform surface relief resulting from etching greatly enhances pinning in the surface layer but does not affect the surface barrier. These results are manifested by the fact that extrema of the critical current appear when the field is perpendicular to principal planes on which etch pits are produced, but do not appear when the field is parallel to these planes. Further experiments will be needed to elucidate the effect of the surface barrier on the critical current.

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²H. R. Hart and P. S. Swartz, Phys. Rev. 156, 403 (1967).

³W. C. H. Joiner and G. E. Kuhl, Phys. Rev. 168, 413 (1968).

⁴W. F. Druyvesteyn and J. Volger, Philips Research Reports 19, 359 (1964).

⁵N. Ya. Fogel', V. G. Volotskaya, L. N. Korovin, and I. M. Dmitrenko, ZhETF Pis. Red. 9, 180 (1969) [JETP Lett. 9, 105 (1969)].

⁶C. S. Tedmon, R. M. Rose, and J. Wulff, J. Appl. Phys. 36, 829 (1965).

⁷J. A. Good and E. J. Kramer, in Proceedings of the Eleventh International Conference on Low Temperature Physics, Vol. 2, 1968, p. 920.

⁸I. O. Kulik, Zh. Eksp. Teor. Fiz. 55, 889 (1968) [Sov. Phys.-JETP 28, 461 (1969)].

¹P. S. Swartz and H. R. Hart, Phys. Rev. 137A, 818 (1965).