Dislocation superconductivity at above-critical temperatures and fluctuations of the superconducting phase in tin

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A magnetometer with sensitivity 0.01 μG was used to measure the dependence of the magnetic moments of single crystals of tin and indium on the magnetic field at $H < 15$ Oe and on the temperature $3\text{K} < T < 4.2$ K. The dependence of the resistance of a tin sample on H and T was measured under the same conditions. It was observed that an additional magnetic moment $M_D \propto \exp(-T/0.01\text{ K})$ is induced by the dislocations in tin single crystals in fields $H < 3$ Oe and at temperatures $T_c < T < (T_c + 0.15\text{ K})$. Its maximum value $M_D^{\max}$ is observed in a field $H \approx 0.7$ Oe. At $T = T_c$, the value of $M_D^{\max}$ per 10^6 dislocations is approximately 80 μG. Under the same conditions, a decrease, $\Delta R_0 \propto \exp(-T/0.6\text{ K})$, is observed in the sample resistance. The observed phenomenon is attributed to the existence, at $T > T_c$, of superconductivity in a volume of radius $\sim 10^{-5}$ cm around the dislocation. From the measured values of the fluctuation diamagnetic moment, the following values were obtained for the superconductivity parameters of tin: $\xi_0 = (3.4 \pm 0.5) \times 10^{-5}$ cm, $\chi = 0.13 \pm 0.01$, and $dH_{c2}dT|_{Tc} = 20.5 \pm 1$ Oe/K. The measured values of the fluctuation diamagnetic moment of indium agree with the results of Gollub et al. (Phys. Rev. B 7, 3039, 1973).

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The measurement of the magnetic moment of superconductors at temperatures above T_c in a weak field (on the order of several or several dozen oersteds) is of interest because in this region there appears a diamagnetic moment due to the onset of fluctuation germs of the superconducting phase, and from the dependence of this moment on the magnetic field and on the temperature one can determine the values of dH_{c2}/dT at $T = T_c$, the Ginzburg-Landau parameter $\varkappa$, and the correlation length ξ_0. For type-I superconductors these quantities could be determined only for samples having a small dimension, such as microspheres, thin wires, or thin films, by measuring the penetration depth, the onset of superconductivity in longitudinal an magnetic field, or the metastable states produced when microsamples become superconducting in the presence of a magnetic field. Effects that may be connected with the crystal structure of the sample are not observable in microsamples.

Measurements of a fluctuation diamagnetic moment of approximately 10^{-6} G at a temperature $T \sim T_c + 0.01$ K in a field ~ 2 Oe, with the required sensitivty, became possible only recently through the use of a superconducting quantum interferometer. The only successful experiment in this region is that of Gollub *et al.*,[1] who measured the fluctuation diamagnetic moment of indium of lead (and of several alloys. Doll[2] observed a diamagnetic moment $\sim 10^{-3}$ G in tin single crystals at a temperature $\sim T_c + 0.01$ K in a field ~ 0.1 Oe, but the effect was neither investigated nor explained.

In the experiments reported here we measured the nonlinear part of the magnetic moment as a function of the magnetic field and of the temperature in highly perfect single crystals of tin (electron mean free path on the order of several millimeters). To check the experimental setup and to develop the procedure, we measured the fluctuation diamagnetic moment near T_c in indium samples.

EXPERIMENT

The principal measurement installation was the superconducting magnetic-flux quantum interferometer (SQUID) described in Ref. 3. The magnetic flux from sample *4* was fed to the sensitive element *1* of the SQUID with the aid of a superconducting magnetic-flux transformer *2* (Fig. 1). The transformer had a differential input end. The sample was placed in a superconducting solenoid whose field was calibrated against the known $H_c(T)$ dependence, at $T < T_c$, for indium and tin.[4] The SQUID and the solenoid with the sample were shielded from each other as well as from external magnetic field by superconducting lead shields *6*.

The main difference between our instrument and those desribed earlied (e.g., Ref. 1) lies in the use of a differential system of coils as the input circuit of the superconducting magnetic-flux transformer. The differential system of the transformer was balanced with relative accuracy up to 10^{-8}. The high balance accuracy of the differential system makes it possible

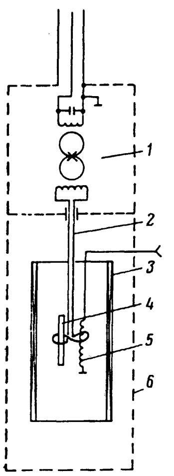

FIG. 1. Diagram of magnetometer: *1*—SQUID, *2*—superconducting magnetic-flux transformer, *3*—superconducting solenoid, *4*—sample, *5*—equivalent coil for calibration, *6*—superconducting lead screen.

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to measure the nonlinear part of the magnetic moment M as a function of the magnetic field H at a constant temperature, in contrast to the customary measurements in a frozen-in magnetic field.

The recording system of the instrument was calibrated with a coil of copper wire of 0.02 mm diameter, wound on a quartz capillary of 1.6 mm diameter. The coil played the role of the equivalent of the sample and was placed in instrument in place of the sample. Experiments were also performed in which, as shown in Fig. 1, the equivalent coil *5* was placed alongside the sample in the second arm of the differential system of the flux transformer *2*. With this arrangement, the current through the equivalent coil could be used to cancel the signal from sample *4*. After calibration and a check on the reproducibility from experiment to experiment in the entire range of fields and temperatures attainable in this instrument, the electric calibration circuits were removed, since they were a source of noise. The attained sensitivity of the instrument to the magnetic flux through the sample was 10 nMx, which corresponds to a moment of 0.01 μG for a sample cross section 1 mm[2] at a signal/noise ratio equal to 3.

The temperature was determined from the ^{4}He vapor pressure. Owing to the parasitic heat inflow and to the thermal resistance between the sample and the helium bath, the sample temperature was 0.008 K higher than the temperature of the helium bath. This temperature difference was determined from the known critical temperatures of the indium and tin.[4]

To monitor the installation, measurements were made on the previously investigated[1] indium. The samples were made of high-purity indium, characterized by an electron lifetime $\sim 8\times 10^{-10}$ sec at $T\sim 4$ K. The main measurements, however, were made on tin samples. Single crystals measuring $1\times 1\times 12$ mm were grown by the procedure described in Ref. 5; we used also previously investigated[6] samples. The samples were made of tin 99.9999% pure, characterized by an electron lifetime $\sim 2\times 10^{-9}$ sec and an electron mean free path on the order of several millimeters at helium temperature. The crystallographic fourfold axis C_4 was oriented along the sample accurate to $\sim 1°$. The orientation of the crystollagraphic axes was monitored by x-ray diffraction.

The correctness of the estimated electron mean free path is confirmed by the fact that the induced eddy currents in the plane of the cross section of the sample when the magnetic field was scanned were independent of temperature, accurate to $\sim 1\%$, at $3.7\ \mathrm{K} < T < 4.2$ K, and decreased 30–40% after the sample surface was etched. It can thus be concluded that the electrons are scattered mainly by the faces of the sample, the distance between which is 1 mm. At the same time, when measuring the resistance along a sample 12 mm long, the sample resistance changed by 20–30% under the same change of temperature.

In addition to measuring the dependence of the tin magnetic moment M on the magnetic field H and on

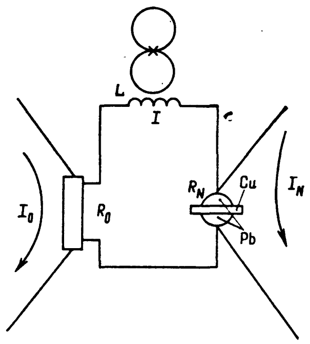

FIG. 2. Resistance-measurement scheme: L—coil for coupling to the SQUID, R_0—sample, R_N—comparison resistance.

the temperature T, we measured also the dc resistance R_0 of the sample. To decrease the influence of the emf's due to charge motion in the crossed electric and magnetic fields, the measurements were made with $\mathbf{E}\parallel\mathbf{H}$. The cold part of the electric circuit is shown in Fig. 2. All the elements of the potentiometer loop were made of superconducting material, with the exception of the comparison resistor R_N, which is a disk, 1 mm in diameter, of copper foil 0.15 mm thick; $R_N\approx(1/2)R_0$. The currents I_0 through the sample R_0 and I_N through R_N could be varied and measured independently of each other. Such a setup made it possible to measure R_0 by two methods:

1) At a fixed field H, by simultaneously turning the currents I_0 and I_N on and off and by varying one of them, the current I through the coil used to couple to the SQUID could always be made equal to zero. In this case $R_0=R_NI_N/I_0$.

2) At fixed currents I_0 and I_N, by registering the current I as a function of the field H, it is possible to obtain $R_0=R_N(I_N+I)/(I_0-I)\approx R_N[I_N/I_0+(I_0-I_N)I/I_0^2]$ at $I\ll I_0$ and $I\ll I_N$. No influence of the small current I through the potential contacts on the measured quantity R_0 was observed.

To sensitivity of the circuit to the current I at zero field H reached 10 nA at a signal/noise ratio equal to 3. In a field $H\sim 5$ Oe, the sensitivity was limited to 100 nA because of the influence of the noise emf produced in the potentiometer loop by the instability of the solenoid current. At a resistance $R_N\sim 10^{-9}\ \Omega$ the voltage sensitivity of the circuit is $\sim 10^{-16}$ V.

The instrument was shielded against the earth's magnetic field by a permalloy shield placed outside the cryostat. The residual field inside the shield (in the absence of the cryostat) at the point where the sample was located had a longitudinal component ~ 1 mOe and a transverse component ~ 0.1 mOe. The residual field of the shield was measured at room temperature with a permalloy pickup. The value of the residual magnetic field during the time of the experiment can be estimated from the shift of the symmetry center of the $M(H)$ curves relative to the point of zero current through the solenoid. This value did not exceed 1 mOe.

MEASUREMENT RESULTS

1. Indium

The experimentally obtained plots (Fig. 3) of the magnetic moment M' of an indium sample, as functions

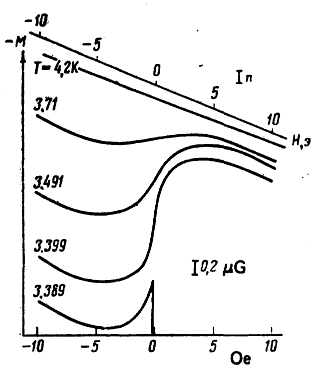

FIG. 3. Plots obtained in experiment with indium. In the plot for $T=3.389$ the jump of the magnetic moment near $H=0$ is due to onset of superconductivity of the sample. The sloping scale at the top indicates the linear variation of the signal as a result of inaccurate balancing of the differential transformer. The temperature of the ^{4}He bath is indicated.

of the field H, contain two components:

1) a linear section due to the imperfect balancing of the differential system of the flux transformer,

2) a nonlinear signal due to fluctuations of the superconducting phase.

To measure the fluctuation moment it was necessary to separate first the linear signal. This was done on the basis of experimental plots obtained at 4.2 K, when the fluctuation moment is negligibly small and the measurement produces a practically exclusively linear signal. Comparison of the experimental results with the theory proposed in Refs. 7, which was confirmed by the data of Ref. 1, is shown in Fig. 4 for a field 0.6 Oe $=0.3\,H_s$. In analogy with Ref. 1, here $H_s =0.11\,\Phi_0/2\pi\xi_0^2$ is the field in which the measured diamagnetic moment is half the value calculated in Ref. 8 by the Ginzburg-Landau theory. As seen from Fig. 4, the agreement between theory and experiment is good. Equally good agreement was obtained also for other values of the magnetic field.

2. Tin

Figure 5 shows plots obtained in an experiment in

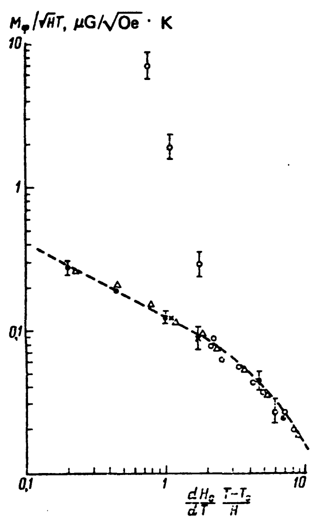

FIG. 4. Fluctuation diamagnetic moment at $T > T_c$ in normalized coordinates: Δ—indium[1]; ●—indium, $H=0.6$ Oe; O—tin, $H=0.9$ Oe; ×—tin, $H=0.9$ Oe after eliminating the additional diamagnetism. Dashed curve—result of theoretical calculation.[4]

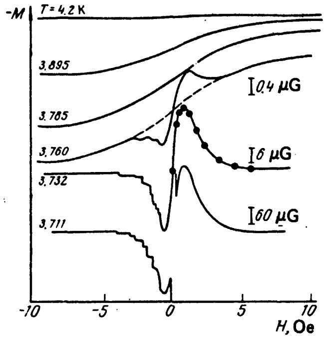

FIG. 5. Typical plots of the experiments with tin. On the right are indicated the ordinate scales. The four upper curves are plotted in the same scale and with a time constant ~1 sec, while in the remaining cases the time constant is smaller by approximately a factor of 100. The points on the plot at $T=3.732$ K are the results of a calculation. The break near $H=0$ on the plot for $T=3.711$ K is due to the onset of superconductivity of the sample. The direction of the plotting is from left to right. The temperature of the ^{4}He bath is indicated.

which we succeeded in cancelling out the linear component of the signal practically to zero. Thus, these plots show at 4.2 K $\geq T \geq$ 3.8 K only the signal due to the fluctuation moment. Usually the nonlinear part of the signal plot was separated by the same method as for the indium sample.

Further reduction of the results was carried out under the assumption that the theory proposed in Ref. 7 is valid. The plots of the fluctuation moment $M_f(H)$ at constant temperature T were used to plot $M_f(T)$ and constant field H. After extrapolating these plots to T_c we succeeded, in analogy with Ref. 1, in determining the quantity

$$H_s=3.1\pm0.5\,\text{Oe}.$$

It follows therefore that

$$\xi_0=(0.11\Phi_0/2\pi H_s)^{1/2}=3.4\pm0.5\cdot10^{-5}\,\text{cm}.$$

From the plots of $M_f/H^{1/2}T$ against $(T-T_c)/H$ at constant H we determined the value

$$dH_{c2}/dT\,|_{T_c}=-29.5\pm1\,\text{Oe/K},$$

and accordingly the Ginzburg-Landau parameter

$$\varkappa=\frac{dH_{c2}}{dT}\bigg|_{T_c}\bigg/\sqrt{2}\,\frac{dH_c}{dT}\bigg|_{T_c}=0{,}13\pm0{,}01$$

(we have used here the value $dH_c/dT\,|_{T_c}=164$ kOe from Ref. 4). A comparison of the results for a field 0.9 Oe $=0.3\,H_s$ with the theory[8] and with the measurements of the fluctuation moment in indium is shown in Fig. 4; they are seen to coincide at $dH_{c2}/dT\,|_{T_c}(T-T_c)/H>2$.

In addition to the considered fluctuation moment in the temperature interval $T_c<T<(T_c+0.1\text{ K})$, an additional magnetic moment $M_D(H)$ is observed on the $M(H)$ curves, characterized by the following features:

1) The field at which the additional diamganetic moment reaches its maximum $M_D^{\max}$ does not depend on temperature and is equal to ≈0.7 Oe. At constant temperature and in an increasing magnetic field,

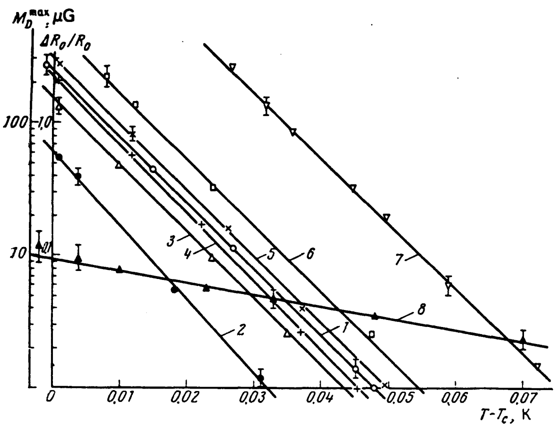

FIG. 6. Temperature dependence of the maximum value of the additional moment M_X^{max}: *1*—fresh sample, *2*—after etching; *3, 4, 5, 6,* —after *1, 2, 3,* and *6* deformation cycles, respectively, *7*—after a large deformation, *8*—decrease $\Delta R_0/R_0$ of sample resistance at $H=0$.

$M_D(H)$ is well described by the formula

$$M_D(H)|_T \propto He^{-H/h}, \quad h=0.7\,\text{Oe}.$$

The results of the calculation are shown by the points on the plot at $T=3.732$ K in Fig. 5.

2) The moment M_D increases rapidly and exponentially with decreasing temperature. Figure 6 shows the measured values of M_D^{max} at various temperatures, as well as an interpolated line *1*, described by the equation

$$M_D^{max}=A\exp[-(T-T_c)/\tau], \quad A=250\ \mu\text{G}\ , \quad \tau=0.01\ \text{K}.$$

Here τ is independent of the field H.

3) When the magnetic field is decreased from 3 to 0.7 Oe, the moment M_D increases jumpwise (10–15 jumps can be discerned). These jumps are not observed when the moment M_D decreases with increasing field H. Hysteresis of this type is typical of systems that have metastable states, when the onset of a new phase requires a germ with a dimension that increases a certain critical value. When the temperature is lowered, the first jump shifts linearly into the region of strong fields, with a derivative

$$dH_1/dT=-29.6\pm0.2\ \text{Oe/K}.$$

Extrapolation to $H=0$ yields a temperature $T_1=T_c+0.15$ K.

To ascertain the nature of the additional diamagnetism, a series of measurements were made with a sample whose surface was etched, as well as with a deformed sample. The surface was etched with concentrated nitric acid (the white coating produced on the sample can be easily washed off with acetone). After etching the sample surface, the moment M_D^{max} decreases by a factor 4–5 (line *2* on Fig. 6).

The next step was to introduce dislocations in the sample. This was done by bending the sample at room temperature around a cylindrical form of 10 cm radius, and then straightening the sample. Estimating the number of dislocations from the relative elongation of the sample faces, and recognizing that the lattice constant of tin in the C_4 direction is ~ 3.2 Å, we find that each such deformation cycle introduces into the sample ~ 10^6 dislocations. It turned out that M_D increases, with accuracy ~ 10%, in proportion to the number of deformation cycles (at up to six cycles). This proportionality indicates that there is no interaction between the dislocations. The lines *3, 4, 5,* and *6* in Fig. 6 show the value of M_D^{max} after respectively 1, 2, 3, and 6 deformation cycles. It follows thus from Fig. 6 that each 10^6 dislocations in a field 0.7 Oe at a temperature $T=T_c$ account for an additional diamagnetic moment ~ 80 μG. The most perfect of the investigated samples, judging from the value of M_D (the smallest of all samples) contained ~ (2–3) × 10^4 dislocations. The line *7* of Fig. 6 was obtained for a sample that was crushed between two glass plates. Etching a deformed sample produces practically no change of M_D.

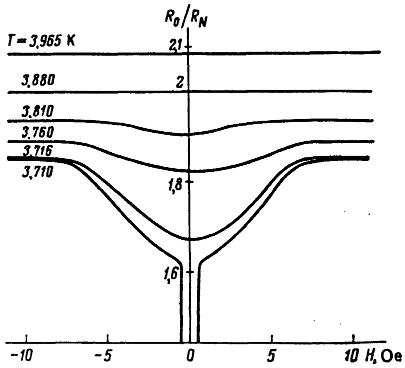

FIG. 7. Dependence of the sample resistance on the magnetic field and on the temperature. In the plot for $T=3.710$ K near $H=0$ the resistance drops to zero (superconductivity). The temperature of the ^{4}He bath is indicated.

In the course of measuring R_0 it was first established that a current up to 10 mA through the sample does not influence the value of M_D. It was then found that the field H applied to the sample affects the state of the electric contacts with the sample and interferes with the measurements of its resistance. To eliminate this interference the locations of the contacts (the ends of the sample) were shielded against the magnetic field by placing around them superconducting lead tubes electrically insulated from the sample. Of course, these shields made the field H on the sample inhomogeneous, so that the obtained $R_0(H)$ dependences (at $H\neq 0$) must be regarded only as approximate.

In measurements made in a homogeneous fields (without shields on the ends of the sample), at $T>T_c$ and $H>8$ Oe, the sample resistance decreased in proportion to T^5, and in the temperature range 4.2 k > T > 3.8 K it was independent of H accurate to ~ 3%. Under conditions identical with those at which M_D are observed, a decrease ΔR_0 of the sample resistance, which depended on H, was observed. Figure 6 shows a plot of $\Delta R_0/R_0$ at $H=0$ against temperature, as well as an interpolation line *8* corresponding to the equation

$$\Delta R_0/R_0=B\exp[-(T-T_c)/\tau'], \quad B=0.094, \ \tau'=0.06\ \text{K}.$$

TABLE I.

Parameter	Single crystal	Thin films (Ref. 9)	Microspheres (Ref. 10)
ξ_0, 10^{-5} cm	3.4±0.5	2.8±0.3	–
$\varkappa$	0.13±0.01	0.138±0.005	0.093±0.001
$dH_{c2}/dT\mid_{T_c}$	29.5±1	31.3±0.5	21.00±0.02

DISCUSSION

The tin superconductivity parameters obtained from measurements of the fluctuation moment are given in the table. The values of the same parameters obtained in other studies are shown for comparison. It is seen from the table that our results agree well enough with results obtained in experiments with thin films. The discrepancy with results obtained with microdroplets can apparently be attributed to the anisotropy of the tin in the superconducting state, which reaches 70% according to the data of Ref. 11. To clarify this question, tests must be made on tin single crystals with various orientations.

The appearance of the additional diamagnetic moment M_D can be interpreted as the appearance, in the tin samples, of regions in which the magnetic field H does not penetrate. Extrapolating the straight line 7 of Fig. 6 to T_c, we find that the largest moment M_D^{max} (of the crushed sample) reaches 5 mG at $T=T_c$, which is 5% of the diamagnetic moment of the sample when entirely in the superconducting state. Since the observed phenomenon takes place at temperatures close to T_c, it is natural to regard it as connected with superconductivity. It must thus be assumed that a small part of the sample volume becomes superconducting at a temperature higher than critcial ($T-T_c \leq 0.15$ K). Measurements of the sample resistance confirm this hypothesis. It appears that this phenomenon was the cause of the diamagnetism observed in tin in Ref. 2.

We note that it has been long known that a plastically deformed sample has a gradual transition into the superconducting state and consequently the first symptoms of superconductivity appear at $T>T_c$ (we assume, as usual, that T_c corresponds to the midpoint of the transition).

We consider now the following model of the phenomenon: we assume that each dislocation of length $L=1$ mm is surrounded by a cylindrical region from which the magnetic field has been pushed out; the diameter of the region is $\xi_0=3.4\times10^{-5}$ cm (we used for the estimates the value of ξ_0 for bulk superconductivity). We find that the total volume for $N=10^6$ dislocations is $V_0=\pi\xi_0^2LN/4\approx10^{-4}$ cm^3, which amounts to 1% of the volume of the entire sample. At the same time, even at $T=T_c$ the measured moment M_D^{max} for 10^6 dislocations does not exceed 0.1% of the diamagnetic moment of the superconducting sample. Apparently, thus, there is either no complete forcing out of the magnetic field from the region around the dislocation, i.e., the penetration depth $\lambda\geq\xi_0$, or else the transverse dimension of the superconducting region is less than ξ_0. We assume that in a field $H=0$ and at $T>T_c$ the summary superconducting volume depends on the temperature in the following manner:

$$V_s \propto \exp[-(T-T_c)/\tau].$$

In this case the only parameter that depends on H is T_c, and accurate to terms of first order of smallness we have

$$V_s \propto \exp\left[-\frac{T-T_c}{\tau}\right]\exp\left[\frac{H}{dH_c/dT|_{T_c}\tau}\right].$$

After substituting the numerical values $\tau=0.01$ K and $dH_c/dT_{T_c}\approx160$ Oe/K, we get

$$V_s \propto \exp[-(T-T_c)/0.01\,\mathrm{K}]\exp[-H/1.6\,\mathrm{Oe}],$$

i.e., the field in which M_D^{max} should be observed does not depend on temperature and is equal to 1.6 Oe. The measured field in which M_D^{max} is observed is equal to 0.7 Oe; for estimates made under such crude approximations, this proximity of the values should be regarded as good agreement.

It follows from experiments with deformed samples that the additional diamagnetism is due to dislocations and that in the performed experiments, as already noted, the dislocation density is low—there is no interaction between them. The decrease of M_D after etching the surface of a free sample is explained, in accordance with the advanced premises, by the fact that in the course of cooling and removal of the crystal from the mold in which it was grown, only the surface layer of the metal was damaged, and it is this which is removed by etching.

The most likely is the onset of a superconducting cylindrical region of some radius $r_s(T)$ around each dislocation, and the increase of this region with decreasing temperature; then the total superconducting volume is $v_s=[r_s(T)]^2LN$.

The superconducting regions near the dislocations have obviously small transverse dimensions. Therefore the observed jumps in the $M(H)$ plot must be ascribed to a transition of the crystal regions around the dislocations from a metastable normal state into a superconducting state. The large number (10–15) of the observed jumps can be attributed to the fact that different dislocations can have different crystallographic orientations and can have different directions relative to the magnetic field. With this jump-production mechanism, the observed field $H_1(T)$ is the limit of the existence of the metastable normal state for some group of dislocations. This reasoning justifies the agreement between the values of $dH_{c2}/dT|_{T_c}$ and dH_1/dT measured in the present study.

The most probable cause of the change of T_c in the crystal region adjacent to the dislocation can be the field of the mechanical stresses around the dislocation. It is known that under isotropic compression and tension the change of T_c in tin is $dT_c/dp=(4.5-5.5)\times10^{-5}$K/atm.[12] The shift of T_c determined by measuring the $H_1(T)$ dependence is 0.15 K. Thus, the pressure that causes the shift of T_c should be estimated at $p\approx3\times10^3$ atm. This is larger by one order than the yield point of tin,[13] the latter being determined by the motion of the dislocations, and is smaller by two orders than Young's modulus for tin.[13] A calculation of the change of T_c, but for the case of a superconducting-region dimension large compared with ξ_0, was carried out in Ref. 14.

Another possible cause of the change of T_c can be the dislocation-line oscillations, which can lead to another mechanism of attraction between the electrons. An estimate of the effect of the dislocations on T_c, but for the case of high density of the (interacting) dislocations, is given in Ref. 15. The predict shift of T_c is 0.1 K.

The two presented comparisons, unfortunately, comprise all the published data on our problem. It is obviously necessary to continue the experimental and theoretical investigations of the dislocation superconductivity observed in the present study, all the more since the obtained data seem to pertain to the case of non-interacting dislocations, a case not investigated before.

The fact that the indium and lead samples[1] did not display the phenomenon observed here in tin is obviously due to the softness of these metals. At room temperature, indium and lead are inelastically deformed and no noticeable stresses are preserved in them.

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