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"Intermediate Mixed" State of Type-II Superconductors*

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For magnetic fields in the range $(1-D)H_{c1} < H < H_t$, magnetization curves of low-κ type-II Pb-In and In-Bi alloys with nonzero demagnetization coefficients D exhibit a linear dependence of the magnetic moment on applied field. H_{c1} is the lower critical field and H_t is the field at which this linear dependence terminates. For finite D, $H_t < H_{c1}$. This behavior can be explained by a model of alternate regions of superconducting and mixed states. This array may be metastable. The mixed-state regions are all presumed to have the same temperature-dependent fluxoid-lattice spacing characteristic of H_{c1} when $D=0$. Thus, as the magnetic field is increased isothermally, the mixed-state regions grow until the whole sample has been converted into the mixed state. This model implies the existence of an attractive interaction between flux lines in low-κ materials.

I. INTRODUCTION

In a recent systematic study[1] of the effects of the demagnetization coefficient D on the magnetization curves of Pb-In and In-Bi alloys, we have verified the Cape-Zimmerman[2] equation

$$-4\pi(dM/dH)_{H_{c2}} = [\beta(2\kappa_2^2-1)+D]^{-1}, \tag{1}$$

where β is a constant taken to be 1.16 for a triangular flux lattice[3] and κ_2 is the generalized Ginzburg-Landau (GL) parameter. Another generalized GL parameter κ_1 was also obtained as a function of temperature and composition by using the Maki[4] results

$$H_c = H_{c2}/(\sqrt{2}\kappa_1), \tag{2}$$

where H_c is the thermodynamic critical field and H_{c2} is the upper critical field. Our measurements also indicated that $\kappa_1 = \kappa_2$ for all compositions and temperatures studied, in agreement with the results of Kinsel, Lynton, and Serin[5-7] for In-Bi alloys with $D=0$. In the course of this work, we observed an "intermediate-state-like" behavior, similar to that seen in type-I superconductors, which is illustrated in Fig. 1. Both the low-κ Pb-2 at.% In and In-1.5 at.% Bi samples undergo an isothermal transition from type-I intermediate-state-like behavior to type-II behavior as the applied magnetic field is increased above some characteristic field H_t. This should not be confused with the observations of Kinsel *et al.*[5-7] on an In-1.5 at.% Bi sample which at higher temperatures exhibited type-I characteristics, but at lower temperatures demonstrated type-II behavior. Both of our samples also exhibited exclusively type-I behavior near T_c. At lower temperatures, the samples are type II for $D=0$ (i.e., $\kappa > 0.71$). However, for a finite D they exhibit intermediate-state-like behavior over a certain region of applied field, i.e., $(1-D)H_{c1} < H < H_t$, where H_{c1} is the lower critical field and H_t is the field at which the sample has fully transformed into the mixed state. As we shall show below, $H_t < H_{c1}$ for finite D. Kinsel *et al.*[5-7] did not observe this latter effect because their samples were measured in a longitudinal field and thus demagnetization effects were absent.

There are electron-microscope measurements on these and similar alloy systems which bear directly on the interpretation of our macroscopic magnetization measurements. Thus, it is neces-

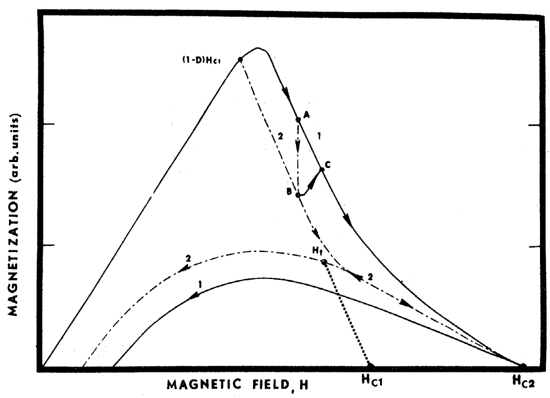

FIG. 1. Magnetic moment as a function of applied field for a Pb–2 at.% In rod (see curve a in Fig. 2). The dashed line 2 represents the measured "reversible" moment when a small ac field is applied transverse to both the dc field and the sample axis.

sary to review the Bitter pattern measurements of flux arrays in Pb-In foils and platelets (with $D \simeq 1$). Träuble and Essmann[8,9] and Sarma[10–13] increased the magnetic field applied perpendicular to a sample well beyond H_{c1}, decreased the field below H_{c1}, and then measured the resulting fluxoid structure. Sarma, on inhomogeneous Pb–2 at.% In disks, observed that the triangular flux-lattice characteristic of a type-II superconductor broke down into an "intermediate-state" structure as the applied field was reduced. The intermediate-state structure appeared at higher fields in regions of low indium content and at lower fields in regions of higher indium content. Sarma asserted that all regions contained sufficient indium to be type-II material. The "intermediate state" consisted of regions of pure superconducting material alternating with either laminae or bundles of flux lines. At 4.2 °K, all of the mixed-state regions had the same triangular lattice spacing, approximately 5500 Å. Sarma did not observe this behavior for Pb-In samples having a $\kappa > 1.2$.

Krägeloh[14] studied a well-annealed homogeneous Pb–1.92 at.% Tl disk sample by a similar electron-microscope method. The κ of that sample was slightly greater than $1/\sqrt{2}$ at T_c. In a perpendicular magnetic field H_{c1} was 730 Oe and $(1-D)H_{c1}$ was 182 Oe at a temperature of 1.25 °K. At that temperature and with an applied field of 343 Oe, he observed alternating regions which were either in the Meissner state or in the mixed state. All of the mixed-state regions had the same triangular lattice spacing, approximately 2500 Å. Essmann[15] recently observed that the lattice spacing in the mixed-state regions remained constant as the size of the regions grew with increasing applied field. He noted this behavior only for samples with $0.71 < \kappa < 1.5$. He also observed square flux-lattice arrays in some instances for Pb-In foils with κ slightly greater than 0.71. Thus, these microspic measurements are the basis for our interpreting the "intermediate-state" regions of our type-II magnetization curves in terms of alternate regions of Meissner-state and mixed-state material.

EXPERIMENTAL DETAILS AND RESULTS

The results for two samples are reported here: an In–1.5 at.% Bi rod and a Pb–2 at.% In rod. The In-Bi sample was extruded from a slug which had been melted under vacuum while being shaken to ensure homogeneity. The sample was then annealed before being studied. The Pb-In alloy was first prepared by being melted under vacuum and shaken. Part of the slug was then transferred to a sample tube, remelted, and reshaken. The cylindrically shaped specimen was then cooled until solidification occurred and then plunged into liquid nitrogen. The rods were 0.10 in. in diameter and 1.00 in. in length. From these dimensions, D was calculated to be 0.48 for each rod in a magnetic field transverse to the axis.

The Pb-In sample was immediately electropolished after being cast and then electroplated with chromium to prevent adsorption of gases. Earlier measurements showed that the magnetization curves of freshly prepared samples were much more reversible than those of samples exposed to the air. Furthermore, if a sample which has been exposed to the air for a substantial length of time was outgassed in a vacuum of about 10^{-4} Torr and the remeasured immediately, the magnetization curve obtained was identical with that observed immediately after the sample was first prepared. Electroplating with chromium allowed the samples to remain exposed to the atmosphere without affecting their magnetization curves. The magnetization measurements were made using a Foner vibrating sample magnetometer (VSM) manufactured by Princeton Applied Research Corp.

The solid lines labeled 1 in Fig. 1 represent VSM data taken in increasing and decreasing fields. For fields larger than that corresponding to the peak in the magnetization curve, the magnetic moment decreases linearly from its peak value with nearly the same field dependence as would be obtained for a type-I material having an identical demagnetization factor. The moment $M_1(H)$ then changes slope near H_t and then decreases almost linearly to zero at H_{c2}.

As part of a systematic study of chrome-plated Pb-In alloy compositions[16] containing up to 28 at.% In, it was observed that "jarring" of the sample either mechanically or by superimposing a 60-Hz transverse field (of about 20-Oe amplitude for the 2 at.% In case) perpendicular to both the dc field and to the sample axis resulted in a more nearly reversible magnetization curve. It is believed that this ac field unpins the flux lines which would other-

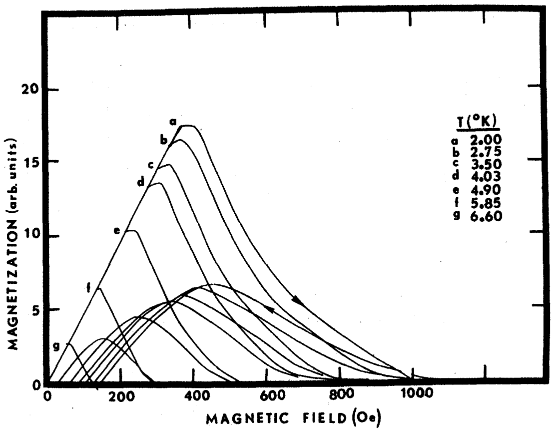

FIG. 2. Magnetic moment as a function of applied field for a Pb–2 at.% In rod at various temperatures, $D = 0.48$.

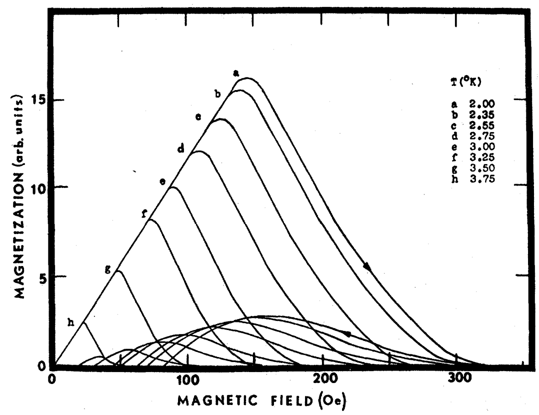

FIG. 3. Magnetic moment as a function of applied field for an In–1.5 at.% Bi rod at various temperatures, $D = 0.48$.

wise be responsible for the hysteretic behavior. The superimposed ac-field data are given in Fig. 1 by the dashed curve, labeled 2. To obtain the "jarred" curve $M_2(H)$ one stops the field sweep and the recorder pen is lifted at some field H (point A on curve 1). The ac field is applied briefly and then turned off. The recorder pen is lowered and the magnetic moment $M_2(H)$ is measured (point B, curve 2). Increasing the amplitude of the ac field at point B produced no further decrease in the magnetization. The field sweep is then restarted and the magnetic moment follows the path BC shown in Fig. 1. This procedure is then repeated many times, to yield the set of points $M_2(H)$. It should be noted that the application of an ac field caused the linear decrease in magnetic moment to commence at the same applied dc field at which deviations from the Meissner slope were observed to occur in the absence of a superimposed ac field. Furthermore, the ac field produced a linear section in the magnetization curve which was nearly parallel to the unjarred portion for $(1-D)H_{c1} < H < H_t$, and which, when extrapolated to the field axis, yielded the value of H_{c1}. It is significant that sample jarring did not alter the "intermediate-mixed-state" behavior shown in Figs. 2 and 3, which illustrate the unjarred results. Note that at lower temperatures where $\kappa > 0.71$, the magnetic moment has the same slope in the field region $(1-D)H_{c1} < H < H_t$ as do the curves at the highest temperatures where the sample is type I. This slope is not quite the $1/4\pi D$ which one would obtain by assuming the cylindrical sample to be an ellopsoid with the same length and diameter. This discrepancy may be due to end effects of the sample. The experimentally determined D is 0.44 ± 0.04, as indicated in Table I. The D calculated from the sample geometry is 0.48.

As did Campbell *et al.*,[17] we[16] observed that reversible curves, for applied fields near H_{c2}, lay midway between the increasing and decreasing curves recorded for unjarred data. The ac-field data on the samples of this study further substantiate this conclusion.

In analyzing the present data the following procedure was used.

(a) κ_2 was obtained from a measured value of $[dM_2(H)/dH]_{H_{c2}}$ by using Eq. (1) and the D determined from the slope of the magnetization curve at fields just above $(1-D)H_{c1}$.

(b) It was assumed that $\kappa_1(t) = \kappa_2(t)$, so that $H_c(t)^{\text{calc}}$ could be calculated from the experimental value of $H_{c2}(t)$ and $\kappa_2(t)$ using Eq. (2). Our results[16] for other Pb-In alloys demonstrated this agreement within the experimental error of 7% for κ_1 and 5% for κ_2.

TABLE I. Pb–2 at.% In rod. All fields in Oe.

T (°K)	κ_2	H_{c2}	H_{c1}	D^{eff}	H_c^{calc}	H_c^{meas}	B_{c1}	d_c (Å)
2.00	1.03	980	650	0.45	675	757	396	2480
2.75	0.97	885	600	0.43	645	718	320	2900
3.50	0.93	750	512	0.42	570	612	224	3290
4.03	0.90	660	482	0.47	520	550	234	3220
4.90	0.88	480	400	0.45	385	447	117	3690
5.85	0.83	280	235	0.40	240	251	110	4700
6.60	0.71	117	117	···	117	117	···	···

(c) The area under the magnetization curve given by the lines connecting the points $0-(1-D)H_{c1}-H_t-H_{c2}$ was measured using a planimeter and H_c^{meas} was determined from the relation $\int_0^{H_{c2}} M dh = (H_c^{\text{meas}})^2/8\pi$.

The results of this procedure appear in Table I for a Pb–2 at.% In sample. H_c^{calc} is 5–15% smaller than H_c^{meas}. If one plots H_c^{calc} vs t^2 and extrapolates the data to $t=0$, one obtains $H_c^{\text{calc}}(0)$ of 750 ± 25 Oe. Similarly, $H_c(0)^{\text{meas}} = 810 \pm 25$ Oe. This latter value agrees exactly with that interpolated from $H_c(0)$ vs composition points determined from specific-heat data of Culbert, Farrell, and Chandrasekhar.[18] This exact agreement is fortuitous since some hysteresis is present in the magnetization curves from which $H_c(0)^{\text{meas}}$ was obtained. The discrepancy with $H_c(0)^{\text{calc}}$ lies within the experimental error involved in assuming $\kappa_1(t) = \kappa_2(t)$. The values obtained for $H_c(t=0.6)$ and $H_{c1}(t=0.6)$ are in reasonable agreement with the results of Livingston's[19] measurements on highly reversible Pb-In specimens.

DISCUSSION

Clearly the "intermediate state" of our type-II superconductors [i.e., $(1-D)H_{c1} < H < H_t$] is not an intermediate state in the same sense as that of a type-I superconductor, where completely normal and completely superconducting laminae alternate. Rather, there are regions which are almost completely superconducting and others which we contend are in the mixed state with a local magnetic induction approximately that found at H_{c1} when $D=0$. A model is suggested by results shown in Figs. 2 and 3 as well as the flux-lattice observations presented by Krägeloh.[14] This model assumes that for fields between $(1-D)H_{c1}$ and H_t, the volume of the mixed-state domains increases linearly with applied magnetic field. The flux-lattice spacing in each domain remains constant throughout this field interval and is equal to that characteristic of B_{c1}, the magnetic induction which penetrates the sample at an applied field H_{c1} when $D=0$. For fields greater than H_t, the sample is entirely in the mixed state and the lattice spacing now decreases with increasing magnetic field. This model is analogous to the behavior observed in the intermediate state of type-I materials, where $H = H_c$ in the normal domains and their volume increases linearly with applied field. The Sarma,[11] Krägeloh,[14] and Essmann[15] results are the basis of the model that the flux-lattice parameter is constant in all mixed-state regions at a given field and, furthermore, is independent of applied field for $(1-D)H_{c1} < H < H_t$.

Eilenberger and Büttner[20] have recently calculated that the potential between two fluxoids has an oscillatory behavior for low-κ materials. Their calculations predict the existence of an attractive interaction between fluxoids separated by certain distances. However, their equations have not yet been numerically evaluated so that quantitative comparison with experiment is not possible presently. Recently, Cleary[21] has raised objections to the approach taken by Eilenberger and Büttner in determining the vortex interactions in low-κ materials. Consequently the theoretical picture is still unclear.

It should be noted that this model implies the existence of a maximum distance d_c between adjacent fluxoids in superconductors with $\kappa \sim 1$; i.e., flux lines do not form an array with a spacing larger than d_c. This is equivalent to asserting that a first-order transition occurs at H_{c1}, that is, a discontinuity in M should take place, provided that the magnetization is reversible and $D=0$. For the case $D \neq 0$, Fig. 4 schematically presents the magnetization curves to be expected. A discontinuity in slope at H_t should appear on the line AB for various values of D. In fact, such a discontinuity was not observed. This may be due to hysteresis masking this effect, end effects in the sample, or may simply reflect the fact that the transition at H_{c1} when $D=0$ is of second order. If the latter is the case, this implies that the intermediate mixed state observed by the electron-microscopy techniques is in fact metastable.

It should be noted that the assertion of a first-order transition at H_{c1} when $D=0$ is in contradiction to the measurements of Serin[22] as well as the calculations by Kramer[23] based on the data of Finnemore *et al.*[24] on a very pure niobium sample. Serin reported the existence of a λ transition at H_{c1}. For such a λ transition, $B=0$ at H_{c1}. Then flux penetrates in a continuous fashion as H is increased, and d_c would be indefinite for initial flux

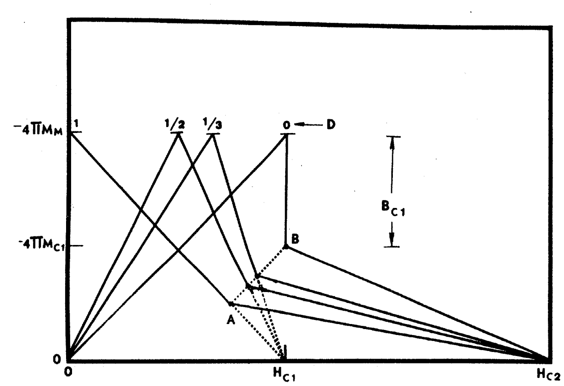

FIG. 4. Idealized magnetic moment as a function of applied field for a low-κ material formed into a longitudinal rod ($D=0$), a sphere ($D=\frac{1}{3}$), a transverse rod ($D=\frac{1}{2}$), and a plate ($D=1$). The heavy dotted line segment AB is the locus of possible points (M_t, H_t) at which the intermediate mixed state terminates for various geometries having demagnetization coefficients in the range $1 > D > 0$.

penetration. It may be that a first-order transition exists for Pb-In at H_{c1} even if Nb exhibits a λ transition.

It should be noted that Fetter and Hohenberg[25] predict that for fields *just* above $(1-D)H_{c1}$ the magnetization should decrease with a slope of $1/4\pi D$ for any type-II superconductor. For higher fields, the dependence of M vs H is not considered in their paper. In particular, the *long* linear region observed in the magnetization curves presented here [for $H_{c1}(1-D) \leq H \leq H_t$] is not explicitly predicted by the Fetter-Hohenberg theory. We believe that the length of the linear region, if this represents a thermodynamically reversible state, implies a first-order transition at H_{c1} when $D=0$. The basis for this assertion is that H_c, and consequently the area under the magnetization curve, must be independent of D. If, as shown in Fig. 4, the "reversible" magnetization curve for $D \neq 0$ consists of two straight lines for $H > (1-D)H_{c1}$, which intersect at H_t, a simple calculation shows that this area is equivalent to that under the magnetization curve for $D=0$. Thus, a first-order transition would exist. Our present data have not proven this unambiguously because our curves have some rounding near H_t despite the mechanical or ac-field "jarring" of the sample to reduce hysteresis. As the hysteresis was reduced by these methods the magnetization curves approached the "ideal" ones pictured in Fig. 4. This suggests that a first-order transition exists at H_{c1} when $D=0$.

We can estimate the maximum B_{c1} and hence the smallest possible critical lattice spacing d_c for a given specimen and temperature in the following manner. Our model assumes that the B_{c1} in each mixed-state region is the same as that which penetrates the sample at H_{c1} when $D=0$. In that case, the largest possible B_{c1} is given by extrapolating the linear portion of the magnetization curve near H_{c2} back to H_{c1}. This determines M_{c1}, the magnetic moment in the mixed state at H_{c1}. Then, using Eq. (1),

$$B_{c1}=H_{c1}+4\pi M_{c1}=H_{c1}-(H_{c2}-H_{c1})/1.16(2\kappa_2^2-1) . \tag{3}$$

Once B_{c1} has been determined, the flux-lattice spacing that exists in the intermediate mixed state can be determined assuming a triangular flux lattice from[26]

$$d_c^2=(2/\sqrt{3})(\phi_0/B_{c1}) , \tag{4}$$

where ϕ_0 is the flux quantum. Table I presents the B_{c1} and d_c calculated in this manner for a Pb–2 at.% In sample and Table II for an In–1.5 at.% Bi sample.

It must be reiterated that these arguments depend crucially on whether or not appreciable hysteresis is present in the magnetization curves. If, for example, hysteresis always exists for $H_{c1}(1-D) < H < H_t$, then the state represented by this linear region would not be thermodynamically reversible and the conservation-of-area argument invoked by us would be invalid. As a consequence, no prediction could then be made about the order of the transition at H_{c1} when $D=0$. The electron-micrograph studies would then have also been made on a metastable state. It should be noted that no attempt was made in those studies to reduce hysteresis.

TABLE II. In–1.5 at.% Bi rod. All fields in Oe.

T (°K)	H_{c2}	H_{c1}	B_{c1}	d_c (Å)
2.00	296	250	12.1	4000
2.35	282	236	12.0	4100
2.55	250	204	10.9	4500
2.75	210	179	11.2	4400
3.00	170	146	10.1	4900
3.25	136	120	9.7	5100
3.50	93	80	7.6	6500

In connection with the latter possibility it should be noted that the presence of a nonvanishing D may for energetic reasons change the nature of the fluxoid interaction and hence force the sample into an intermediate mixed state, whereas the fluxoid interaction might remain repulsive for $D=0$. For this case also, a first-order transition at H_{c1}, when $D=0$, would not be implied by the electron-micrograph studies or by our magnetization curves. However, if the attractive interaction depends on D as well as on κ, the hysteresis observed for a given sample should also depend upon D, i.e., upon the orientation between the sample axis and the applied field. Measurements are underway to test this hypothesis.

Our systematic Pb-In study[16] did not indicate the long linear decrease in magnetization for $H > H_{c1} \times (1-D)$ with a slope of $1/4\pi D$ for a Pb–5 at.% In sample or for those of higher In content. Although the state might have been masked by hysteresis effects, this supports the conclusion of the electron-micrograph studies that an attractive interaction exists between fluxoids only in low-κ superconductors. Further experimental work on this question is also underway.

SUMMARY

Magnetization curves have been studied for low-κ Pb-In and In-Bi samples having nonzero demagnetization coefficients. These samples display type-I behavior, i.e., exhibit an intermediate state at high temperatures where $\kappa < 1/\sqrt{2}$. At lower temperatures where $\kappa > 1/\sqrt{2}$, type-II behavior is apparent. However, in the region just above $(1-D)H_{c1}$ an intermediate-state-like behavior persists in that the slopes of the M-vs-H curves are parallel to those seen for the higher temperatures where $\kappa < 1/\sqrt{2}$ and extend to some value of field H_t. We have interpreted this region to be evidence for the existence of an intermediate mixed state. This view is supported by the electron-microscope studies of Essmann[15] and others on similar and homologous samples. Such work has demonstrated the existence of alternate Meissner and mixed-state regions. The lattice spacing in all mixed-state regions was the same at a given temperature and field. This spacing was found to remain constant as the applied field was increased from $(1-D)H_{c1}$ to H_t, even though the size of the mixed-state regions increased. Based upon such observations, we have developed a model which describes H_t as that field at which the sample is fully in the mixed state with the flux lattice having its maximum spacing $d_c(t)$ for the temperature. If such a state is thermodynamically reversible, the model implies a first-order transition at H_{c1} when $D=0$. It further implies the existence of attractive forces between fluxoids at least for low-κ materials. Both these implications remain to be justified further.

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