PHYSICAL REVIEW VOLUME 112, NUMBER 1 OCTOBER 1, 1958

Magnetization Curves of Superconductive Tin Alloys*

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(Received June 12, 1958)

We have supplemented a previous investigation of flux trapping by obtaining the magnetization curves of very fine impure tin wires in a transverse external field. For annealed specimens (containing In, Bi, or Cd) in which the impurity is completely dissolved and homogeneously distributed, we find that some flux is frozen-in in decreasing magnetic field when the external field equals half the critical value. However, this flux leaks out monotonically as the field is further reduced, and none remains in zero field. In pure specimens virtually no flux trapping occurs. However, in a specimen containing more Cd than was completely soluble, considerable flux remains frozen-in even in zero field. These results confirm our previous inference that suitably prepared and annealed homogeneous alloy specimens are incapable of freezing-in appreciable amounts of magnetic flux. We further conclude that this is due to the fact that the flux which is initially trapped when the specimens begin to become superconducting readily leaks or migrates out, probably because of the tendency of lines of force to contract as suggested by Faber and Pippard.

INTRODUCTION

WE briefly describe the magnetization curves of very fine impure tin wires with their axes transverse to an external field. These measurements were made to supplement our previous observations[1] of flux trapping in impure tin cylinders of much larger diameter. In the earlier investigation the specimens were placed in a transverse magnetic field considerably greater than the critical value, H_c. The field was reduced to zero, and then the amount of magnetic flux remaining in the specimens was determined. Although we found that only very small amounts of flux were retained even in quite impure specimens (particularly after they had been annealed for extended periods), we had no way of knowing from these measurements how closely the magnetization curves of the alloy specimens approached the curve of a pure specimen. In particular, when the applied field was decreased to $H_c/2$, we could not know whether almost all the flux was expelled from the specimen immediately; or whether a considerable amount of flux remained inside, and subsequently leaked out as the applied field was further reduced. This investigation has shown that the second alternative occurs. That is, in fields less than $H_c/2$, the amount of flux in the specimens decreases monotonically to zero as the applied field is reduced to zero.

SPECIMENS AND APPARATUS

The specimens were prepared by placing a small ingot of the alloy in a soft glass tube. The tube was heated in a flame until the metal melted and the glass became soft, whereupon it was pulled by hand into a fine capillary tube containing a metal wire. Pieces of the capillary tube of uniform cross section were selected and cut into 9.5 mm lengths. Our specimens consisted of 50 such short lengths selected at random from pieces cut from several different capillary tubes. The wires which were measured magnetically were left in the glass tubes. The glass was etched off a random sample of the wires and the diameters were measured. These values are listed in Table I; the average deviation in diameter was about 0.01 mm. The other specimens had comparable diameters. Measurements of the residual resistivity of some of the wires confirmed that their impurity concentrations were comparable to the concentration in the ingot. All the specimens were measured one day after they had been made, and remeasured after having been annealed *in vacuo* at 195°C for the times listed in Table I.

We used fine wires in this investigation primarily to make it likely that annealing for the stated periods would produce complete homogenization of the specimens, at least over their diameters. As a result, in order to get an appreciable magnetic moment in small applied fields, we had to measure, as mentioned above, a composite specimen consisting of 50 short lengths of wire. For convenience, we will henceforth refer to such a composite as "the specimen." This procedure has the advantage, in our opinion, of yielding an average behavior roughly comparable to the average obtainable from separate measurements on fifty individually prepared specimens.

The apparatus for determining the magnetization curves of the specimens was similar to one developed by Shoenberg,[2] but was less sensitive. The individual wires were placed in holes in a plastic block. The holes

TABLE I. Characteristics of specimens.

Atomic concentration	Annealing time (days)	Diameter (mm)
Pure	111	0.17
3.11% In	104	0.13
2.18% Bi	97	...
1.75% Cd	54	0.12
0.58% Cd	220	...

* This work has been partly supported by the Office of Naval Research and by the Rutgers University Research Council.
[1] Budnick, Lynton, and Serin, Phys. Rev. 103, 286 (1956).

[2] D. Shoenberg, *Proceedings of the International Conference on Fundamental Particles and Low Temperatures, Cambridge, 1946* (The Physical Society, London, 1947), Vol. 2, pp. 85 and 93.

were regularly spaced 3.2 mm apart. The block fitted into a holder on which was wound a coil of wire. By sending a small current through this coil, the magnetic moment of the coil could be made to cancel the magnetic moment of the specimens induced by an externally applied field. The magnetic field of the coil itself was negligibly small compared to the external field. The effective magnetic moment of the assembly of specimens plus coil was determined by dropping it into a pickup coil connected to a ballistic galvanometer. The galvanometer deflections on dropping the assembly were observed for two coil currents giving positive net magnetic moments and for two giving negative ones. The coil current which would result in zero net moment was then determined by graphical interpolation. The current value so determined is proportional to the magnetic moment of the specimen in the given applied field.

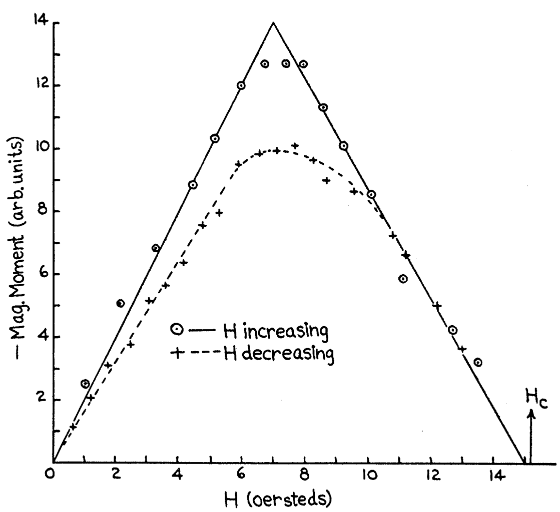

FIG. 1. Magnetization curve of the 3.11% In specimen at $t=0.970$. H_c is the critical field value for bulk specimens.

The procedure followed at any given temperature was to determine the magnetic moment in increasing field until it becomes too small to measure conveniently. The field was then increased to at least twice the critical value and held there for a few minutes. Measurements were then taken in decreasing field. Before changing the temperature to a new value, we always warmed the liquid helium bath to above the transition temperature of the specimens. Whenever the specimens were cooled below the transition temperature, the component of the earth's magnetic field transverse to the wire axes was canceled.

RESULTS

The data obtained with the annealed 3.11% In specimen at a reduced temperature[3] $t=0.970$, are shown in Fig. 1. The data have been normalized to give an initial slope of 2.0. Qualitatively similar curves were obtained at $t=0.93$, and also for the annealed bismuth specimen at the same two temperatures. Moreover, the curves for unannealed In and Bi specimens look quite similar down to temperatures of 0.89.

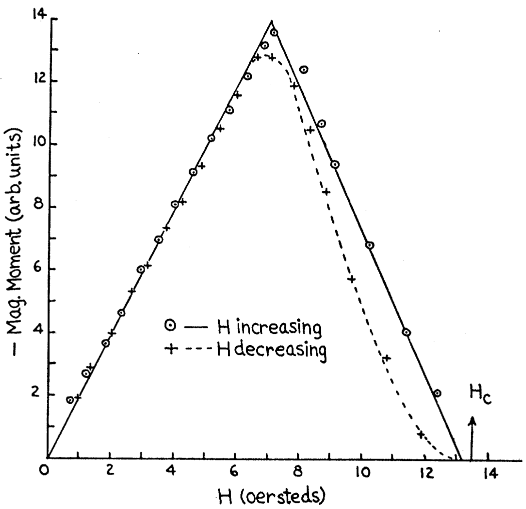

FIG. 2. Magnetization curve of the pure tin specimen at $t=0.975$. H_c is the critical field value for bulk specimens.

It is to be noted in Fig. 1 that in increasing magnetic field, the magnetization curve quite closely approximates the ideal isosceles triangle[4] expected for a superconducting cylinder in a transverse field. However, as the field is decreased from above the critical value, appreciable amounts of flux remain in the specimen near $H_c/2$. This flux then steadily leaks out of the specimen as the field is decreased further, until none remains when the applied field becomes zero.

In contrast, we show in Fig. 2 the data obtained for annealed pure tin at $t=0.975$. A similar curve was obtained at $t=0.93$, and the curves for unannealed pure

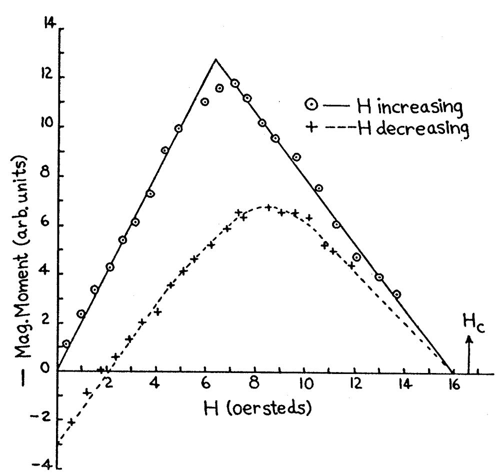

FIG. 3. Magnetization curve of the 1.75% Cd specimen at $t=0.967$. H_c is the critical field value for bulk specimens.

[3] The reduced temperature is $t=T/T_c$, where T is the absolute temperature and T_c the transition temperature. We shall sometimes call this quantity simply the temperature.

[4] B. Serin, in *Encyclopedia of Physics*, edited by S. Flügge (Springer Verlag, Berlin, 1956), Vol. 15, pp. 220–222.

tin wires were also qualitatively indistinguishable from Fig. 2. On the right side of the curve, we attribute the too small values of the magnetic moment, observed when the field is first reduced below H_c, to the supercooling of the specimen, different wires supercooling by differing amounts. The important feature of Fig. 2 is the fact that almost as soon as the field is reduced below $H_c/2$, all the flux is expelled from the specimen, and the magnetization values obtained in decreasing field are indistinguishable from those obtained in increasing field.

By way of further contrast with Figs. 1 and 2, we show in Fig. 3 the magnetization curve at $t=0.967$ of the annealed 1.75% Cd specimen. Similar curves were obtained at all temperatures. The limit of solid solubility of cadmium in tin has been determined metallurgically[5] to be about 1%, and this value was confirmed by residual resistivity measurements made in this laboratory. Thus, the wires in this specimen undoubtedly consisted of a mechanical mixture of two phases. As can be seen from Fig. 3, its magnetization curve is typically similar to those that have been observed previously in alloys.[6] A considerable amount of flux is trapped once the field is reduced below $H_c/2$; and while some of it leaks out when the field is further reduced, about 25% remains trapped inside the specimen in zero applied field. A specimen containing only 0.58% Cd, on the other hand, exhibited magnetization curves which (aside from the absence of supercooling) were practically the same as the curves for the pure specimen. Thus, there is no question that appreciable amounts of flux can be frozen into even these very small wires, provided only that they are sufficiently inhomogeneous.

It took us several minutes to make the four magnetic moment determinations at any given field value. Except occasionally at fields close to half the critical value, we observed no indications that the magnetic moment of the specimen changed during the course of the measurements at a given field. We believe, therefore, that after a change in the external magnetic field, the magnetic moment attained its final value in these specimens in less than one minute.

DISCUSSION

Because of the sharpness of the magnetization curves in increasing field, and also because we were able to obtain good data quite close to the transition temperature, we believe that the individual wires of the specimens had quite similar properties. It must be noted, though, that the magnetization curves of our pure specimen do not show the unusual intermediate-state feature of a "horn" near $H_c/2$ found in small specimens by Desirant and Shoenberg.[7] However, careful scrutiny of their magnetization curves reveals that the horn is pronounced only when their specimen was a single extruded wire; the curves for their composite specimen of wires in glass are quite similar to ours. Thus, we do not feel that our failure to observe this feature is of importance.

We did not choose to investigate whether the transition from a magnetization curve of the type characteristic of the pure specimen (Fig. 2) to the type observed with the alloy specimens (Fig. 1) set in sharply at some critical impurity concentration as has been suggested by Pippard.[8] The main reason for this was that the curves for the two different impurities, bismuth and indium, while qualitatively similar, were quantitatively quite different. Thus, it would seem to be most difficult to arrive at an objective criterion for a transition from one type of curve to the other. On the basis of our experience in these matters, we believe that regardless of what the theoretical situation may be, practically, the transition from one type of behavior to the other will occur gradually with changing impurity concentration.

We believe that this investigation permits three conclusions:

(1) The measurements confirm the inference drawn in our earlier paper[1] that alloy specimens when suitably prepared and annealed are incapable of freezing-in appreciable amounts of magnetic flux.

(2) It would appear that for an alloy an appreciable amount of magnetic flux remains inside the specimen when it first attempts to become completely superconducting in decreasing fields. This flux subsequently leaks or migrates out of the specimen as the field is reduced to zero, probably because of the tendency of lines of force to contract as suggested by Faber and Pippard.[9] It may very well be that, in our small specimens, migration was facilitated simply because the flux had to move only a small distance. But this does not change the conclusion that it is possible, in principle, for magnetic flux to migrate out of specimens of any size, although flaws in the specimen may make this unlikely.

(3) Since the cylinders of larger diameters used in our earlier investigation of flux trapping retained very little flux when well annealed, it seems reasonable to infer that their magnetization curves were similar to the one shown in Fig. 1. It appears therefore that it is possible to produce alloy specimens having magnetization curves approximating the ideal curve at least as closely as the curve in this figure.

ACKNOWLEDGMENTS

This investigation grew out of a conversation with Dr. A. B. Pippard which we gratefully acknowledge. We are also indebted to Mr. A. Siemons for constructing the apparatus.

[5] *Equilibrium Data for Tin Alloys* (Tin Research Institute, 1949).
[6] E.g., D. Shoenberg, Proc. Roy. Soc. (London) **A155**, 712 (1936).
[7] M. Desirant and D. Shoenberg, Proc. Roy. Soc. (London) **A194**, 63 (1948).
[8] A. B. Pippard, Trans. Roy. Soc. (London) **A248**, 97 (1955).
[9] T. E. Faber and A. B. Pippard, in *Progress in Low Temperature Physics*, edited by C. J. Gorter (Interscience Publishers, Inc., New York, 1955), Vol. I, Chap. 9, p. 179.