Studies in Superconductivity

III. Sn, Cb, Ta, and Pb Wires

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The electrical and magnetic properties (in fields of 0–80 oersteds) of Sn, Cb, Ta, and Pb wires of several diameters (25–250 microns) have been studied in the superconducting state. In addition x-ray powder photographs (made with copper $K\alpha$ radiation) have been taken of the wires studied. The experimental results obtained showed that a low resistance ratio, R/R_0 (R_0=room temperature resistance), high transition temperature, and small transition range were always found together for a given metal. For lead the 25,127- and 254-micron wires studied, all exhibited a transition temperature of 7.20±0.01°K, a fact which is attributed to the similar crystalline character of these wires.

IN the course of an investigation of the properties of attenuated superconductors with a view to their use as receiving elements in low temperature radiometry[1,2] we studied several different metals in the form of very fine wires. In our search for a metal wire having the desirable characteristics of high resistance just above the transition region, a narrow transition range (i.e., large value of dR/dT) and a transition temperature in a temperature region easily accessible to us (above 3°K) we tested the electrical and magnetic properties of fine filaments of tin, lead, columbium, and tantalum wires in the superconducting state before selecting the latter as the metal from which a bolometer was constructed.[2] In general our studies were confined to the measurement of the change of the electrical resistance with temperature in several magnetic fields (0–80 oersteds) and with small ($\sim 10^{-4}$ amp.) measuring currents. Certain of the results obtained are new, while others constitute a substantiation of certain isolated experiments reported by other investigators. It is the purpose of this paper to treat of the results obtained for the above-mentioned wires.

EXPERIMENTAL

All the wires studied were obtained from commercial sources and were very pure according to the statements of these manufacturers.[3] These wires varied in diameter from 25 to 250 microns. X-ray powder photographs were taken (with copper $K\alpha$ radiation; sample stationary) of each wire to determine its crystalline character as well as the presence of impurities. The results obtained are listed in Tables I, II, and III. Only the 254-micron columbium and 230-micron tantalum wires showed the presence of impurities, faint lines for SiC appearing on each photograph.

A 30–32 mm length of each specimen was mounted in the low temperature cryostat, previously described, which could accommodate as many as four samples in a single experiment.[4] Each wire was suspended between massive binding posts, to which potential and current leads were affixed.[5] These supports were mounted on a copper plate, but were electrically insulated from it by thin varnished mica sheets. This plate in turn was soldered to the top of a heavy-walled copper helium vessel, thus bringing the wires into excellent thermal contact with the vessel.

The temperature of this helium vessel was

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[1] Andrews, Brucksch, Jr., Ziegler, and Blanchard, Phys. Rev. **59**, 1045 (1941).

[2] Andrews, Brucksch, Jr., Ziegler, and Blanchard, Rev. Sci. Inst. **13**, 281 (1942). Brucksch, Jr., and Ziegler, Phys. Rev. **62**, 348 (1942). These papers will be referred to as Papers I and II, respectively.

[3] Extruded wires of Sn and Pb were obtained from Baker and Company, Newark, New Jersey. Hard-drawn wires of Ta and Cb were purchased from the Fansteel Metallurgical Corporation, North Chicago, Illinois. A special sample of 0.001" Cb sheet was procured from A. D. Mackay, 198 Broadway, New York City.

[4] For a description of the cryostat see Papers I and II. The operation, temperature control, electrical measuring devices, and temperature scale used in this research are discussed in these papers.

[5] Sn and Pb wires were soldered directly to the binding posts by means of woods alloy. Columbium and tantalum wires were mounted by first spot welding a small tab of iron ribbon to each of the wire extremities [see J. Strong, *Procedures in Experimental Physics* (Prentice-Hall, 1939), p. 145]. It was then possible to solder the wires into place.

TABLE I. Tin wires.

Wire diameter microns	Crystallinity	$R/R \times 10^3$ at top of transition	T°_c K zero magnetic field	ΔT° Transition range 10%–90%		T°_c K $H_c=80$ oersteds	ΔT° Transition R/R_0 10–90%		dH_c/dT_c oersteds deg.$^{-1}$
				10%–90%	1%–99%		10–90%	1–99%	
25	Small crystals	31.5	3.46	0.090	0.440	2.69	0.215	0.575	107
254	Small crystals	26.9	3.60	.035	.070	2.81	.220	.615	109
220[1]		0.98	3.724	.023	—	—	—	—	116
190[2]		0.96	3.735	.020	.045	—	—	—	139[3]

[1] Silsbee, Scott, and Brickwedde, J. Research Nat. Bur. Stand. **18**, 295 (1937).
[2] Tuyn and Onnes, Leiden Comm. **181** (1922).
[3] de Haas and Engelkes, Physica **4**, 325 (1937) (for a single crystal).

TABLE II. Columbium wires.

Wire diameter microns	Crystallinity	$R/R_0 \times 10^3$ at top of transition	$R/R_0 \times 10^3$ at bottom of transition	T°_c K zero magnetic field	ΔT° Transition range R/R_0		T°_c K ($H=84$ oersteds)	ΔT° Transition range R/R_0		dH_c/dT_c oersteds deg.$^{-1}$
					10%–90%	1%–99%		10%–90%	1%–99%	
'''										
28	Small crystals[a]	456	5.2	5.09	0.020	0.030	—	—	—	—
254	Small crystals[a]	270	1.0	8.64	.005	.035	8.57	0.015	0.055	1200
300×25	Polycrystalline	87	1.6	9.58	.005	.025	9.50	.025	.120	1050
		35[1]		9.3		.10				

[a] Powder photographs showed extra lines indentified as SiC. These lines disappeared when wire diameter was decreased 20 percent by etching, indicating a surface impurity.
[1] Meissner, Franz, and Westerhoff, Ann. d. Physik, **17**, 593 (1933). This sample was 99.9 percent Cb. Meissner and Franz, Zeits. f. Physik **63**, 558 (1930), also studied a columbium sample of purity 98.43 percent Cb. This specimen was a strip 600×90μ and had a transition range of 8.54–8.69°K. The data for our 254-micron wire compared very well with this sample as regards T_c and R/R_0.

varied in two ways. Below 5°K this was accomplished by regulation of the vapor pressure of the boiling helium (produced by the Simon expansion method). In the range 5–10°K we employed the technique (described in Paper II) of expanding only a part of the highly compressed helium gas present in the above-mentioned vessel. This partial expansion produced partial cooling, but no actual liquefaction of helium. When a sufficiently low temperature had been reached such that the sample became superconducting, expansion was stopped and the helium vessel allowed to warm up gradually, resistance *vs.* temperature measurements being carried out. Because of the large heat capacity of the helium gas usually remaining in the vessel, this rate was quite slow (~0.05°/min.).

Temperatures were measured by means of a constantan resistance thermometer coiled about the helium vessel. The temperature so measured has been called the *relative* temperature and could be measured with a precision of ±0.008°. These *relative* temperatures, in turn were corrected by the aid of a calibrated helium gas thermometer of the type described by Woodcock.[6] It is estimated that these relative temperatures are correct to ±0.05° at 3°K, ±0.1° at 7°K, and ±0.25° at 9.5°K on the absolute temperature scale.[7]

[6] A. H. Woodcock, Can. J. Research **A16**, 133 (1938).

EXPERIMENTAL RESULTS

The experimental observations for tin, columbium and tantalum are summarized in Tables I, II, and III. The symbols used require a few words of explanation. R_0 represents the resistance of the specimen at room temperature. The ratio R/R_0 represents the fraction of this resistance which remains at a given temperature in the transition range. The columns headed 10–90 percent and 1–99 percent represent the temperature interval in which the ratio R/R_0 for the sample changed by this percentage, 100 percent representing the value of the ratio in the normal state just above the transition. Throughout this paper the critical temperature T_c is taken to be that temperature at which the resistance of the sample is one-half the value in the normal state just above the superconducting region.

The variation of resistance with temperature was taken in several constant transverse mag-

[7] For a more complete discussion of the temperature scale see Paper II.

TABLE III. Tantalum wires.

Wire diameter microns	Crystallinity	$R/R_0\times10^3$ at top of transition	T_c ($H=0$) °K	$\Delta T°$ transition range 10–90% R/R_0	dH_c/dT_c oersteds deg.$^{-1}$
28	Polycrystalline	389	<3.19[c]	—	—
127[a]	Polycrystalline	163	4.11	0.030	770
230	Polycrystalline[b]	91	4.28	.010	333
50[1]		35.3	4.38	.030	—
75[2]		260	3.96	.050	1250
127[2]		260	4.07	.055	1020

[a] This sample was also tested when coiled as a helix on a mandrel 3 times the wire diameter. Measurements showed $T_c=3.49°$, $R/R_0\times10^3=124$, 10 percent to 90 percent $R/R_0=0.040°$. The displacement of T_c was considered directly the result of introducing additional strain in the wire by cold working.
[b] Powder photographs showed extra lines identified as SiC. These lines disappeared when wire diameter was reduced 20 percent by etching, indicating surface impurity.
[c] This wire not superconducting at 3.19°K.
[1] Meissner and Franz, Zeits. f. Physik **63**, 558 (1930).
[2] Silsbee, Scott, and Brickwedde, Jr. Research Nat. Bur. Stand. **18**, 295 (1937).

netic fields (0–80 oersteds) with constant current flow in the wire. The average slope of the critical magnetic field (H_c) function of temperature (dH_c/dT_c) was taken graphically from the plot of H_c *versus* T_c.

The crystallinity of the samples is described as large crystals, small crystals, or polycrystalline depending upon the x-ray photographs obtained. The first term implies that the photograph consisted largely of spots, the second spots with some diffraction rings, the third typical diffraction rings.

Three lead wires 25, 127, and 254 microns in diameter were studied; x-ray powder diagrams (copper $K\alpha$ radiation) showed all three wires to consist of large crystals. All three wires showed R/R_0 ratios of 1.8×10^{-3} just above the superconducting region. The transition interval was very small for all wires ($\sim0.04°$ for 1–99 percent R/R_0) and the transition temperatures were all 7.20 ± 0.01°K in zero magnetic field. These results may be compared with those of Onnes and Tuyn.[8] These investigators found $R/R_0=1.0\times10^{-3}$ and $T_c=7.2$°K for a very pure annealed lead crystal. In a field of 84 oersteds the 25-micron wire exhibited a somewhat broader transition ($\sim0.135°$). For magnetic fields varying from 0–84 oersteds $dH_c/dT_c=170$ oersteds per deg.[9]

[8] H. K. Onnes and W. Tuyn, Leiden Comm. **b160** (1922).

DISCUSSION OF RESULTS

The experimental results obtained showed that a low resistance ratio, high transition temperature, and small transition range were always found together for a given sample of the metal. The fact that the smallest wire always exhibited the highest R/R_0 ratio just above the transition as well as the lowest transition temperature and broadest transition is to be attributed, we believe, to the fact that the strains set up in drawing the wires are probably largest in these wires. The lead wires are exceptions to this, but here all wires consisted of large crystals which probably accounts for the sharpness of the transitions and uniform R/R_0 value.

The effect of small magnetic fields (0–80 oersteds) was to depress the transition temperature, the H_c *vs.* T_c curves being essentially straight lines. For Sn, Pb, and Ta the observed depressions are in general agreement with results obtained by other investigators. For columbium the only data available for comparison are the observations of Daunt and Mendelssohn[10] in the temperature range 1.5–4.2°K, where rather high fields were necessary. When their H_c *vs.* T_c data are extrapolated to 9.3°K the value of dH_c/dT_c so obtained is approximately the same as that found for our samples.

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[9] Because of difficulties with the electrical circuits the corresponding data for the 127- and 254-micron wires were not completed. The limited measurements made gave a dH_c/dT_c of approximately the same value as that for the 25-micron wire.
[10] J. G. Daunt and K. Mendelssohn, Proc. Roy. Soc. **A160**, 127 (1937).