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TECHNICAL REPORT NO. 3

PROJECT NO. 116-18

STUDIES OF COMPOUNDS FOR SUPERCONDUCTIVITY

By

W. T. ZIEGLER AND R. A. YOUNG

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NAVY DEPARTMENT, OFFICE OF NAVAL RESEARCH
CONTRACT NO. N6-ori-192, TASK ORDER I
NR 016-406

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SUMMARY

X A number of metal carbides, borides, nitrides, and a hydride, in the form of powders, have been examined for superconductivity down to 1.8° K using a magnetic method.¹⁰ The substances tested included TiC, VC, ZrC, TaC, WC, ZrB₂, NbB₂, TaB₂, WB, MoB, TiB₂, ThB₂, LaN, CeN, NbN, and LaB₂.^{2,45} All specimens were characterized by X-ray diffraction methods and, in most instances, by chemical analysis. Of these substances only NbN gave evidence for superconductivity. The results for NbN are in general agreement with the work of others.^{1,4,10,24} The results for TiC and VC are in agreement with those found by Meissner et al.,^{18,21} who used an electrical resistance method, while those for TaB₂ and CbB₂ are in agreement with observations recently reported by Hulm and Matthias¹³ using a magnetic method. On the other hand, ZrC, TaC, WC, MoB and zirconium boride had previously been reported to exhibit superconductivity above 1.8° K.^{13,18,21}

Particle size measurements were made on the carbide and boride powders. The particles were large enough that magnetic field penetration effects should cause no difficulty, if a penetration depth of 1×10^{-5} cm, observed for pure metals, is assumed to apply to the present compounds.

The failure to observe superconductivity in the substances ZrC, TaC, WC, MoB and zirconium boride in the present study is discussed in terms of a postulated physical distribution of superconducting impurities of undetermined composition which are assumed to account for the superconductivity observed in these compounds by other investigators.

STUDIES OF COMPOUNDS FOR SUPERCONDUCTIVITY*

INTRODUCTION

The occurrence of superconductivity in binary compounds containing a metal and a nonmetal has been known for more than 20 years as a result of the extensive work of Meissner and his co-workers.^{18,20,21} Most of the compounds found to exhibit the effect are binary compounds between superconducting metals and boron, carbon, or nitrogen. On the other hand, the carbides of tungsten and the carbides, a boride of molybdenum, and two nitrides of molybdenum also show superconducting behavior, even though the pure metals do not become superconducting at the lowest temperatures tried ($< 1^{\circ}\text{K}$).

A survey of the literature has shown that, in almost all instances, the occurrence of superconductivity in compounds has been observed by studying the change of electrical resistance with temperature. In all instances in which the effect was observed by this means, the electrical resistance of the substance was found to decrease with temperature from room temperature to the transition temperature in the manner characteristic of metals.³³

The difficulty of preparing these substances as simple stoichiometric compounds has presented the possibility that the observed superconductivity may arise from superconducting filaments (either of the parent metal or a compound) present in the specimen rather than from the bulk specimen itself.

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*A report of this work was presented in less detail by W. T. Ziegler at the Oxford Conference on Low Temperature Physics, held at Oxford, England, August 22-28, 1951.

This doubt has suggested the re-examination of these substances by means of magnetic methods, since, as Shoenberg has pointed out,³¹ these methods should yield information concerning the behavior of the entire volume of the specimen, rather than of possible superconducting filaments.

Relatively little work has been published on the magnetic transition into superconductivity in compounds and metals which are difficult to purify. Shoenberg³⁰ has examined titanium by an a.c. magnetic induction method and reported it not to be a superconductor down to 1.0° K, whereas electrical resistance measurements by Meissner and others^{19,20} had shown that superconductivity set in below about 1.8° K. A similar anomaly has been reported by Shoenberg for uranium.³¹

Recently Hudson,¹¹ using a magnetic induction method, has reported that Mo_2C was not superconducting down to 1.3° K, whereas Meissner and Franz¹⁸ had reported an extended electrical transition into superconductivity in the range 2.5° to 3.1° K. Darby et al.⁶ have reported PbS, PbSe, and PbTe to become superconductors below 5° K using both electrical resistance and magnetic induction methods. Hudson,¹¹ on the other hand, also using a magnetic induction method, was unable to confirm their results for PbTe and PbS.

Studies are now in progress in our laboratory to re-examine by a magnetic induction method a number of compounds previously found to be superconducting by the electrical resistance method. A number of compounds not previously tested for superconductivity are also being studied.

The present paper deals with the results of studies made during the past year on 16 metal carbides, borides, nitrides, and a hydride. The compounds studied were*: TiC, VC, ZrC, WC, TaC, ZrB₂, NbB₂, TaB₂, WB, MoB, ThB₂, TiB_x, LaN, CeN, NbN, and LaH_{2.45}. Of these substances only NbN exhibited superconductivity above the lowest temperature used, about 1.8° K.

All materials were in the form of rather fine powders. They were examined by X-ray powder diffraction techniques using copper K α radiation. The photographs so obtained were analysed with the aid of published data. Partial chemical analyses were also available for all compounds. Particle size measurements were made on the carbide and boride powders. A detailed description of the materials and the method used, together with a discussion of the results obtained follows.

DESCRIPTION OF COMPOUNDS

A. Metal Carbides

The carbides were obtained through the courtesy of Dr. F. H. Horn, Research Laboratory, General Electric Company. They were prepared by the Carboloy Company, Inc., which provided the analyses shown in Table I. In each instance the value of x in the formula MC_x has been calculated from the principal metal content and the "combined carbon" (i.e., total carbon minus free carbon). Where only the carbon content was given, the remainder was taken to be the metal.

*The formulas used are descriptive rather than exact in that they do not, in general, show the exact chemical composition.

TABLE I
CHEMICAL ANALYSIS OF CARBIDES

Carbide Powder	Total Carbon (%)	Free Carbon (%)	Metal (%)	Others (%)	Calculated x in MC _x
TiC	19.03	0.22	79.01	0.57 Fe	0.948
ZrC	10.32	0.34	88.10	0.83 Ta, Nb	0.860
VC	16.18	0.13	82.8		0.820
WC	6.07	0.01			0.989
TaC	6.32	0.23			0.980

Table II summarizes the X-ray diffraction results obtained. A more detailed discussion of each carbide is given below.

TABLE II
COMPOSITION OF CARBIDES

Metal	Calculated x in MC _x	X-ray Diffraction Analysis		
		Major Constituent	Minor Constituent	Not Identified ^b
Titanium	0.948	TiC	Ti (?) ^a TaC (?)	4-5 f and vf lines
Vanadium	0.820	VC	V	Several m, 15 f lines
Zirconium	0.860	ZrC	Cb and/or Ta Zr (?)	Several m, 15-20 f and vf lines
Tantalum	0.980	TaC	None	No lines
Tungsten	0.989	WC	None identified.	About 20 vf to vvf lines

- (a) A question mark (?) implies a few lines corresponding to this structure present.
(b) m = medium; f = faint; vf = very faint, etc.

From a consideration of the chemical analyses and X-ray diffraction results it was concluded that none of the specimens contained more than a trace of free metal. The tantalum carbide sample appeared to be essentially pure TaC. The other carbides all indicated the presence of small amounts of impurities. Both zirconium carbide and vanadium carbide deviated appreciably from the simple stoichiometric formula MC.

a. Titanium Carbide. This material gave an excellent X-ray diffraction photograph with sharp lines. The major constituent was identified as TiC by comparison with the data of Norton and Mowry²⁷ who reported TiC to have the NaCl-type structure with $a_0 = 4.329 \text{ \AA}$. The observed value was $4.34 \pm 0.01 \text{ \AA}$. The presence of a trace of Ti and TaC was also indicated. Four or five faint (f) and very faint (vf) lines could not be identified.

b. Vanadium Carbide. This material gave a fairly good picture with somewhat diffuse lines. The major constituent was identified as VC by comparison with the data of Dawihl and Rix,⁷ who reported VC to have the NaCl-type structure with $a_0 = 4.15 \text{ \AA}$, and the data of Norton and Mowry,²⁷ who found an a_0 of $4.169 \text{ \AA}$. The observed value was $4.17 \pm 0.01 \text{ \AA}$. A trace of vanadium metal was found. Several medium (m) and about 15 f lines were not identified.

c. Zirconium Carbide. This material gave an excellent picture with sharp lines. The major constituent was identified as ZrC by comparison with the data of Norton and Mowry²⁷

who reported ZrC to have the NaCl-type structure with $a_0 = 4.689 \text{ \AA}$. The observed value was $4.70 \pm 0.01 \text{ \AA}$. A trace of niobium and/or tantalum metal was also present. The presence of a trace of zirconium metal was questionable. A small amount of a ZrC-like structure with an a_0 about $0.01 \text{ \AA}$ smaller was also noted. Several m, 15-20 f and vf lines were not identified.

d. Tantalum Carbide. This material gave a good picture though all lines were somewhat diffuse, indicating small particle size. The major constituent was identified as TaC by comparison with the data of Norton and Mowry²⁷ who reported TaC to have the NaCl-type structure with $a_0 = 4.457 \text{ \AA}$. The observed value was $4.46 \pm 0.01 \text{ \AA}$. No extraneous lines were present.

e. Tungsten Carbide. This material gave an excellent picture with very sharp lines. The major constituent was identified as WC by comparison with the data of Becker² who reported WC to have a hexagonal structure with $a = 2.94$ and $c = 2.86 \text{ \AA}$. No evidence for the presence of tungsten metal or W_2C was found. About 20 vf and vvf lines were not identified.

B. Metal Borides

The metal borides, obtained from Cooper Metallurgical Associates, Cleveland, Ohio, were made by direct combination at high temperatures between the powdered metal and elemental boron, both of high purity. All specimens were rather finely

divided dark gray powders. Table III summarizes the results of the chemical and X-ray diffraction analyses. The chemical analyses were supplied with the specimens. A more detailed description of each boride is given below:

TABLE III
COMPOSITION OF BORIDES

<u>Metal</u>	<u>Boron</u> (%)	<u>Calc. x</u> <u>in MB_x</u>	<u>X-ray Diffraction Analysis</u>		
			<u>Major</u> <u>Constituent</u>	<u>Minor</u> <u>Constituent</u>	<u>Not</u> <u>Identified^a</u>
Molybdenum	10.13	0.99	MoB	Mo ₂ B Mo	Few f lines
Tungsten	5.70	1.03	WB	None	No lines
Thorium	8.74	2.04	ThB ₂ (?) ^b	Th ThB ₆ (?)	Many f lines
Titanium	18.33	0.99	TiB _x x = 2-2.5	None identified.	7 f lines
Niobium	18.40	1.94	NbB ₂	Nb (trace)	Many mf, f and vf lines
Zirconium	19.76	2.35	ZrB ₂	None identified.	Few f lines
Tantalum	9.48	1.75	TaB ₂	None identified.	Two vf lines

(a) mf = medium faint; f = faint; vf = very faint.

(b) A question mark implies some lines believed to be due to this substance present.

a. Molybdenum Boride. This material gave an X-ray diffraction photograph with sharp lines which exhibited some spottiness. Comparison of the "d" values from this film with the data given by Kiessling¹⁶ indicated that the major constituent was MoB with the probable presence of traces of Mo₂B

and Mo. Kiessling has assigned a simple tetragonal structure to MoB, with $a = 3.105 \text{ \AA}$ and $c = 16.97 \text{ \AA}$. A few faint lines in the observed pattern could not be identified.

b. Tungsten Boride. This material gave an X-ray diffraction pattern in which all the lines agreed with Kiessling's data¹⁶ for WB. Kiessling assigned a tetragonal structure to WB with $a = 3.115 \text{ \AA}$ and $c = 16.93 \text{ \AA}$. The lines were sharp and showed about the same spottiness as for the molybdenum boride.

c. Thorium Boride. The boride analysis supplied with this material corresponded to the formula $\text{ThB}_{2.04}$. The diffraction pattern from this material contained 69 lines. Of these the 19 strongest lines could be assigned to a face-centered cubic structure having an a_0 of $5.58 \pm 0.01 \text{ \AA}$. A trace of thorium metal was present. Of the remaining lines a few were medium faint, the rest being faint or weaker. Many of these remaining lines could be accounted for by the presence of a small amount of ThB_6 , using the lattice parameter ($a_0 = 4.32 \text{ \AA}$) given by Stackelberg and Neumann.³² However, several of the lines expected to be strongest (as judged by comparison with the published data³² for CaB_6) were absent.

Thorium metal has a face-centered cubic lattice with $a_0 = 5.074 \text{ \AA}$. The unidentified face-centered cubic structure mentioned above may represent the structure of ThB_2 , in which the thorium metal atom lattice has undergone an expansion from $a_0 = 5.074 \text{ \AA}$ to $5.58 \text{ \AA}$ because of the introduction of boron.

d. Titanium Boride. The X-ray diffraction pattern of this material was compared with the very detailed study of Ehrlich⁸ who investigated the structure of the TiB_x system over the range $0 \leq x \leq 3$. Detailed comparison of the 23 lines obtained for this material with the work of Ehrlich indicated that the strongest lines corresponded very closely with the pattern given by Ehrlich for TiB_x where x ranges from 2 to 2.5, with a somewhat less satisfactory fit for x over the entire range 1 to 2.5. This latter assignment leaves seven faint lines unidentified. There was no evidence for free titanium. These results suggest that the chemical analysis may be in error since it yields the formula $TiB_{0.99}$.

e. Niobium Boride. The X-ray diffraction pattern given by this material contained 71 lines of which 39 were medium faint or stronger. Comparison with the data of Norton et al.²⁸ for the diboride, NbB_2 , showed that this substance was the major constituent, which is in good agreement with the formula $NbB_{1.94}$ assigned from chemical analysis. These investigators reported NbB_2 to have a hexagonal structure with $a = 3.086 \text{ \AA}$ and $c = 3.306 \text{ \AA}$. A trace of niobium was also present. One ms, several mf, and 30-40 f, vf to vvf lines could not be identified.

f. Zirconium Boride. Comparison of the diffraction pattern given by this material with the data of Norton et al.²⁸ for ZrB_2 showed that the major constituent was ZrB_2 . These investigators reported ZrB_2 to have a hexagonal structure

with $a = 3.170 \text{ \AA}$ and $c = 3.533 \text{ \AA}$. No free zirconium was found. A few faint and diffuse lines remained unidentified.

g. Tantalum Boride. Comparison of the diffraction pattern given by this material with the data of Norton et al.²⁸ for TaB_2 showed that all except two νf lines could be assigned to the diboride. These investigators reported TaB_2 to have a hexagonal structure with $a = 3.088 \text{ \AA}$ and $c = 3.241 \text{ \AA}$.

C. Metal Nitrides

a. Lanthanum and Cerium Nitrides. These nitrides were made by direct combination at $800\text{--}900^\circ \text{C}$ between the rare-earth metal filings and nitrogen gas at one atmosphere pressure. This method differed from that previously used by Muthmann and Kraft,²⁵ Neumann, Kroger and Kunz,²⁶ and Iandelli and Botti¹⁴ in that the nitrogen absorption could be measured volumetrically, as well as gravimetrically. All operations were carried out in a dry, inert (nitrogen or helium) atmosphere to prevent reaction with air or moisture.

The lanthanum nitride samples were prepared using lanthanum metal obtained from two sources. One of the lanthanum samples, designated as "Cooper No. 2," was obtained from Cooper Metallurgical Associates, Cleveland, Ohio, which also supplied the cerium metal. The second, designated as "Spedding," was obtained through the courtesy of Dr. F. H. Spedding, Iowa State College, Ames, Iowa.

The "Cooper No. 2" lanthanum was analysed by spectrographic, spectrophotometric, chemical, and ion exchange techniques. The

results of these analyses gave: La, 94.8%; Ce, 1.1%; Pr, 0.2%; Nd, 0.4%; other rare earths, 0.2%; Si, 0.7% (estimated); Fe, 0.8% (estimated); carbon and others, present; total accounted for 98.2%. On the basis of the overall analysis the total rare earth content, calculated as lanthanum, has been taken as 97%.

Spectrographic analysis of the "Spedding" lanthanum showed that it contained 0.13% Be, 0.1% Mg and traces (estimated to be a few hundredths of one per cent each) of calcium, aluminum, and iron. The sample appeared to be free of other rare earths. The total rare earth content, expressed as lanthanum, was found by direct precipitation as oxalate under carefully controlled conditions to be 97.3%. This low result may arise from the presence of carbon or dissolved gases such as oxygen. In view of the presumed high purity of this material the lanthanum content has been taken somewhat arbitrarily to be 100%.

The Cooper cerium metal was analysed by the same general techniques used for lanthanum. These analyses gave: Ce, 92.2%; La, none; Pr, 0.4%; Nd, 0.6%; other rare earths, none; Si, 0.3%; Fe, 2.5%; carbon and others, present; total accounted for, 96.0%. The total rare earth content, calculated as cerium, was found by direct precipitation as oxalate to be 94.9%. On the basis of all facts concerning the analyses, the total rare earth content, expressed as cerium, has been taken to be 95%.

A summary of the details of preparation of the nitrides is given in Table IV. The two lanthanum nitrides were black free-flowing powders, which appeared to be homogeneous. The formula of these products was LaN_x , $0.91 \leq x \leq 0.99$, where the

value of x depended somewhat upon whether the volumetric or the gravimetric measurement was used in the calculation. Muthmann and Kraft²⁵ and Neumann et al.²⁶ had observed $x = 1 \pm 0.08$ and $0.824 \leq x \leq 0.830$, respectively.

TABLE IV
NITRIDATION OF LANTHANUM AND CERIUM METALS

Expt. No.	Metal	Metal Filings Weight (g)	x in LaN_x^a		Experimental Conditions ^b
			Weight Increase	Vol. Meas.	
26	Lanthanum (Cooper No. 2)	0.4240	0.99	0.95	20 hours at 920°
29	Lanthanum (Spedding)	0.5626	0.97	0.91	20 hours at 920°
30	Cerium (Cooper No. 1)	0.8290	0.86	0.86	1.5 hours at 700°, 18 hours at 800°

(a) The value of x was calculated on the assumption that the original metal samples had the following purities: Cooper No. 2, 97% La; Spedding, 100% La, Cooper No. 1, 95% Ce.

(b) Temperatures in °C. Nitrogen pressure, 1 atm.

The cerium nitride was a reddish brown powder with a faint luster. It had the formula CeN_x , $x = 0.86$. Muthmann and Kraft²⁵ found an x of 0.89 as an average of nine experiments; Neumann et al.²⁶ reported that x varied from 0.71 to 0.87.

Powder X-ray diffraction studies of the lanthanum nitride products showed no significant difference between them. The structure was found to be of the NaCl-type with $a_0 = 5.284 \pm 0.004$ kx-units, in agreement with the observations of Iandelli and

Bottl¹⁴ who reported the same structure with $a_0 = 5.27$ kx-units. Since the nitride has the same lattice and lattice parameter as face-centered cubic lanthanum metal with $a_0 = 5.285 \pm 0.005$ kx-units, it was impossible to determine whether or not any unreacted lanthanum was present in the products.

The cerium nitride also exhibited a face-centered cubic lattice with $a_0 = 5.014 \pm 0.005$ kx-units. All lines observed could be assigned to this structure. Iandelli and Bottl¹⁴ have reported CeN to have the NaCl-type structure with $a_0 = 5.01$ kx-units.

b. Niobium Nitride. This material was supplied by Dr. F. H. Horn, Research Laboratory, General Electric Company, Schenectady, New York, as part of a cooperative study on Nb-Ta alloys and their nitrides. The nitride was prepared by heating niobium metal powder for 4 to 4-1/2 hours at 1500° C in pure dry nitrogen. The metal was obtained from Fansteel Metallurgical Corporation; it was stated to be 99.9% pure. The nitride was in the form of a coarse gray powder.

X-ray examination of the nitride showed it to be primarily NbN with $a_0 = 4.37 \pm 0.01$ kx-units.¹⁰ A trace of Nb and/or Ta metal appeared to be present. A large number of f to vvf lines could not be identified. These lines did not fit the pattern for Nb₂N reported by Brauer.³

D. Lanthanum Hydride

The lanthanum hydride was prepared by direct combination between lanthanum metal filings (Cooper No. 3) and pure dry hydrogen gas at 210-250° C. The lanthanum metal had a

composition very similar to that reported for the Cooper No. 2 specimen. On the basis of the overall analysis the total rare earth content, calculated as lanthanum, was taken to be 97%. The apparatus used was the same as in the preparation of the rare earth nitrides.

The products were black friable powders. They were handled at all times so as to prevent contact with moisture and air. The details of the preparations are shown in Table V. The resulting products had a composition corresponding approximately to $\text{LaH}_{2.45}$. Muthmann and Kraft²⁵ and Rossi,²⁹ using a similar method, have reported similar products having the approximate formulas LaH_3 and $\text{LaH}_{2.4}$, respectively.

Powder X-ray diffraction photographs gave a pattern corresponding to a f.c.c. lattice with $a_0 = 5.625 \pm 0.007$ kx-units, which is in good agreement with the value of 5.62-3 kx-units reported by Rossi.²⁹ The lines were somewhat diffuse. The pattern of f.c.c. lanthanum ($a_0 = 5.285$ kx-units) was not observed in these products.

TABLE V
PREPARATION OF LANTHANUM HYDRIDE

Expt. No.	Weight of La Metal (g)	x in LaH_x ^b		Experimental Conditions ^c
		Wgt. Inc.	Vol. Meas.	
37 ^a	0.8382	2.40	2.44	Reaction started, ^d 210°; 8 hours at 290° $\pm 10^\circ$.
38	0.9721	2.66	2.47	Reaction started, ^d 225°; 19-1/2 hours at 290° $\pm 10^\circ$.
^(a) This product used in superconductivity studies. ^(b) Calculated on basis that metal contained 97% La. ^(c) Temperatures in °C. Hydrogen pressure was 1 atm. ^(d) Reaction took place rapidly at this temperature.				

EXPERIMENTAL METHOD

The cryostat used differed in only minor details from one described by Horn and Ziegler.¹⁰ The Simon expansion method was used to produce the liquid helium. From the main helium reservoir was suspended the experimental chamber, into which helium could be condensed at will by bringing low pressure helium gas into contact with the main helium reservoir. The copper experimental chamber (Figure 1A) consisted of two parts, an upper section to which the gas and the constantan resistance thermometers were attached, and a lower section in which the specimens to be tested for superconductivity were located.

The temperature of the experimental chamber was measured by means of a helium gas thermometer. This thermometer was of the type described by Mendelssohn,²² except that helium pressure was measured both by a Bourdon gauge and a capillary mercury manometer. The thermometer was filled with helium gas at one atmosphere pressure while at room temperature and then sealed off.

Temperatures were calculated from the helium pressure on the assumption that the volume of the capillary tubing (0.020 inch i.d. copper-nickel alloy) connecting the manometer and gauge volume, V_r , at room temperature, and the volume of its thermometer bulb proper, V_b , was negligible. Gas imperfection was taken into account by making use of the virial coefficients given by Keesom.¹⁵ The ratio of the two volumes, V_r/V_b , was determined by experiment at the normal boiling point of hydrogen, the triple point of hydrogen and at the normal boiling point of helium. This ratio had a value of 11.9

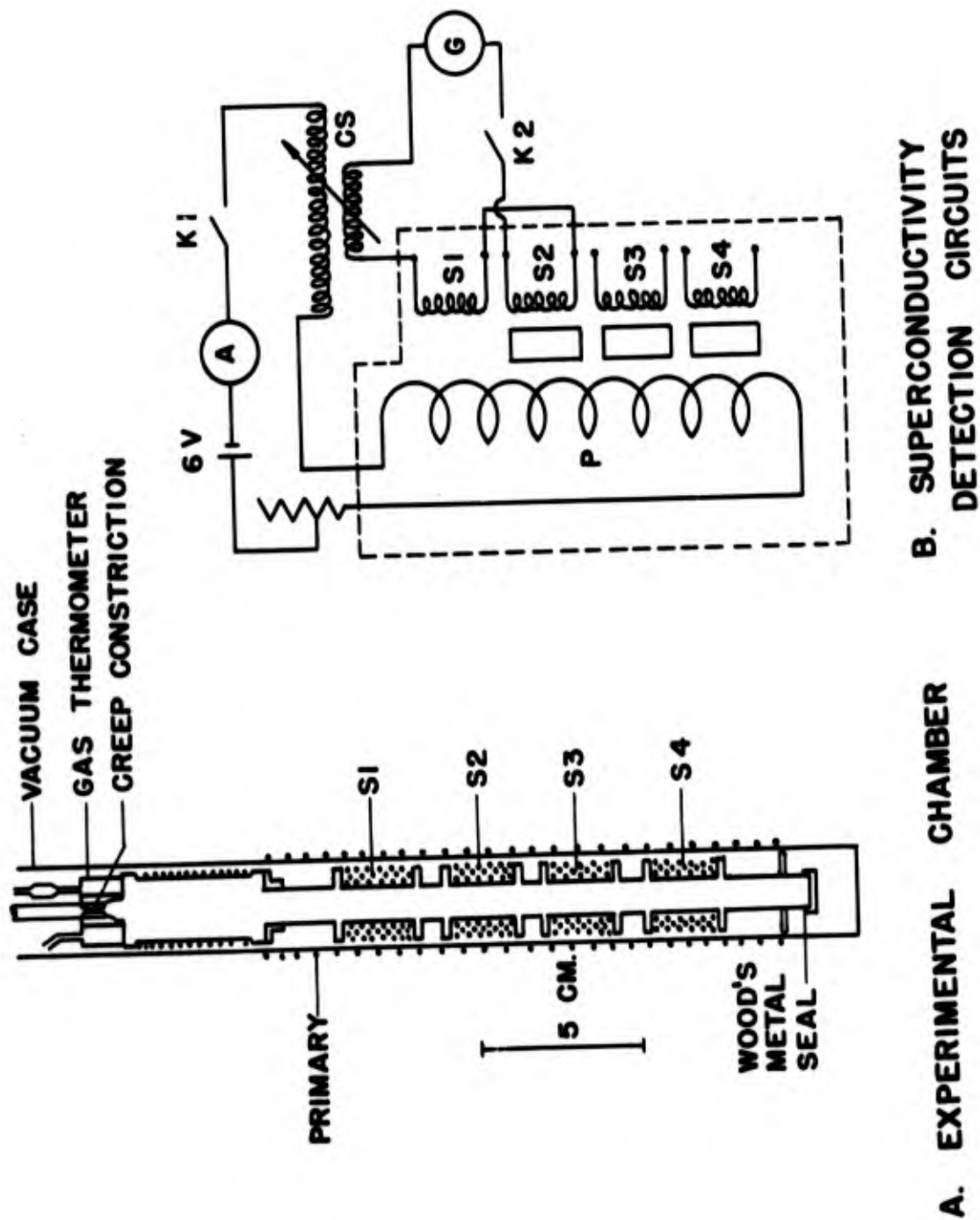


Figure 1. Magnetic Measurements System

at the two hydrogen points and a value of 12.4 at the helium point. V_b was equal to 1.6 cc.

The sensitivity of the gas thermometer was about 0.04° per mm. Hg in the range 3 to 5° K and 0.08° per mm. Hg in the range 14- 20° K. Readings were made to 0.5 mm. In the range below about 2.6° K the gas thermometer acted as a vapor pressure thermometer. The vapor pressure of the liquid helium in the experimental chamber was also used to determine the temperature below 4.2° K.

The absolute accuracy of the temperature scale is believed to be about 0.05° in the range below 5° K, 0.1° in the range $5-7^\circ$ K and 14- 20° K and several tenths of a degree in the range between 7° and 14° K.

A resistance thermometer, constructed of B and S No. 40 constantan wire, also was used to follow the temperature changes of the experimental chamber. This thermometer had a resistance of 1069 ohms at 4.2° K and a dR/dT of approximately one ohm per degree over the range 1.8 to 20° K. Temperature changes of 0.01° - 0.02° could be detected with this thermometer.

The system for making the magnetic measurements consisted of a primary coil, wound on the lower end of the brass vacuum case, and four equally spaced secondary coils S1, S2, S3, and S4, wound on the outside of the experimental chamber and coaxial with the primary coil. The primary coil, constructed of 1117 turns of #30 AWG copper wire, was 15.1 cm. long. The secondary coils consisted of about 3000 turns of #40 AWG copper wire. Each coil was 2.2 cm. long. In all experiments

coil S1, a compensating coil, was always empty, the specimens under test being located in coils S2, S3, and S4.

The circuits for detecting superconductivity are shown in Figure 1B. The experimental procedure was as follows: with the experimental chamber at a fixed temperature, coil S1 and another coil, for example coil S2, were connected by means of a suitable external switching arrangement in such a manner that the voltages induced in them opposed each other when the tap key (K1) in the primary circuit was closed. A primary current of 0.27 amperes was usually used. With the tap key (K2) closed, the galvanometer ordinarily showed a deflection when the primary was energized. This unbalance could be reduced to zero by suitably adjusting an external variable inductance, CS. With the circuit balanced and K2 closed, K1 was closed at 10-15 second intervals during a series of measurements in which the temperature of the experimental chamber was slowly lowered (or raised); any unbalancing of the coils was observed as a galvanometer deflection.

The powdered samples were generally mounted in a 7 mm. o.d. pyrex glass tube, partly open at the top end, and were in direct contact with the helium. In a few instances (TiC, VC, $\text{LaH}_{2.45}$, CeN, and LaN), the measurements were made on samples in pyrex capsules sealed at room temperature under 20 cm. Hg pressure of helium. The samples had weights varying from 0.7 to 2.5 gm., and usually had a length of 20-22 mm. and a diameter of 4.5-4.9 mm. The actual volume was determined from the weight of the sample and published or estimated density.

EXPERIMENTAL RESULTS

The experimental results for the various compounds tested are summarized in Table VI, VII, and VIII, in comparison with experimental work of other investigators. In these tables the major constituent given was that found by X-ray diffraction analysis. As mentioned previously, of the substances tested, only NbN was found to exhibit a magnetic transition into superconductivity above about 1.8° K, the lowest temperature used in these studies. Tungsten boride, thorium boride, lanthanum nitride and hydride, and cerium nitride had not previously been tested for superconductivity.

The sensitivity of the magnetic detection system was such that the NbN specimen gave a total galvanometer deflection of 4.2 cm. in passing from the normal to the superconducting state. On the basis of this and other measurements, it was estimated that the occurrence of superconductivity could have been detected in a volume corresponding to five to ten per cent of the volume of the samples used.

Reference to Table VI and VII shows that the failure to observe superconductivity in ZrC, TaC, and WC and zirconium boride is in disagreement with the observations of Meissner et al.,^{18,21} who used the electrical resistance method. The nonappearance of superconductivity in MoB is in disagreement with the recent observations of Hulm and Matthias,¹³ who found a transition at 4.4° K using a magnetic induction method. On the other hand, the magnetic transition observed for NbN is in general agreement with the results found by

TABLE VI
SUPERCONDUCTIVITY OF METAL CARBIDES

Carbide	Major Constituent	Transition Temp. ^a and Range, °K		Method ^c	Observer ^d
		This Research	Lit.		
TiC _{0.95}	TiC	(1.8°) ^b	1.1° (?)	R	Meissner(1)
			(1.31°)	R	Meissner(2)
VC _{0.82}	VC	(1.8°)	(1.33°)	R	Meissner(1)
ZrC _{0.86}	ZrC	(1.8°)	3.7°,	R	Meissner(2)
			3.3°-4.1°		
TaC _{0.98}	TaC	(1.8°)	9.3°,	R	Meissner(1)
			9.2°-9.4°	R	Meissner(2)
			7.6°-9.5°		
WC _{0.99}	WC	(1.8°)	3.4°,	R	Meissner(1)
			2.5°-4.0°		

(a) Temperature corresponding to 50 per cent of total resistance change in transition.

(b) Temperatures in parentheses mean substance not superconducting down to this temperature.

(c) R = electrical resistance method.

(d) References: (1) Meissner and Franz, Z. Physik **65**, 30 (1930).

(2) Meissner, Franz, and Westerhoff, ibid., **75**, 521 (1932).

TABLE VII
SUPERCONDUCTIVITY OF METAL BORIDES

Boride	Major Constituent	Transition Temp. and Range, °K		Method ^b	Observer ^c
		This Research	Lit.		
MoB _{0.99}	MoB	(1.74°) ^a	4.4°	M	Hulm(1)
WB _{1.03}	WB	(1.77°)			
ThB _{2.04}	ThB ₂ (?)	(1.77°)			
TiB _{0.99}	TiB _{2-2.5} (?)	(1.8°)	(1.26°)	R	Meissner(2)
NbB _{1.94}	NbB ₂	(1.8°)	(1.27°) (9.5°)	M R	Hulm(1) Aschermann(3)
ZrB _{2.35}	ZrB ₂	(1.8°)	3.1°, 2.9°-3.3°	R	Meissner(2)
TaB _{1.75}	TaB ₂	(1.8°)	(1.32°)	M	Hulm(1)

(a) Temperatures in parentheses mean substance not superconducting down to this temperature.

(b) R = electrical resistance method. M = magnetic induction method.

(c) References: (1) Hulm and Matthias, Phy. Rev. **82**, 273 (1951).
(2) Meissner, Franz and Westerhoff, Z. Physik **75**, 521 (1932).
(3) Aschermann, Friederich, Justi and Kramer, Physik Z. **42**, 349 (1941).

TABLE VIII
SUPERCONDUCTIVITY OF NITRIDES AND HYDRIDE

<u>Compound</u>	<u>Transition Temp. and Range, °K</u>		<u>Method^c</u>	<u>Observer^d</u>
	<u>This Research</u>	<u>Lit.</u>		
NbN	16.0°, 14.6°-16.8°	14.7°	M,R	Horn(1)
		13.9°-15.3°		
		15°-16.5° ^b	R	Aschermann(2)
		14°-15°	R	Milton(3)
		15.2°	M	Cook(4)
		13.5°-16.2°	M	Cook(4)
LaN _{0.97} (Cooper No. 2)	(1.8°) ^a			
LaN _{0.94} (Spedding)	(1.8°)			
CeN _{0.86} (Cooper)	(1.8°)			
LaH _{2.45} (Cooper No. 3)	(1.8°)			

- (a) Temperatures in parentheses mean substance not superconducting down to this temperature.
 (b) On warming only. On cooling, temperatures as high as 23° K obtained.
 (c) M = magnetic induction method. R = electrical resistance method.
 (d) References: (1) Horn, Dissertation, Johns Hopkins Univ., Baltimore, Md., 1942; Horn and Ziegler, J. Am. Chem. Soc. **69**, 2762 (1947).
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both electrical resistance and magnetic induction methods. 1,4,9,10,24

The failure to observe superconductivity in the lanthanum nitrides and hydride is interesting since the transition temperatures of the three lanthanum metal specimens had previously been found to be³⁴: Cooper No. 2, 3.2°; Cooper No. 3, 3.5°; Spedding, 5.2°. The cerium metal from which the nitride was prepared was not superconducting down to 1.8° K.

Several reasons for the failure to observe superconductivity in ZrC, TaC, WC, and MoB suggested themselves. One of these was that the apparatus was not functioning properly, possibly due to thermal nonequilibrium. However, the detection of superconductivity in bulk specimens of lanthanum, lead, and tin metals, lanthanum filings and powdered NbN with the apparatus seemed to exclude this possibility.

The apparatus was further checked by studying a tin powder which consisted of spheres having a size range of approximately 15 to 50 microns. This material was obtained through the courtesy of Dr. M. C. Steele of the Naval Research Laboratory, Washington, D. C. The powder was free-flowing; it had a yellowish appearance indicating the presence of some oxide on the surface of the tin particles. Tests of this powder gave a magnetic transition into superconductivity over the range 3.63° to 3.49° K. This is somewhat lower than the generally accepted transition temperature (3.7° K) for bulk tin. However, the calculated maximum field generated by the measuring primary was 27 oersteds, which could be expected to produce a lowering as great as 0.15°.

The measurements on LaN, CeN, LaH_{2.45}, TiC, and VC were made on samples sealed in capsules under 20 cm. Hg helium pressure at room temperature. The possibility that the helium gas was adsorbed at low temperatures, thus resulting in poor heat transfer, is not entirely excluded. However, experiments with lanthanum metal filings sealed under similar conditions showed no significant hysteresis in the transition at 4° K. In general, specimens were kept below 4.2° K for as long as an hour, and below the lambda point (2.19° K) for times ranging from 3 to 18 minutes.

In order to obtain some information on the possible magnitude of field penetration effects, the particle size distribution of the carbide and boride powders was determined by measuring microscopically the linear dimensions of 100 particles which had been suitably dispersed on a glass slide.⁵ The particle size distribution observed was expressed in terms of the smallest diameter, d_v , possessed by those particles whose mass constituted 50 per cent of the total mass of the sample.⁵ The appearance of the particles of all powders examined could best be described as roughly spherical. Table IX summarizes the results obtained. It will be noted that the MoB, ZrC, and WC specimens consisted of relatively large particles. Electron-microscopic examination of the TaC powder showed that it contained a considerable fraction below one micron in size.

The field penetration depth in powders of alloy systems such as the carbides and borides has apparently not been investigated. If one takes for the penetration depth, λ , the

rough value of 1×10^{-5} cm. observed in metals,¹⁷ it would appear that the values of d_v for all substances studied was sufficiently large so that field penetration would not be expected to play a significant role in the present measurements.

TABLE IX
PARTICLE SIZE MEASUREMENTS

Borides		Carbides	
Substance	Particle Size ^a d_v (microns)	Substance	Particle Size d_v (microns)
WB	1.3	TaC	1.1
ThB ₂	1.3	TiC	2.3
NbN	1.8	VC	3.7
TaB ₂	1.8	ZrC	6.2
TiB _x	2.0	WC	11.2
MoB	3.2		
ZrB ₂	3.4		

(a) d_v = smallest diameter of those particles whose mass constituted 50 per cent of the total mass of the sample.

The disagreement between the results of Meissner and co-workers^{18,21} and the present work regarding the occurrence of superconductivity in TaC, WC, and ZrC, might be explained, as Shoenberg^{30,31} has suggested for titanium and uranium, by assuming the presence of superconducting filaments in Meissner's specimens of these carbides. A similar explanation would resolve the conflicting observations of Hudson¹¹ and Meissner and Franz¹⁸ regarding the superconductivity of Mo₂C. Unfortunately, no specimens of these substances appear to have been studied by both electrical and magnetic methods.

Hudson,¹² using the magnetic induction method, has made an interesting study of two Sn-Ge "alloys" containing 2.3 and

9.3 atomic per cent of tin, respectively, in which the tin is concentrated along the grain boundaries. He observed a rather broad diamagnetic transition beginning at 4° K. Hudson attributed this behavior to the presence of superconducting filaments of tin. Calculations by him indicated that approximately 50 per cent of the volume of the alloy of lower tin content and the entire volume of the alloy of higher tin content participated in the magnetic transition.

In view of these observations of Hudson on the Sn-Ge alloys, it would appear that the precise physical structure of the "superconducting filaments" might be an important variable. Thus, if the filaments are thread-like, superconductivity might be observed electrically but not magnetically. On the other hand, if the superconducting filaments possess a sponge-like structure²³ with the bulk material in the holes of the sponge, as in the Ge-Sn alloy experiments of Hudson,¹² then both an electrical and a magnetic transition might be observed. The physical structure of such filamentous inclusions might be expected to depend considerably on the method of preparation.

The disagreement between the results of Hulm and Matthias¹³ and the present work regarding the superconductivity of MoB may be due to some such difference in the arrangement of a superconducting inclusion. The conflicting observations of Darby et al.⁶ and Hudson¹¹ on the superconductivity of PbTe and PbS, mentioned earlier, might be explained in a similar fashion.

The possibility that superconductivity in many compounds may be due to rather specific amounts of trace impurities, distributed in a homogeneous manner through the bulk material and which make possible a superconducting state has not been excluded.

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