ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia

FINAL REPORT

PROJECT NO. 116-18

INVESTIGATION OF FUNDAMENTAL PROPERTIES OF ELEMENTS AND THEIR COMPOUNDS INCLUDING THE RARE EARTHS AT VERY LOW TEMPERATURES WITH PARTICULAR EMPHASIS UPON SUPERCONDUCTIVITY

By

W. T. ZIEGLER

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

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JUNE 30, 1957

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I. INTRODUCTION

Contract No. N6-ori-192, Task Order I (Code No. NR 016-406), between the Office of Naval Research and the Engineering Experiment Station of the Georgia Institute of Technology was initiated on October 1, 1946 and terminated on June 30, 1957. For the period July 1, 1955 to June 30, 1957 it has operated under a "no-additional-funds" extension. Beginning on July 1, 1953, the equipment developed for use under the contract was also used, with permission of ONR, for related work on a National Science Foundation grant (G-447).

The contract has been concerned in its entirety with research at low temperatures on the fundamental properties of the elements and their compounds including the rare earths, with particular emphasis on superconductivity. This broad program has led to contract supported studies dealing with 1) crystal structure and superconductivity of rare earth metals and rare earth compounds, 2) superconductivity of transition metal and other compounds, 3) rare earth chemistry, and low temperature calorimetry. The results obtained in these areas, described in greater detail in following sections, have been published in 7 papers, 4 technical reports (see Appendix, A) and 5 Master of Science theses (2 in Physics and 3 in Chemical Engineering, see Appendix, B).

At the inception of the contract no facilities for low temperature research existed at Georgia Tech, although plans to develop such a laboratory were already underway and some precision electrical equipment had been purchased. Accordingly considerable time was spent in building a hydrogen liquefier [illegible] Simon expansion-type helium liquefier (cap. 70 ml of liquid helium expansion) and the necessary auxiliary equipment. This equipment was installed in space provided in a new building which was occupied October 21, 1947. Liquid helium was first produced on April 20, 1948.

A very important concept in the operation of this contract has been a steady effort to relate the research being carried out to the graduate program of the institution. This has been done, whenever possible by 1) providing thesis material (topics) which were either a part of or closely related to the main research objectives of the contract, 2) employing or using students (rather than full-time employees) as research assistants, and 3) aiding research programs in individual departments through consultation, occasional calibration of equipment, etc.

Altogether 20 graduate students, 14 M. S. (10 in Chemical Engineering, 2 in Chemistry and 2 in Physics) and 6 Ph. D. (4 in Chemical Engineering and 2 in Chemistry), were associated with the project at one time or another. In addition 8 undergraduate seniors (7 in Chemical Engineering and 1 in Mechanical Engineering) were employed as technical assistants. The preponderance of students in Chemical Engineering arose from the fact that the principal investigator (Dr. W. T. Ziegler) is associated with the School of Chemical Engineering. These students produced 5 theses (3 M. S. in Chemical Engineering and 2 M. S. in Physics) directly connected with the contract (Appendix B) and 6 theses (3 M. S. and 3 Ph. D. in Chemical Engineering) which contributed indirectly to the research objectives of the contract (see Appendix, C).

It should perhaps be pointed out that the Ph. D. program at this institution was not started until the fall of 1947, when the students were first accepted for work on this degree in the School of Chemical Engineering. This contract has contributed very significantly to the development of the Ph. D. program at Georgia Tech, particularly in Chemical Engineering.

II. RESEARCH RESULTS

A. Superconductivity of Rare Earth Metals and Rare Earth Compounds

One of the research objectives of this contract was to examine the rare earth metals and certain of their compounds for superconductivity. When this work was begun in 1946 only lanthanum and cerium metals had been examined for superconductivity. Mendelssohn and Daunt[17] had found lanthanum to be a superconductor with a transition temperature (T_c) of 4.71° K. This result was confirmed by Shoenberg[20], who found a T_c of 4.2° K. McLennan et al[13], using the electrical resistance method, had earlier failed to find superconductivity in both lanthanum and cerium. Magnetic measurements by us on commercially available specimens of lanthanum[3, 25], cerium[3, 25], praseodymium[26] and neodymium[26] confirmed the fact that lanthanum was a superconductor, but indicated that the latter three elements were not superconductors above 2° K, the lowest temperature reached in these experiments.

The purity of these materials was not high, so it was intended to prepare the pure metals from pure salts which were to have been prepared from crude rare earth concentrates. However, the rapid development of ion exchange techniques for separating the rare earth elements, given tremendous impetus because of the occurrence of these elements as products of nuclear fission, led during 1947-1950 to the production by Dr. F. H. Spedding and his co-workers of pure specimens of a number of the rare earth metals. Several of these materials (Ce, Pr, Nd) were examined by Goodman[4] for superconductivity to 0.25° K with negative results.

These developments led us to concentrate our interest on lanthanum metal. Some of these experiments were carried out on lanthanum metal which was made available to us by Dr. Spedding.

Studies of the phase relations[27] in the lanthanum metal system showed that both the hexagonal close-packed (hcp) and face-centered cubic (fcc) modifications were superconductors. The fcc modification was prepared in well characterized form and its superconducting transition temperature was found to be about 5.5° K for the purest specimens[27]. The hcp modification could not be prepared in a strain-free state, but in a strained condition it was found to be a superconductor near 3.9° K[27]. X-ray diffraction and heat treating studies showed that the phase change hcp⇌fcc was more complicated than had previously been supposed.

Studies of rare earth compounds were limited to lanthanum and cerium nitrides, and lanthanum hydride having the compositions:[29, 30] $LaN_{0.97}$, $CeN_{0.87}$ and $LaH_{2.45}$. None proved to be a superconductor above 1.8° K.

B. Superconductivity of Transition Metal and Other Compounds

The occurrence of superconductivity in binary compounds containing a metal and a non-metal has been known for more than 20 years as a result of the extensive electrical resistance studies of Meissner and his co-workers[14, 15, 16]. The difficulty of preparing many of these compounds in pure form presented the possibility that the superconductivity observed in certain of them might have arisen from superconducting filaments (either of the parent metal or a compound) present in the specimen in small amounts, rather than from the bulk specimen itself. This doubt suggested the re-examination of these substances by means of magnetic methods, for, as Shoenberg[21] has pointed out, these methods should yield information concerning the behavior of the entire volume of the specimen rather than of the possible superconducting filaments alone.

It appeared worthwhile to re-examine for superconductivity using a magnetic method, a number of compounds previously studied using the electrical resistance method. The substances studied[30] were TiC, VC, ZrC, TaC, WC, ZrB_2, NbB_2, TaB_2, WB, MoB, TiB_x, ThB_2, and NbN. Of these substances only NbN gave evidence of superconductivity down to 1.8° K. The results for NbN was in general agreement with the work of others. The failure to observe superconductivity in TiC and VC is in agreement with the electrical resistance measurements of Meissner et al[14, 16], while the absence of superconductivity in ZrC, TaC and WC is in disagreement with the observations of these investigators. Matthias and Hulm[11], also using a magnetic method, likewise found no evidence for superconductivity in WC down to 1.28° K.

The failure to observe superconductivity in NbB_2 and TaB_2 is in agreement with the magnetic studies of Hulm and Matthias[9]. These investigators also reported TiB_2 not to be a superconductor down to 1.28° K[11], in agreement with our results for a boride having the composition $TiB_{2-2.5}$ and with those of Meissner, et al[14], for a titanium boride specimen of unstated composition. No transition was observed in ZrB_2, whereas Meissner, et al[15], using the resistance method, reported a transition near 3.1° K in a zirconium boride of unstated composition.

The disagreement between the negative result for MoB obtained by us and the positive result reported by Hulm and Matthias[9] for this compound, together with the observations of Hudson[6] on Sn-Ge alloys, led to the proposal that certain sponge-like distributions of superconducting impurities in compounds might result in large magnetic effects which could be mistaken for superconduction of the major phase. The later work of Matthias and Hulm[11] on MoB appears to support this view. A similar explanation has been advanced independently[5, 7, 8] to account for the conflicting magnetic observations in PbS.

Superconductivity studies on compounds were continued after June 30, 1953 under a grant (G-477) from the National Science Foundation for the period July 1, 1953 to July 1, 1955. This grant was extended on a "no-additional-funds" basis through June 30, 1957. Under this NSF grant studies have been made of Ag_3Sb, Sb_2Te_3, FeS_2, and the Sn-Sb and In-Bi systems. One new superconductor, In_2Bi, was discovered (Tc = 5.6° K). These studies were made possible by the use of the hydrogen liquefier and helium cryostat built under the ONR contract. A paper covering this work is in preparation. Part of the results were reported at the National Science Foundation Conference on Low Temperature Physics and Chemistry, held at Baton Rouge, Louisiana, December 28-30 (1955).

C. Rare Earth Chemistry

When work was first begun under this contract the rare earth metals were not commercially available in high purity. Hence, from the beginning plans were made to prepare a number of the more common rare earth metals from pure compounds, which in turn were to be prepared from crude rare earth concentrates. In addition, methods of analysis of rare earth metals had to be developed, and methods for preparing lanthanum and cerium hydrides and nitrides were worked out.

During 1948-1950 much effort was directed toward the preparation of pure La_2O_3, Pr_6O_{11} and Nd_2O_3 using the newly developed ion exchange techniques for separation of the tripositive rare earth ions, and to the development and perfection of methods for the analysis of rare earth materials. Some of this work was carried on by graduate students in M. S. thesis research, although, in certain instances, the students were not actually employed on the contract while the thesis work was in progress.

These studies led to the development of a spectrochemical method for the analysis of lanthanum in the presence of praseodymium[22] and to a study of a number of the variables, such as effect of pH of eluent on the separation of La^{+++}, Nd^{+++} and Pr^{+++} using 5 per cent ammonium citrate solutions as the eluent, and Dowex-50 as the ion exchange resin[10]. The separation of samarium from rare earth mixtures using similar techniques was also investigated[23]. A brief report of some of this work has been published[28]. Some of the unpublished results have been of use to chemical engineers working at the Oak Ridge National Laboratory. These ion exchange studies made it possible for us to prepare significant amounts (25-100 gms.) of high purity La_2O_3, Nd_2O_3 and Pr_6O_{11} (99.9$^+$ per cent).

Experiments to prepare lanthanum metal by electrolysis were undertaken and a few grams of lanthanum was produced. However, while these studies were being carried out it became increasingly clear that Dr. F. H. Spedding and his group at the Institute for Atomic Research, Iowa State University, were making rapid progress in this field and by 1950 had prepared a number of the rare earth metals in large amounts and of high purity. Early in 1950 Dr. Spedding was kind enough to make available to us a small quantity of lanthanum metal and in 1951 some cerium praseodymium and neodymium metals as well. In view of this fact, no further attempts were made to produce the rare earth metals, and attention was directed to the making of lanthanum hydride and nitride[24].

D. Low Temperature Calorimetry

The availability of high purity rare earth oxides, notably La_2O_3, Pr_6O_{11} and Nd_2O_3, led to a proposal to determine the thermodynamic properties of these materials over a broad temperature range. Specific heat measurements of these oxides were to be made over the range 15°-320° K under the ONR contract. At the same time specific heat measurements were to be undertaken, sponsored by the School of Chemical Engineering, of the heat capacity of the oxides over the range 300-1200° K.

A high temperature calorimeter was constructed and measurements were made of the heat capacity of La_2O_3, Pr_6O_{11} and Nd_2O_3 over the range 30° C to 900° C[1]. The Pr_6O_{11} and Nd_2O_3 used were prepared as part of the rare earth chemistry studies described above. More recently similar measurements have been completed for CeO_2, but have not yet been published.

Unfortunately, the low temperature specific heat program has not been carried out with such success. This is due in part to delays in construction of the calorimeter, to the lack of graduate students having an interest in calorimetry and to some errors in judgment regarding the planning of this program.

An adiabatic calorimeter, modeled after one constructed by Scott, et al[19], was built as part of the research work carried out by Dr. H. A. McGee, Jr., for the Ph. D. degree in Chemical Engineering. The calorimeter is so constructed that either liquids or solids can be measured In return for Dr. McGee's willingness to help with the construction of the calorimeter he was allowed to use the calorimeter to carry out a thermodynamic study of the conversion of α-pinene to β-pinene. This study requires that heat capacity measurements be made of these organic compounds over the range 15°-315° K. The calorimeter performed very satisfactorily. A description of the calorimeter is included in Dr. McGee's Ph. D. thesis with appropriate acknowledgement to ONR[12]. The expenses of Dr. McGee's experimental studies were borne by the School of Chemical Engineering.

The decision to allow Dr. McGee to use the calorimeter for the pinene problem was based on several considerations, chief among them being that the pinene problem is of definite technological interest and was felt to be a more suitable research topic for a Ph. D. thesis in Chemical Engineering than the measurement of the specific heat of rare earth oxides. It was also hoped that Dr. McGee would be able to help with the rare earth measurements during and after completing his own measurements. This latter hope did not materialize since Dr. McGee left immediately after completing his measurements in January, 1955 to take a post-doctoral position at the University of Wisconsin.[*]

These remarks are presented as an explanation rather than a justification of the events and decisions made. It is regretted that no actual low temperature heat capacity data was obtained for the rare earth oxides. It is still hoped that these measurements can be completed at a later date.

Early in the planning of the calorimeter studies it was decided to construct several precision platinum resistance thermometers for use in these studies. These were to be calibrated against a precision platinum resistance thermometer purchased from the Leeds and Northrup Company and calibrated by the National Bureau of Standards over the range 10° K to 90° K and above the boiling point of oxygen on the International Temperature Scale. The NBS calibrated thermometer was to be reserved as a primary reference standard.

Four strain-free platinum resistance thermometers of the four-lead coiled-helix type, wound on mica crosses, as described by Meyers[18], were constructed. These thermometers have room temperature resistances of 31 to 35 ohms. After annealing at 500-600° C for several hours, the strain-free platinum coil, mounted on its mica cross was enclosed in a copper case, the leads being brought out through commercially available metal-to-glass terminals (Electrical Industries, Inc. Type AA-40T-S). These terminals consist of four small tubes (0.040 inch O. D.) sealed in a special glass

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[*] Dr. McGee is now employed in the low temperature group of the Ordnance Missile Laboratories, Redstone Arsenal, Huntsville, Alabama

of the Pyrex type. The finished thermometers were 9.5 mm in diameter and 80 mm long. The upper end of the thermometer was enlarged to form a section 12.5 mm long by 17.5 mm in diameter in which the four sealed terminals are located. The thermometers were filled with helium gas at room temperature and sealed off under one atmosphere pressure.

The thermometers were calibrated in a special cryostat somewhat similar to one described by Blue and Hicks[2]. This "thermometer comparator" consisted essentially of an 1100 gm copper block provided with holes for the four thermometers. This block was surrounded by an adiabatic shield in a high vacuum space. The temperature of the copper block could be controlled so that drift rates of the order of 0.001° per minute or less were achieved in much of the range from 10° to 320° K.

Three of these thermometers (the fourth developed a "short") were calibrated over the range 11° K to 320° K. A careful analysis of all the data taken showed that these thermometers did not possess the high degree of stability and accuracy desired of thermometers which were to be used in calorimetric experiments of the highest precision. The reproducibility varied from about 0.005° to about 0.025° C. The reasons for this variability are not known. Some of the variation may have arisen as a result of strains introduced into the annealed coils in the process of enclosing the coils in their copper cases. Stray voltages in the measuring circuits during calibration may also have been a contributing factor. Our experiences with these thermometers convinced us that the construction of precision resistance thermometers is no easy task.

The operating characteristics of the thermometer comparator cryostat suggested its possible use for measuring the thermal conductivity of metal rods over the range 11° to 320° K. Mr. W. H. Wright has explored this possible use of the cryostat by measuring the thermal conductivity of a free-machining yellow brass (62 Cu, 35 Zn, 3 Pb) and of cadimum over the range 80° to 300°K as his M. S. thesis in Chemical Engineering. The results which were obtained indicate that a cryostat of this design may be useful for the measurement of thermal conductivity over a wide temperature range where accuracy of 1-5 per cent is adequate.

Respectfully submitted:

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Paul K. Calaway, Director
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IV. APPENDIXES

A. Published Papers and Technical Reports

1. Published Papers

1. Ziegler, W. T., "The Superconductivity of Lanthanum and Cerium," J. Chem. Phys. 16, 838(1948).
2. Ziegler, W. T., "Georgia Tech Constructs New Low Temperature Laboratory," Refrig. Eng. 56, 402-4(1948).
3. Ziegler, W. T., "Separation of Rare Earth Elements, Particularly Praseodymium and Neodymium," Research Engineer (Georgia Institute of Technology) 7-10, 19-21, November, 1949.
4. Spicer, W. M., and Ziegler, W. T., "Spectrochemical Determination of Lanthanum in Praseodymium Metal," Anal. Chem. 21, 1422-3(1949).
5. Young, R. A., and Ziegler, W. T., "The Cryostat Structure of Lanthanu Nitride," J. Am. Chem. Soc. 74, 5251-3(1952).
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7. Ziegler, W. T., and Young, R. A., "Studies of Compounds for Superconductivity," Phys. Rev. 90, 115-19(1953).

2. Technical Reports

1. Ziegler, W. T., "Superconductivity of Lanthanum, Cerium, Prasoedymium and Neodymium," Tech. Rpt. No. 1, dated January 20, 1949.
2. Ziegler, W. T., Young, R. A., and Floyd, A. L., Jr., "Crystal Structure and Superconductivity of Lanthanum," Tech Rpt. No. 2, dated March 2, 1950.
3. Ziegler, W. T., and Young, R. A., "Studies of Compounds for Superconductivity," Tech. Rpt. No. 3, dated November 28, 1951.
4. Ziegler, W. T., and Young, R. A., "Superconductivity Studies," Tech. Rpt. No. 4, dated June 16, 1953.

B. Completed Theses Directly Related to ONR Project

1. Newell, C., Jr., "Construction and Testing of a Hydrogen Liquefier," M. S. Thesis in Chem. Engr., June, 1948.

2. Phillips, W. A., III, "The Erection of a Liquid Oxygen Plant and the Design of This Plant to Produce Liquid Nitrogen," M. S. Thesis in Chem. Engr., June, 1948.

3. Floyd, A. L., Jr., "Effect of Temperature on the Crystal Structure of Lanthanum, Cerium Neodymium and Praseodymium. The Relation between Crystal Structure and Superconductivity," M. S. Thesis in Physics, June, 1949.

4. Turner, M. A., "The Separation of Samarium from Rare Earth Mixtures by Means of Ion-Exchange," M. S. Thesis in Chem. Engr., June, 1949.

5. Young, R. A., "The Crystal Structure and Superconductivity of Lanthanum Nitride," M. S. Thesis in Physics, December, 1950.

C. Completed Theses Closely Related to ONR Project

1. Blomeke, J. O., "The Heat Content, Specific Heat and Entropy of La_2O_3, Pr_6O_{11} and Nd_2O_3 Between 30° and 900° C," Ph. D. Thesis in Chem. Engr., September, 1950.

2. Johnson, W. N. "The Separation of Neodymium and Praseodymium by Ion Exchange. I. Effect of Column Length, Loading and pH of Eluent," M. S. Thesis in Chem. Engr., June, 1950.

3. Lafond, W. F., "The Separation of Neodymium and Praseodymium by Ion Exchange. II. Effect of pH of Eluent on the Elution of Neodymium and Praseodymium," M. S. Thesis in Chem. Engr., September, 1950.

4. McGee, H. A., Jr., "The Heat Capacity Over the Range 15°-315° K., Heat of Fusion and Derived Thermodynamic Properties for Alpha - Beta Pinene," Ph. D. Thesis in Chem. Engr., June, 1955.

5. Roberts, J. T., Jr., "An Experimental Study of the Equilibrium Theory of Cation Exchange Chromatography," Ph. D. Thesis in Chem. Engr., October, 1951.

6. Wright, W. H., "Thermal Conductivity of Metals and Alloys at Low Temperatures. Part I. A Survey of Existing Data. Part II. The Thermal Conductivity of a Yellow Brass and of Cadmium," M. S. Thesis in Chem. Engr., (work completed in September, 1954, expected publication date about September, 1957).

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