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TEMPERATURE DEPENDENCE OF ELECTRICAL RESISTIVITY OF METALS

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TEMPERATURE DEPENDENCE OF ELECTRICAL RESISTIVITY OF METALS[1]

Lazarus Weiner, Premo Chiotti, and H. A. Wilhelm

I. ABSTRACT

The purpose of this investigation was to study the temperature dependence of electrical resistivity of thorium and titanium and to determine whether or not the slope of the resistance versus temperature curve of these metals exhibit anomalous discontinuities. Iron was also studied in an attempt to reproduce previously reported results on discontinuities in the slope of the resistance versus temperature curve for this metal.

The results of this investigation indicate that the best value for the resistivity of iodide titanium at 20°C is 49.6 microhm-centimeters, and is 167.5 microhm-centimeters at 850°C. The temperature coefficient of electrical resistance from 0° to 100°C was found to be 0.00397. The room temperature resistivity is somewhat higher than the values of 46.7 and 47.5 microhm-centimeters reported in the literature by Jaffee and Campbell and Van Arkel. The slightly higher results obtained in this investigation were probably due to contamination of the metal by minute amounts of oxygen and nitrogen.

The resistivity of electrolytic iron at 20°C was found to be 9.7 microhm-centimeters, and is 105.5 microhm-centimeters at 900°C. The Curie point was observed to be at 756°C, reproducing the result which Burgess and Kellberg obtained for electrolytic iron.

The resistivity of thorium containing 0.03% beryllium, 0.01% aluminum, 0.11% carbon, and 0.01% nitrogen was found to be 21.7 microhm-centimeters at 20°C. At 965°C, the resistivity of this metal is 64.1 microhm-centimeters. The temperature coefficient of electrical resistance from 0° to 100°C is 0.00277. The resistivity of thorium containing 0.06% beryllium, 0.01% aluminum, 0.04% carbon, and 0.02% nitrogen was found to be 20.4 microhm-centimeters at 900°C. The temperature coefficient of electrical

[1]This report is based on a MS thesis by Lazarus Weiner, submitted December, 1952.

resistance of this metal from 0° to 100°C was found to be 0.00333. The reason why at elevated temperatures the resistivities of the 0.11% carbon sample should be lower than those of the sample containing only 0.0445% carbon is uncertain.

In addition to inspection of resistance versus temperature plots, two rigorous methods were utilized to study the existence of discontinuities in slope of the resistance versus temperature curves of thorium, titanium, and iron. These methods entailed plotting $\frac{R_x}{R_{pt}}$ versus temperature and the application of the least squares method with the closeness of fit criteria to the data.

Visual examination of the resistance data for titanium when plotted on an enlarged scale indicated that the data could best be represented by segments of straight lines and that discontinuities in slope existed. The temperatures of these discontinuities were reproduced to within ± 20°C on separate runs. No obvious discontinuities were observed in the resistance versus temperature curve for iron. Some indication of such discontinuities was observed in the case of thorium.

The graphical method that involved practically simultaneous isothermal measurements of the electrical resistance of both platinum and the metal being studied offered a new approach to the problem. By taking small increments of temperature, $\frac{R_x}{R_{pt}}$ versus temperature curves were plotted. The $\frac{R_x}{R_{pt}}$ versus temperature curves in all cases were smooth curves, thereby giving no indication of the existence of anomalous discontinuities.

An analytical method, which employed the least squares method and the criteria for closeness of fit both to segments of straight lines represented by equations of the type $\frac{R}{R_o}$ = a + bt and to a smooth curve having as its assumed equation $\frac{R}{R_o} = 1.0000 + at + bt^2$, was applied to the resistance data for titanium and for thorium over the temperature ranges in which discontinuities in slope of resistance versus temperature curves were indicated by visual observations. In the case of thorium over the range 0° to 5?0°C, two straight line segments gave a better fit to the data than one smooth curve of the assumed form by a factor of 1.3. However, the precision index was such that this factor has little significance.

In the case of titanium over the range 0° to 516°C, two straight line segments gave a better fit to the data by a factor of 3.9. This factor is sufficiently large that it cannot be disregarded. However, addition of more terms to the equation for the smooth curve would probably reduce this factor.

The results obtained in this investigation in general do not show the existence of discontinuities in the slope of the resistance versus temperature curves of these metals over the temperature ranges studied, except possibly in the case of titanium. For titanium, visual observation of the plotted data and the analytical method indicate the existence of discontinuities, while the $\frac{R_{ti}}{R_{pt}}$ versus temperature curves gave no indication for their existence. Further, the addition of more terms to the equation for the smooth curve would probably reduce the significance of the results obtained by the analytical method.

Further studies on this problem should be conducted. A number of experimental difficulties encountered in this study must be taken into consideration in future investigations. The elimination of temperature gradients in the specimen and between the two specimens is of great importance. Besides using a furnace which at elevated temperatures has temperature gradients as small as possible, a metal sleeve, preferably of titanium or zirconium, should be placed around the specimens inside the furnace tube. Since these metals are both good conductors of heat as well as efficient getters, they would serve to reduce temperature gradients and to remove active residual gases. The use of a silver tube on the outside of the furnace tube would further help to eliminate temperature gradients. Also, the use of small diameter potential and current lead wires will minimize the heat that is conducted away from the specimens. Another major problem to contend with is that of the possible physical changes of the specimen during the run. Crystal growth and the precipitation of impurities, such as oxides and nitrides, in the grain boundaries may give rise to erroneous results. Although these changes are characteristic of the metal, fully annealing the specimens and maintaining a high vacuum will help minimize their effect. Another source of difficulty is the possible presence of metallic vapors due to volatile impurities in the specimens or due to the vapor pressure of the particular metal used. These vapors tend to condense on the current and potential lead-wire-insulators in the colder areas of the furnace and cause shorting between the wires, particularly if the insulating material is porous or at points where the wire extends through an otherwise impervious insulator. Such vapors would also tend to contaminate the platinum specimen. Also, all apparatus should be shielded to present any pickup of stray currents.

The precision in measuring $\frac{R_x}{R_{pt}}$ should be increased and more terms should be used in the equation for the smooth curve. Taking all these factors into consideration and utilizing the methods employed in this investigation, more conclusive evidence should then be available to determine the reality of anomalous discontinuities in the slope of the resistance versus temperature curves of metals.

II. INTRODUCTION

The electrical resistance of metals usually increases with an increase in temperature. At elevated temperatures, the temperature dependence of electrical resistance is generally linear or some function of the form $R_t = R_o(1 + \alpha t + \beta t^2)$. In this equation, R_t is the resistance of some temperature, t the temperature in degrees Centigrade, R_o the resistance of 0°C, and α and β are constants characteristic of the metal.

In the past twenty years, electrical resistance measurements have become a valuable means by which investigators have determined phase boundaries in their studies of phase diagrams. A detectable change in slope will generally occur in the resistance versus temperature curve upon crossing a phase boundary. If there is an increase in the volume upon crossing a boundary involving a nonisothermal transformation, an increase in the slope of this curve will generally be observed and conversely, a negative volume change will generally produce a decrease in the slope of the temperature dependence of electrical resistance. Resistance measurements have also proved very useful in the determination of allotropic and magnetic transformations of metals and alloys. Allotropic and isothermal transformations usually give a detectable discontinuity in the resistance versus temperature curve. The Curie point of some metals and alloys has been determined by electrical resistance measurements. In these cases, an abrupt change in slope of the resistance versus temperature curve has been observed at the Curie temperature. Other discontinuities in the slope of the temperature dependence of electrical resistance have been reported in a number of cases; however, these discontinuities cannot be explained on the basis of either phase boundaries or known transitions, either magnetic or allotropic.

The purpose of this investigation is to study the temperature dependence of electrical resistivity of thorium and titanium and to determine whether or not the slope of the resistance versus temperature curves of these metals exhibit anomalous discontinuities. Iron is also studied in an attempt to reproduce previously reported results on such discontinuities in the slope of the resistance versus temperature for this metal.

III. REVIEW OF LITERATURE

Bittel and Gerlach (1), Potter (2), and Nilsson (3) have shown that the resistance versus temperature curve for nickel has an abrupt change in slope at the Curie point. Likewise, Jaeger and his co-workers (4) and Pallister (5) have investigated the Curie temperature of high purity iron by means of electrical resistance measurements. In this case an abrupt change in slope was observed at the Curie point, but no discontinuity was observed at the beta-gamma transformation temperature. Maricq (6) and Jaeger and his co-workers (7) have utilized this method in the determination of the Curie point of cobalt. Many investigators have also determined the Curie temperature of alloys by this means (8, 9). Grube (10) and Vosskohler (11) have presented thorough surveys of the utility of electrical resistance measurements in the determination of transitions, both magnetic and allotropic, in metals and alloys as well as the determination of other phase boundaries in the study of alloy systems.

W. R. Ham and C. H. Samans (12) stated that anomalous discontinuities occur in the resistance versus temperature curves for all metals of Group VIII of the periodic table; however, their article contained no experimental data to support this statement. They obtained these discontinuities either by electrical resistance measurements on pure metal wires or by electrolytic conductivity measurements on glasses containing oxides of these metals. As of the present, Ham and Samans (13) have published data showing a series of discontinuities only for iron, nickel, and cobalt. These data were obtained by electrolytic conductivity measurements on glasses containing the oxides. By plotting log resistance versus temperature, a series of discontinuities for each was obtained. In their investigation of iron, they reproduced the series of discontinuities that had previously been reported by Ham and Post (14) and Ham and Rast (15) in their studies of the diffusion of hydrogen through pure iron. In this latter method, the plot of the log rate versus the reciprocal of the temperature was observed to contain discontinuities at a series of characteristic temperatures. These temperatures were related by a simple Ritz type equation of the general form $T_n = ct(1/n_o^2 - 1/n^2)$, where T_n is the absolute temperature at which a discontinuity occurs, ct a constant characteristic for each metal, n_0 a constant characteristic of the metal with n taking successive values of $(n_0 + 1)$, $(n_0 + 2)$, ... ,∞. The observed temperatures of the discontinuities in both methods agreed quite well with the theoretical temperatures obtained by the Ritz type equation. Series of anomalous discontinuities were also obtained for nickel and cobalt by the electrolytic conductivity method, and as with iron, the observed temperatures of each series were also related by the Ritz type equation. Ham and Samans reported that the allotropic transformations in iron did not enter into the series. However, with iron, nickel, and cobalt, they observed that the Curie point of each metal was one member of the individual series. They stated that the discontinuities are independent of impurities, concentration, and any form of chemical combination. Therefore, they postulated that these series of anomalous discontinuities are the result of minor electronic transitions. Ham and his co-workers have not as yet published any data obtained by electrical resistance measurements in which they also observed these discontinuities.

Chiotti (16), while conducting electrical resistance measurements on Ames thorium and thorium - carbon alloys, observed minor changes in slope of the temperature dependence of electrical resistance over the range 700° to 950°C. These minor changes could not be correlated with the phase diagram and their reality was somewhat doubtful.

Jaeger, Rosenbohm, and Fonteyne (17), by means of specific heat and electrical resistance measurements on ductile titanium, observed minor transitions in the specific heat versus temperature and resistance versus temperature curves. In both methods, they observed these minor transitions on heating and cooling. They suspected that these were due to impurities in the metal they used.

Reported electrical resistivity values and temperature coefficients of electrical resistance of thorium vary considerably. These variations are probably due to marked effects of impurities in the metal used. Reported values of the resistivity of thorium at [illegible]°C vary from 13 to 26 microhm - centimeters (18, 19, 20, 21, 22). Similarly, the variation in the temperature coefficient of electrical resistance of thorium ranges from 0.0023 degree^{-1} (19) to 0.0038 degree^{-1} (21). With titanium, as with thorium, the purity of the metal enters so markedly into its resistivity values that a large deviation among reported values exists. Investigators (23, 24, 25, 26, 27) using magnesium-reduced titanium have reported values of the resistivity at 20°C between 53.9 and 56.0 microhm - centimeters, although values as high as 78.6 and 82.0 microhm - centimeters have also been observed (28, 29) with the magnesium - reduced titanium. McQuillan (23) Van Arkel (30) and Jaffee and Campbell (33) working with iodide titanium, the purest form available, reported the values of 42.1, 47.5 and 46.6 microhm - centimeters respectively for the resistivity of titanium at 20°C.

IV. MATERIALS

A. Source

The thorium used in this investigation was experimental Ames thorium obtained in the shape of rods, a quarter inch in diameter and six to eight inches in length. Chemical analysis on one rod revealed it contained 0.03% beryllium, $<$ 0.01% aluminum, 0.11% carbon, and $<$ 0.01% nitrogen. A second rod contained 0.06% beryllium, $<$ 0.01% aluminum, 0.04% carbon, and 0.02% nitrogen.

Iodide titanium was obtained from the New Jersey Zinc Company in the form of rods, one sixteenth inch in diameter and ten inches in length. The purity of this metal was given as 99.99%. Spectrographic analysis revealed several impurities in the metal, iron appearing the strongest. Subsequent chemical analysis showed the metal contained 0.0013% iron.

The iron used in this investigation was electrolytic iron wire, 0.23 mm. in diameter and 99.8% purity.

B. Specimen Preparation

Both the thorium and titanium rods were cold swaged down to a wire of approximately 1.0 to 0.7 mm. diameter. The wires were pickled to remove any surface films. A five per cent HF solution was used to clean the titanium wires and a solution consisting of one part concentrated nitric acid, one part water, and a few drops of fluosilicic acid was used to clean the thorium wires. These wires as well as the iron wire were formed into coils with half the turns running clockwise and the other half running counterclockwise as shown in Figure 1.

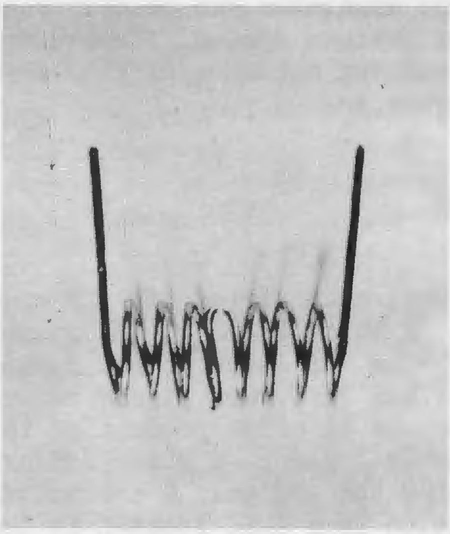

Figure 1. Specimen in the form of coil

Prior to any resistance measurements, the specimens were annealed in a high vacuum of from 10^{-5} to 10^{-6} mm. of mercury. The temperature at which the annealing was carried out was above the recrystallization temperature of the metal and the length of annealing time was sufficient to relieve all stresses that were introduced in the working of the metal.

V. APPARATUS AND METHODS

A. Apparatus for Resistance Measurements

The complete set-up employed in this investigation is shown in Figure 2. This apparatus can be discussed as three separate sections, namely: vacuum system, furnace and temperature measurement equipment, and the actual apparatus used in the electrical resistance measurements.

1. Vacuum system

The vacuum system consisted of a forepump, a diffusion pump, and a cold trap. A Welch Duo-Seal mechanical pump, capable of a vacuum of 1×10^{-4} mm. of mercury, was employed as the forepump. A glass condenser and trap were placed between the forepump and the diffusion pump as an added precaution to insure that no mercury reached the mechanical pump. A glass diffusion pump and a glass cold trap completed the system. A thermocouple gauge, National Research Corporation Type 501, located in the foreline connection of the diffusion pump, was used to indicate foreline pressure. A hot filament gauge, National Research Type 507, located in the high vacuum side between the diffusion pump and the furnace, gave the operating pressure. With this vacuum system, vacuums of between 1×10^{-5} and 1×10^{-6} mm. of mercury could be maintained even at elevated temperatures.

2. Furnace and temperature measurement apparatus

Initially, a horizontal resistance furnace and horizontal quartz tube were used. The tendency of the specimens to touch the sides of the quartz tube and react at elevated temperatures often caused some contamination of the specimens. Also, in the horizontal furnace there was a tendency for the specimens to sag and short against each other at elevated temperatures. A vertical furnace and quartz tube assembly was later employed and found to be satisfactory. All data in this investigation were obtained with this vertical arrangement.

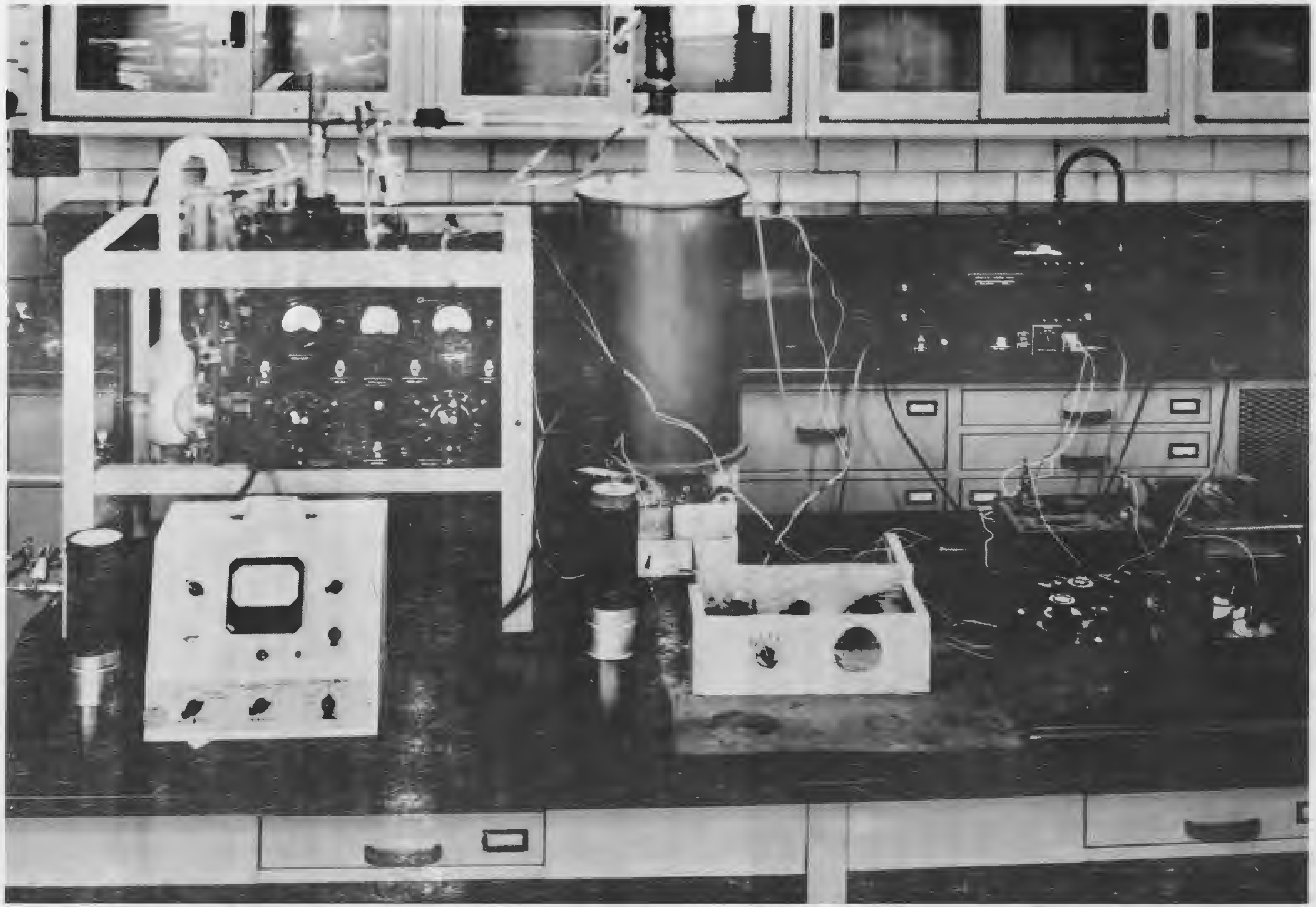

Figure 2. Apparatus for electrical resistance measurements.

Figure 3 shows the arrangement of the specimens within the furance. The vertical quartz tube, twenty-four inches in length and an inch and three-quarters in diameter, was connected to the vacuum system by means of a ground glass joint. All wires within the quartz were protected by alundum insulators. The wires emerged from the tube through fingers in the Pyrex glass cap that was connected to the top end of the quartz tube. This connection was effected by means of a ground joint between the glass cap and a glass taper sealed to the end of the quartz tube. Two circular copper spacers were used to keep the insulated wires in a rigid position and to prevent them from touching. Apiezon wax, vapor pressure about 10^{-6} mm. of mercury, was used to seal the wires in the glass fingers of the cap. Apiezon grease, vapor pressure about 10^{-6} mm. of mercury, was employed as lubricant for the ground glass joints.

The temperature gradient along the axis of the vertical furnace was measured by placing chromel-alumel thermocouples every inch, covering the middle seven inches of the furnace. At 900°C, the gradient was found to be one degree over the middle inch and one half of the furnace, the portion that the specimens occupy. Therefore, a temperature gradient of less than one degree existed between the specimens at temperatures around 800° to 900°C. At temperatures below 800°C, the temperature gradient between the specimens was negligible.

A chromel-alumel thermocouple which had been previously calibrated (31) against primary and secondary fixed points, was used to measure the temperature of the specimens. The thermocouple was placed next to, but not touching the specimens.

3. Electrical resistance measurement apparatus

The apparatus for electrical resistance measurements consisted of a potentiometer, an external galvanometer, and a current supply to provide current through the specimens.

A Rubicon high precision Type B potentiometer was used. This instrument has three ranges: 0.0 to 1.6 volts, 0.0 to 0.16 volts, and 0.0 to 0.016 volts. An Eppley standard cell and a six volt storage battery, as source of auxilary current, were used in conjunction with the potentiometer.

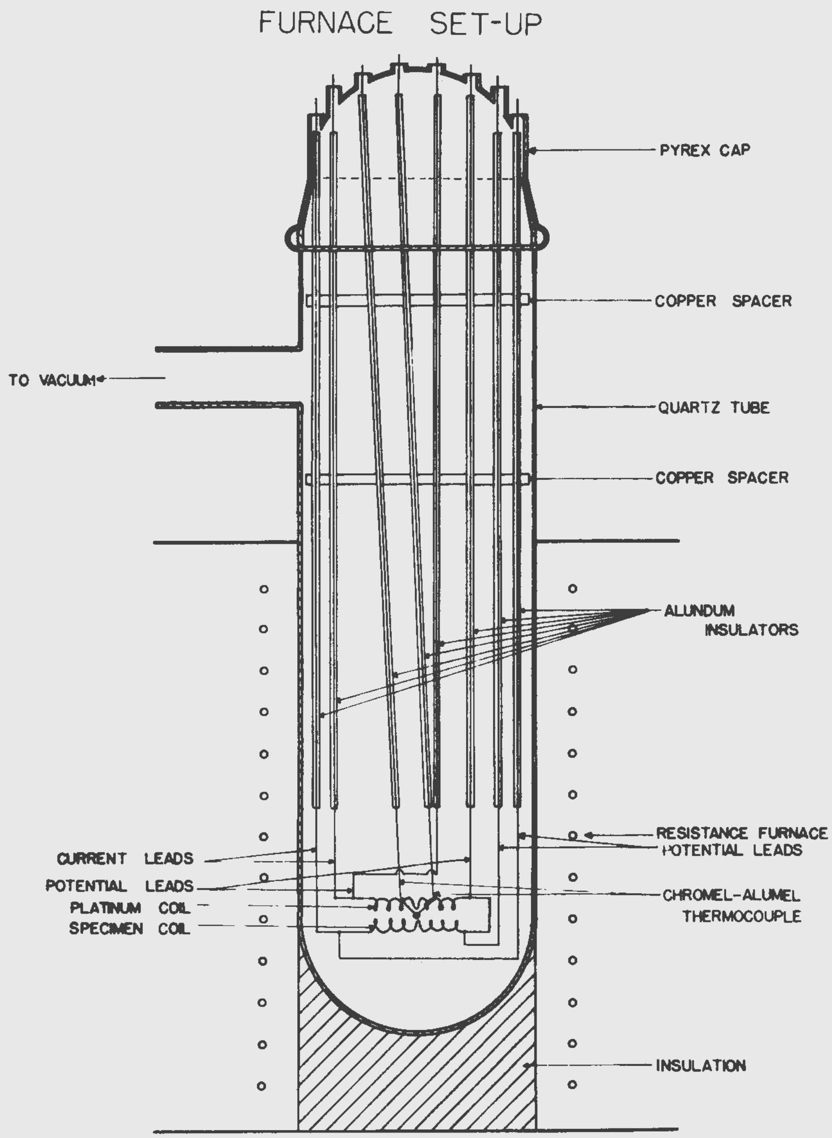

Figure 3. Furnace arrangement of the specimens.

A Rubicon D.C. Spotlight external galvanometer, with a sensitivity of 5.5 microvolts per millimeter, was employed.

A storage battery was used initially as the current source, but due to small fluctuations in its current, it was replaced with a regulated current supply. From this regulated current supply, a constant D.C. current of approximately either 50, 100, 150, or 200 milliamperes could be obtained. Over a period of twenty-eight hours, the maximum variation in current was found to be 0.06 milliamperes. The ripple of the regulated potential was 2 millivolts out of 90 volts D.C.

B. Electrical Resistance Measurements

The potentiometric method of measuring electrical resistance was employed. By sending a known amount of current through the specimen and measuring the potential drop across it, the resistance of the specimen can be obtained. In this investigation two specimens, the metal being investigated and platinum, were connected in series. An external standard resistor was placed in series with the specimens. By measuring the potential drop across it and the potential drop across each of the specimens, the resistance of the specimens (R_x and R_{pt}) can be calculated as follows.

$$R_x = \frac{\text{(potential drop across the specimen)}}{\text{(potential drop across the standard resistor)}} \times \text{(resistance of the standard resistor).}$$

Similarly for the platinum

$$R_{pt} = \frac{\text{(potential drop across the platinum coil)}}{\text{(potential drop across the standard resistor)}} \times \text{(resistance of the standard resistor).}$$

Knowing the resistance and the original dimensions of the specimens, the resistivity of the specimens can be calculated from the equation $\rho = RA/L$ where ρ is the resistivity, R is the resistance as calculated above, A the cross sectional area of the specimen, and L its length.

Figure 4 shows the schematic circuit diagram of the potentiometric method used in this investigation. Titanium wire served for the current leads. The potential leads of the specimens were of the same material as the specimens themselves. In this manner, no thermal emf's were developed by junctions of dissimilar metals at two different temperatures and there was no reaction between the potential leads and the specimens. The standard resistor was manganin wire whose resistance was 0.10350 ohms as measured by a Kelvin Bridge. The rotary switch made it possible to measure the potential drops across the specimen and the platinum coil within ten seconds. Therefore, any change in temperature between these two measurements was negligible and essentially the measurements were made simultaneously.

Heating and cooling rates employed ranged from 25 to 35 centigrade degrees per hour. Temperature measurements were made immediately before and immediately after the potential measurements of the specimens. Since the change in the temperature between the two temperature measurements was never greater than one degree, a mean was taken as the true temperature at the time of the potential measurements of the specimens.

Precaution had to be exercised to prevent heating the specimens by passing too large a current through them. It was found that up to 150 milliamperes was well within the limit of caution, and for most runs, 100 milliamperes were used.

From the temperature, resistance, and resistivity values, resistance versus temperature and resistivity versus temperature curves were plotted.

C. Determination of Discontinuities

Two methods can be employed to determine the existence of anomalous discontinuities in the slope of the resistance versus temperature curves. One method deals directly with the data obtained and is essentially a graphical method, while the second method is an analytical analysis applied to the observed data.

1. Graphical method

The most apparent direct method is to plot the resistance versus temperature curve for a particular specimen and by visual observation note the existence of discontinuities in the slope.

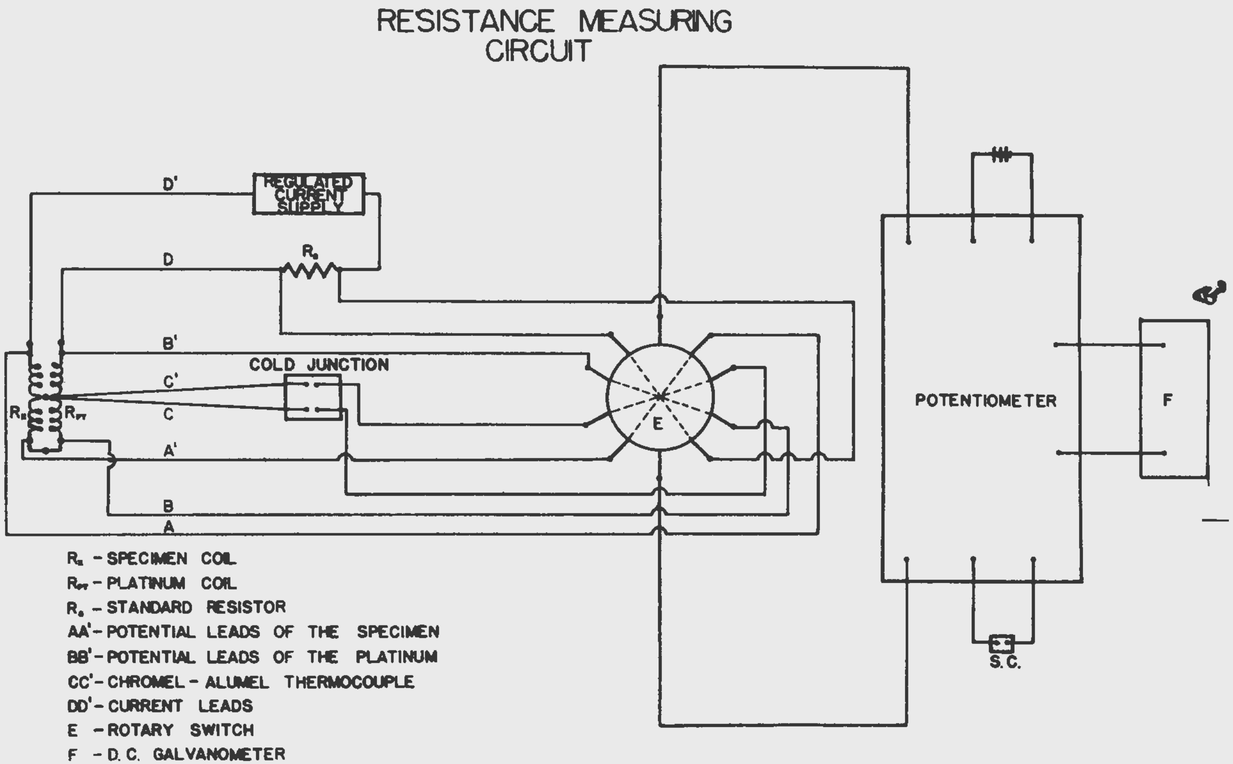

Figure 4. Schematic circuit diagram of the potentiometric method of measuring electrical resistance used in this investigation.

This method, however, is subject to several limitations. First, the reported discontinuities are not pronounced; that is, the changes in slope are small. Secondly, the International temperature scale from 0°C to 660°C is based on the electrical resistance of platinum, whose resistance has been measured at the ice point, the steam point, and the sulfur point (31, p. 21). Intermediate values of temperature are extrapolated from the resistance versus temperature curve of platinum for which a relation of the type $R_t = R_o (1 + At + Bt^2)$ is assumed. Therefore, the existence of a discontinuity for a certain specimen, assuming that such discontinuities also exist for platinum as stated by Ham and that its resistance versus temperature curve can be represented by segments of straight lines, will be masked or made less pronounced. Thus, in order to reveal the existence of these discontinuities, a method independent of temperature is desireable.

Over a temperature range in which the slope of the temperature dependence of electrical resistance of a metal is linear, the resistance versus temperature curve can be represented by the equation $R = R_o(1 + \alpha t)$ where $R_o\alpha$ is the slope of the straight line. Similarly, over a temperature range in which the resistance of platinum increases linearly with increasing temperature, its resistance versus temperature curve can be represented as $R' = R_o' (1 + \alpha' t)$. Dividing these two equations gives the ratio $\frac{R}{R'} = \frac{R_o + R_o\alpha t}{R_o' + R_o'\alpha' t}$. If increments of resistance of the metal and platinum are measured between the same two temperatures over a range in which both functions are linear, the ratio, $\frac{R}{R'}$, will equal a constant.

$$\frac{R_{t_2} - R_{t_1}}{R'_{t_2} - R'_{t_1}} = \frac{R_o + R_o\alpha t_2 - R_o - R_o\alpha t_1}{R_o' + R_o'\alpha' t_2 - R_o' - R_o'\alpha' t_1}$$

$$\frac{\Delta R}{\Delta R'} = \frac{R_o \alpha (t_2 - t_1)}{R_o'\alpha'(t_2 - t_1)}$$

$$\frac{\Delta R}{\Delta R'} = \frac{R_o \alpha}{R_o' \alpha'} = \text{a constant.}$$

Whenever the slope of the resistance versus temperature curve of either the metal or platinum changes, the ratio, $\frac{\Delta R}{\Delta R'}$, will also change.

Therefore, by plotting the $\frac{\Delta R}{\Delta R'}$ versus temperature, a method, independent of temperature other than to indicate the approximate temperatures of the discontinuities, can be utilized to study the reality of these anomalous discontinuities.

If the resistance versus temperature curve for a metal consists of segments of straight lines rather than a smooth curve, the $\frac{\Delta R}{\Delta R'}$ versus temperature curve will be segments of straight lines of zero slope with discontinuities between the segments, corresponding to the discontinuities in the slope of the temperature dependence of electrical resistance of both the metal and platinum. Assuming that the slope of the resistance versus temperature curves for both the platinum and the specimen decreases discontinuously at a number of points along each curve, then the theoretical $\frac{\Delta R}{\Delta R'}$ versus temperature curve would appear as is shown in Figure 5. The upward steps correspond to changes in slope for platinum and the downward steps for changes in slope for the specimen. There may of course be a simultaneous change in slope for both platinum and the specimen in which case the change in $\frac{\Delta R}{\Delta R'}$ may be zero, positive or negative.

If the resistance versus temperature curve did not contain any discontinuities in its slope, and therefore was a smooth curve, the $\frac{\Delta R}{\Delta R'}$ versus temperature curve will also be a smooth curve as the slopes of the resistance versus temperature curves of the metal and platinum would be constantly varying.

A third possibility arises if the resistance versus temperature curve consists of segments of smooth curves of the type $R = R_o (1 + \alpha t + \beta t^2)$. In this case the $\frac{\Delta R}{\Delta R'}$ versus temperature curve will be segments of hyperbolas with discontinuities between them corresponding to the discontinuities in the slope of the resistance versus temperature curves of the metal and platinum. This is developed as follows:

$$R = R_o (1 + \alpha t + \beta t^2).$$

$$R' = R'_o (1 + \alpha' t - \beta' t^2).$$

Dividing these two equations

$$\frac{R}{R'} = \frac{R_o + R_o \alpha t + R_o \beta t^2}{R'_o + R'_o \alpha' t + R'_o \beta' t^2} .$$

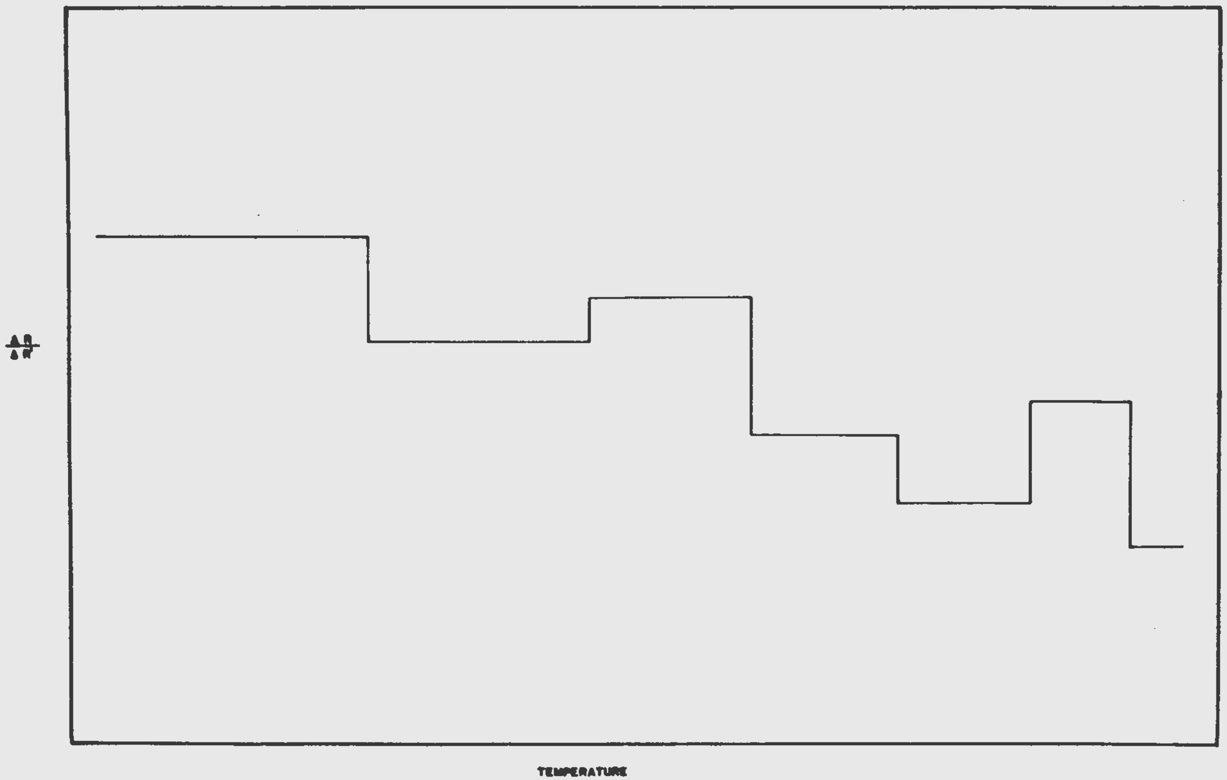

Figure 5. Theroetical $\frac{\Delta R}{\Delta R'}$ versus temperature curve, in the case of resistance versus temperature curves, consisted of segments of straight lines.

Taking increments of resistances of the metal and platinum between the same two temperatures results in the following equation:

$$\frac{\Delta R}{\Delta R'} = \frac{R_o + R_o\alpha t_2 + R_o\beta t_2^2 - R_o - R_o\alpha t_1 - R_o\beta t_1^2}{R_o' + R_o'\alpha' t_2 + R_o'\beta' t_2^2 - R_o' - R_o'\alpha' t_1 - R_o'\beta' t_1^2}$$

$$\frac{\Delta R}{\Delta R'} = \frac{R_o\alpha(t_2 - t_1) + R_o\beta(t_2^2 - t_1^2)}{R_o'\alpha'(t_2 - t_1) + R_o'\beta'(t_2^2 - t_1^2)}.$$

$$\frac{\Delta R}{\Delta R'} = \frac{R_o\alpha + R_o\beta(t_2 + t_1)}{R_o'\alpha' + R_o'\beta'(t_2 + t_1)}.$$

The above equation is of the type: $y = \frac{a + bx}{c + dx}$ or $yc + dyx - bx = a$. The equation for a family of equilateral hyperbolas is: $x'y' = k$. Thus by letting $x' = my + n$ and $y' = px + q$, and substituting these values in the equation of a hyperbola gives:

$$pmyx + mqy + npx + nq = k.$$

This equation is equal to the equation $yc + dyx - bx = a$ with $pm = d$, $mq = c$, $np = -b$, and $k - nq = a$.

Thus it is seen that the equation $\frac{\Delta R}{\Delta R'} = \frac{R_o\alpha + R_o\beta(t_2 + t_1)}{R_o'\alpha' + R_o'\beta'(t_2 + t_1)}$ is an equation of a family of hyperbolas, a different curve arising with a change in either α, β, α' or β'.

In carrying out this method, every precaution must be entertained in order to obtain the most accurate data possible so that the scatter in the values of $\frac{\Delta R}{\Delta R'}$ will be a minimum. Particular attention must be focused to insure that no temperature gradients exist between the specimen coil and the platinum coil.

2. Analytical Method

In those cases in which apparent discontinuities in the slope of the resistance versus temperature curve occur, an analytical method can be utilized to determine whether a smooth curve or straight line segments is the best fit to the data. This can be accomplished by applying the least squares method (32) and the criteria for closeness of fit (32, p.260). The least squares method gives the most probable values for the constants entering the equation of an assumed form, but it does not indicate the best equation for the representation of the given data. Which one of these assumed equations, a smooth curve or straight line segments, gives the best fit to the data can then be determined by applying the criteria for closeness of fit.

Over a temperature range in which the resistance versus temperature curve is a curve of the type $\frac{R}{R_o} = 1 + \alpha t + \beta t^2$, the arbitrary constants α and β can be evaluated by the least squares method. The above equation is of the type $y = 1 + ax + bx^2$. Assuming only y liable to error and that all values were obtained with the same precision, the equations to evaluate a and b are readily developed.

In order to minimize the sum of the squares of the deviations between the observed and calculated values, $\sum (y_o - y)^2$, the sum of the squares of the deviations is differentiated with respect to each constant and set equal to zero. In this relation, $\sum (y_o - y)^2$, y_o is the observed value and y the value defined by the assumed equation. Substituting $y = 1 + ax + bx^2$ in $\sum (y_o - y)^2$ gives:

$$\sum (y_o - 1 - ax - bx^2)^2.$$

$$\frac{\partial \sum}{\partial a} (y_o - 1 - ax - bx^2)^2 = 0 \text{ or } \frac{\sum \partial}{a} (y_o - 1 - ax - bx^2)^2 = 0.$$

$$\frac{\partial \sum}{\partial b} (y_o - 1 - ax - bx^2)^2 = 0 \text{ or } \frac{\sum \partial}{\partial b} (y_o - 1 - ax - bx^2)^2 = 0.$$

Differentiation results in the following equations:

$$\sum xy_o = \sum x + a\sum x^2 + b\sum x^3.$$

$$\sum x^2y_o = \sum x^2 + a\sum x^3 + b\sum x^4 .$$

Solving for a and b gives:

$$a = \frac{\sum x^4 (\sum xy_o - \sum x) - \sum x^3 (\sum x^2y_o - \sum x^2).}{\sum x^2 \sum x^4 - (\sum x^3)^2}$$

$$b = \frac{\Sigma x^2 (\Sigma x^2 y_o - \Sigma x^2) - \Sigma x^3 (\Sigma x y_o - \Sigma x)}{\Sigma x^2 \Sigma x^4 - (\Sigma x^3)^2}.$$

In the case of a straight line function of the type $\frac{R}{R_o} = 1 + \alpha t$, or $y = 1 + ax$, with y only liable to error and all values obtained with the same precision, the most probable value for a can be obtained as follows:

Substituting the value of y in the sum of the squares of the deviations results in

$$\Sigma (y_o - 1 - ax)^2.$$

Differentiating this equation with respect to a and setting it equal to zero gives:

$$\Sigma x y_o = \Sigma x + a \Sigma x^2.$$

Thus the most probable value of a is obtained by the relation

$$a = \frac{\Sigma x y_o - \Sigma x}{\Sigma x^2}$$

The sum of the squares of the deviations between the observed values and calculated values, $\Sigma (y_o - y)^2$, can now be obtained for both a smooth curve and straight line segments. From this summation in each case, the relation for closeness of fit, $\Omega = \frac{\Sigma (y_o - y)^2}{n - m}$ where n is the number of observed values and m the total number of constants in the assumed equation, can be applied to determine whether a smooth curve or segments of straight lines gives the best fit to the experimental data. The equation which gives the smallest value of Ω is considered as the better fit of the data.

VI. EXPERIMENTAL RESULTS

A. Resistivity Values

1. Titanium

Electrical resistance measurements were carried out on three specimens of iodide titanium. The diameters of the specimens were obtained from micrometer measurements and for specimen 3, the diameter was also determined from the weight of a known length of the specimen using as the density of titanium the value 4.54. From the original dimensions of the specimens, the resistivity was calculated for each temperature.

The amount of current passed through the specimens was approximately 100 milliamperes. An average heating or cooling rate of from thirty to thirty-five degrees per hour was employed in all cases and a vacuum of between 3.0×10^{-5} and 3.0×10^{-6} mm. of mercury was maintained.

Tables 1, 2, 3 and 4 give the resistivity values for specimen 1 on heating, specimen 1 on cooling, specimen 2 on heating, and specimen 3 on heating respectively. The temperature dependence of electrical resistivity for the three titanium specimens is shown in Figure 6. The precision of the resistivity values was found to be ±0.4%.

The resistivity values for specimen 1 can be considered the best, since it had the lowest value of resistivity at room temperature, 49.6 microhm-centimeters at 20°C, and its temperature coefficient of electrical resistance, α, had the highest value among the three specimens. From 0° to 100°C, for specimen 1 had the value 0.00397. In all cases, α was obtained from the relation $\alpha = (\rho_{100} - \rho_0)/100\rho_0$. Since α is a measure of purity, that is, the higher the value of α the greater the purity of the sample, it may be concluded that specimen 1 had the least amount of impurities. The resistivity versus temperature curve for specimen 2 is very similar to that for specimen 1, except it is displaced to higher values of resistivity. The resistivity for specimen 2 at 20°C was 51.4 microhm-centimeters. The value for α in this case was 0.00384 which agrees quite well with that for specimen 1. For specimen 3 the resistivity at 20°C was 57.2 microhm-centimeters, much higher than the corresponding value for specimen 1. The value of α for specimen 3 was 0.00344, much lower than the α for specimen 1. These results indicate that specimen 3 contained impurities. These impurities might have been introduced in swaging or as a result of contamination by residual gases, oxygen and nitrogen, at elevated temperatures. Also, as is seen in Figure 6, the resistivity curve for specimen 3 rapidly approaches the curve for specimen 1 at elevated temperatures. This indicates that the effect of contamination on the value of the resistivity of the of the metal is less pronounced as the temperature is increased up to about 840°C.

Table I

Resistivity Values of Titanium Specimen 1[a] on Heating

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
24	50.7	404	118.0
37	52.9	419	120.3
53	55.6	431	122.3
66	58.0	445	124.5
78	60.2	460	126.8
94	63.1	479	129.7
106	65.1	497	132.2
120	67.8	513	134.4
134	70.3	541	138.3
147	72.6	557	140.3
159	74.9	574	142.4
176	78.0	594	144.9
189	80.5	609	146.8
221	86.3	630	149.1
233	88.4	646	150.9
247	91.1	669	153.3
258	92.9	683	154.6
270	95.0	702	156.4
287	98.2	720	158.2
301	100.6	739	159.7
323	104.5	753	160.9
339	107.3	770	162.2
353	109.5	794	164.0
364	111.4	812	165.2
374	113.0	830	166.3
392	115.9	843	167.2

[a]Dimensions of the specimen were 0.114 cm. in diameter and 33.536 cm. in length. The specimen was annealed for twelve hours at a temperature between 650° and 700°C.

TABLE II

Resistivity Values of Titanium Specimen 1[a] on Cooling

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
30	51.7	451	125.5
40	53.7	466	127.8
58	56.6	478	129.5
70	58.9	498	132.3
97	63.7	514	134.6
113	66.5	531	136.9
128	69.2	544	138.8
153	73.8	557	140.5
169	76.7	567	142.0
201	82.7	581	143.5
215	85.4	644	150.7
231	88.1	652	151.5
246	91.3	668	153.2
263	94.1	686	154.9
282	97.3	704	156.6
304	101.1	720	158.0
318	103.5	739	159.7
331	105.7	751	160.6
344	108.2	775	162.4
366	111.8	786	163.3
379	114.0	799	164.2
392	116.1	813	165.0
406	118.5	825	165.8
420	120.7	837	166.4
434	122.9	848	166.5

[a]Dimensions of the specimen were 0.114 cm. in diameter and 33.536 cm. in length. The specimen was annealed for twelve hours at a temperature between 650° and 700°C.

TABLE III

Resistivity Values of Titanium Specimen 2[a] on Heating

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
27	52.8	430	123.7
44	56.1	456	128.1
61	58.9	475	130.7
76	62.2	495	133.9
95	64.9	521	137.4
109	67.7	535	139.3
128	71.1	555	141.9
146	74.2	569	143.6
163	77.4	591	146.1
181	80.5	606	148.0
199	83.8	628	150.6
217	87.1	641	152.0
233	90.0	658	153.7
247	92.6	672	155.2
266	96.0	693	157.2
287	99.7	719	159.5
311	104.2	743	161.5
328	107.3	755	163.2
343	109.8	772	164.3
360	112.6	800	165.5
387	116.9	836	167.8
413	121.2		

[a]Dimensions of the specimen were 0.114 cm. in diameter and 33.500 cm. in length. The specimen was annealed for ten hours at a temperature between 675° and 700°C.

TABLE IV

Resistivity Values of Titanium Specimen 3[a] on Heating

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
32	59.5	471	134.1
47	62.6	486	136.2
58	64.8	500	138.1
69	66.9	517	140.2
98	72.0	532	141.9
123	76.9	549	144.0
138	79.7	565	145.7
158	83.2	584	147.8
173	86.0	598	149.3
192	89.4	613	150.8
214	93.4	629	152.4
233	96.7	645	153.9
255	100.5	667	155.9
271	103.3	686	157.4
287	106.0	698	158.4
301	108.4	712	159.4
317	111.0	734	160.9
336	114.2	751	162.1
355	117.2	768	163.2
372	119.8	788	164.5
393	122.9	806	165.5
404	124.7	828	166.7
423	127.5	837	167.3
458	132.3		

[a]Dimensions of the specimen were 0.0840 cm. in diameter and 19.953 cm. in length. The specimen was annealed for eight hours at a temperature between 700° and 725°C.

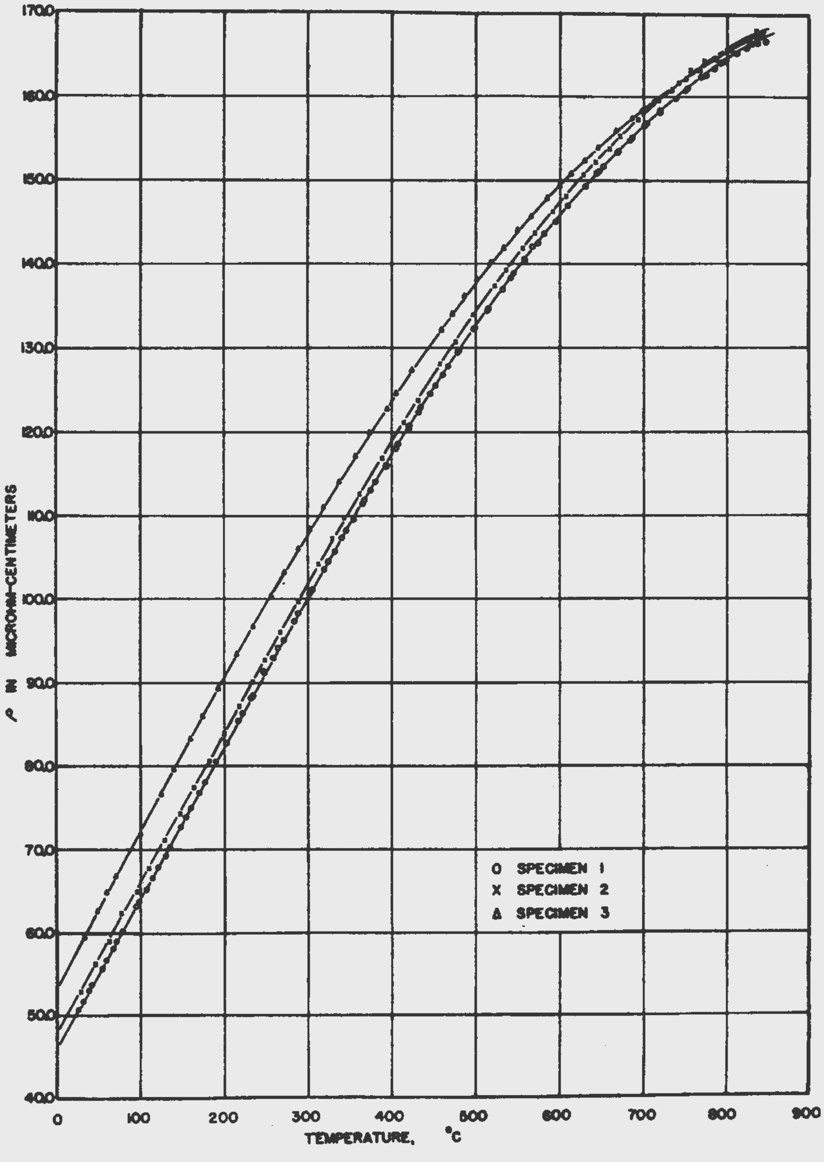

Figure 6. Temperature dependence of electrical resistivity of iodide titanium.

McQuillan (23) obtained for the resistivity of iodide titanium at 20°C the value of 42.1 microhm-centimeters. At this same temperature, Jaffee and Campbell (33) report a value of 46.7 microhm-centimeters for iodide titanium. Van Arkel (30) gives the resistivity of iodide titanium at 20°C as 47.5 microhm-centimeters. The best value obtained in this investigation was 49.6 microhm-centimeters at 20°C for iodide titanium. McQuillan used specimens 0.008 inches thick by 1/4 inch wide by about 2 1/2 inches long. The probable error in determining the dimensions of the specimen may, in part at least, account for the fact that his value is much lower than those obtained by Van Arkel and Jaffee and Campbell. The deviations between this value and those of Jaffee and Campbell and Van Arkel may have been caused by contamination of the specimen by impurities such as oxygen and nitrogen. According to Jaffee and Campbell (33), an addition of approximately 0.10% by weight of either oxygen, nitrogen or a combination of both will increase the resistivity of iodide titanium from 47.7 to 50.7 microhm-centimeters at 25.6°C. The lowest resistivity value obtained in this investigation at 25°C was 50.7 microhm-centimeters. On this basis, it may be concluded that the metal used in this investigation probably became contaminated by oxygen or nitrogen, thereby giving rise to deviations between the resistivity values obtained here and those previously reported.

2. Iron

A specimen of electrolytic iron wire, 0.0230 cm. in diameter and 16.391 cm. in length, was annealed for four hours at a temperature between 600° and 700°C before any resistance measurements were carried out. A heating and cooling run was conducted under a vacuum of between 4.0×10^{-5} and 5.6×10^{-7} mm. of mercury. A current of approximately 50 milliamperes was passed through the specimen. The heating and cooling rates were approximately thirty-four degrees per hour.

Tables 5 and 6 give the resistivity values obtained. These values are plotted in Figure 7. By means of extrapolation, the Curie point was found to be at 756°C. This value agrees very well with the value of 757°C obtained by Burgess and Kellberg (34). The precision of the resistivity values was found to be ±0.3%.

Comparing the resistivity values for electrolytic iron obtained in this investigation with reported values which are considered the most reliable, namely, Burgess and Kellberg and Ribbeck (34, p.184), one finds agreement up to 300°C. Above this temperature to the Curie point, the resistivity values obtained from this research are higher than those of Burgess and Kellberg and Ribbeck, by as much as 6%.

TABLE V

Resistivity Values of Iron on Heating

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
30	10.3	480	54.2
45	11.3	500	56.9
59	12.0	526	60.8
79	13.3	542	63.0
106	15.1	555	65.1
128	16.6	575	68.0
139	17.5	597	71.5
153	18.6	617	74.8
172	20.0	642	79.2
183	21.1	666	83.5
206	23.0	679	86.0
222	24.4	696	89.8
258	27.9	717	94.8
269	29.0	732	97.0
309	33.2	746	98.3
325	35.0	765	99.9
342	36.8	782	101.0
361	39.1	796	101.8
378	41.1	824	102.8
393	43.0	837	103.4
409	44.9	855	104.0
422	46.6	872	104.5
442	49.1	894	105.2
464	51.7		

TABLE VI

Resistivity Values of Iron on Cooling

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
28	10.2	478	53.3
41	10.8	492	55.3
55	11.7	508	57.6
80	13.2	524	59.8
94	14.2	568	66.2
116	15.7	586	68.9
132	16.9	601	71.4
151	18.3	620	74.4
169	19.8	639	77.1
191	21.6	662	82.3
228	24.8	681	86.2
246	26.5	700	90.1
264	28.3	713	93.0
284	30.4	730	96.1
304	32.4	752	98.3
325	34.6	773	99.9
344	36.8	793	101.2
362	38.9	813	102.2
382	41.2	829	102.9
404	43.8	848	103.6
424	46.3	866	104.2
443	48.6	887	105.0
462	51.1		

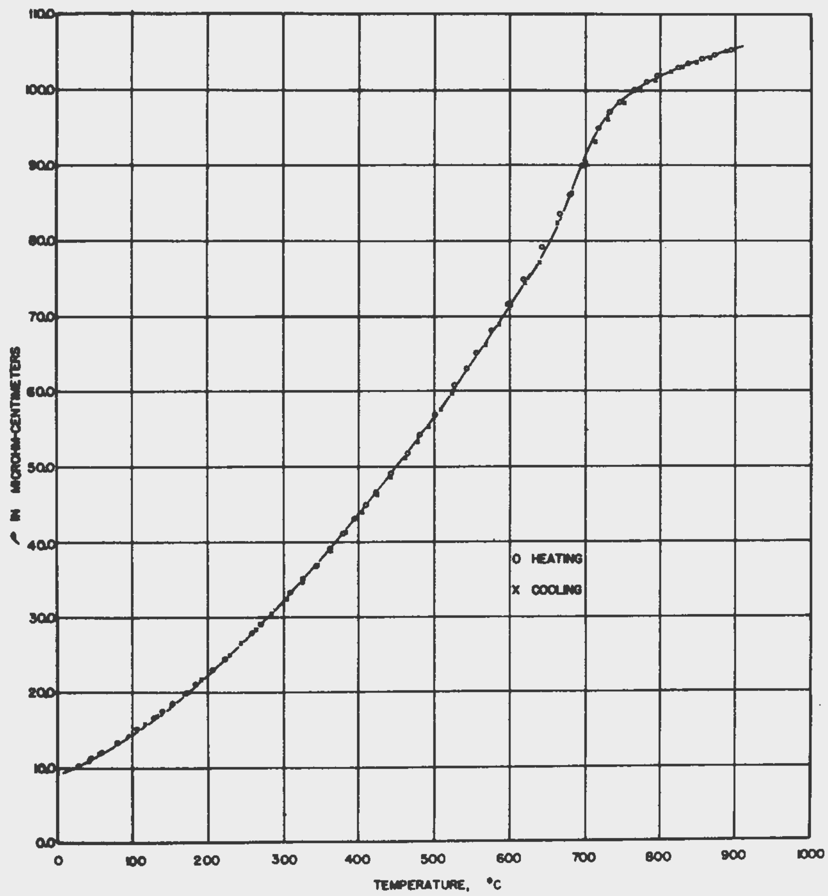

Figure 7. Temperature dependence of electrical resistivity of electrolytic iron.

3. Thorium

Electrical resistance measurements were carried out on four specimens of Ames experimental thorium. Specimens 1 and 2 were of metal containing 0.03% beryllium, $<$ 0.01% aluminum, 0.11% carbon, and $<$ 0.01% nitrogen. Specimens 3 and 4 were of metal containing 0.06% beryllium, $<$ 0.01% aluminum, 0.04% carbon, and 0.02% nitrogen. The diameters of the specimens were determined both by micrometer measurements and by weighing a known length of the specimen. The density used in calculating the diameters was 11.72, the theoretical value. Both these methods gave diameter values that agreed quite well. From the original dimensions of the specimens, the resistivity was calculated for each temperature.

For all specimens, a current of approximately 100 milliamperes was used. An average heating or cooling rate of between thirty and thirty-five degrees per hour was employed in all cases and a vacuum of between 1.8×10^{-5} and 3.8×10^{-6} mm. of mercury was maintained during the runs on specimens 1, 3 and 4. A vacuum of only 5.0×10^{-5} mm. of mercury was maintained in the case of specimen 2.

Tables 7, 8, 9, 10 and 11 give the resistivity values for specimen 1 on heating, specimen 1 on cooling, specimen 2 on heating, specimen 3 on heating, and specimen 4 on heating respectively. Figure 8 shows the temperature dependence of electrical resistivity for specimens 1 and 2, while Figure 9 gives that for specimens 3 and 4. The precision of the resistivity values was found to be $\pm$0.4%.

Resistivity values for specimen 2 are higher than the corresponding ones for specimen 1 from room temperature to approximately 350°C. Above this temperature, the values for specimen 2 are approximately the same as those of specimen 1. The cause of this behavior is not known. The resistivity for the thorium containing 0.11% carbon (specimen 1) was found to be 21.7 microhm-centimeters at 20°C. The temperature coefficient of electrical resistance for this sample over the range 0° to 100°C was 0.00277.

Resistivity values for specimens 3 and 4, containing 0.0445% carbon, fall on one curve as is seen in Figure 9. The resistivity of 20°C was 20.4 microhm-centimeters, while the temperature coefficient of electrical resistance over the range 0° to 100°C was found to be 0.00333.

TABLE VII

Resistivity Values of Thorium Specimen 1[a] on Heating

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
31	22.4	404	42.5
50	23.5	422	43.4
66	24.4	445	44.4
82	25.3	468	45.5
100	26.3	483	46.2
119	27.4	499	47.0
137	28.4	517	47.8
158	29.5	532	48.5
173	30.3	552	49.5
188	31.1	569	50.2
219	32.7	586	50.9
235	33.7	602	51.6
252	34.6	619	52.4
267	35.4	634	53.1
299	37.1	652	53.8
323	38.3	668	54.5
338	39.2	686	55.3
356	40.1	700	55.8
374	41.0	718	56.5
387	41.7	735	57.2

[a]Dimensions of the specimen were 0.0921 cm. in diameter and 33.993 cm. in length. The specimen was annealed for two and one-half hours at a temperature between 800° and 900°C.

TABLE VIII

Resistivity Values of Thorium Specimen 1[a] on Cooling

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
30	22.3	540	48.9
58	23.9	559	49.7
72	24.5	575	50.5
91	25.6	589	51.1
106	26.4	604	51.7
121	27.2	629	52.8
146	28.6	645	53.4
160	29.4	661	54.1
176	30.3	677	54.7
192	31.1	692	55.4
211	32.2	708	56.0
228	33.1	726	56.7
314	37.7	773	58.2
330	38.6	786	58.7
345	39.4	804	59.3
368	40.5	821	59.9
388	41.5	839	60.5
405	42.3	858	61.2
422	43.2	880	61.9
442	44.2	899	62.4
458	45.0	917	63.0
474	45.8	935	63.5
502	47.1	953	63.9
520	48.0	965	64.1

[a]Dimensions of the specimen were 0.0921 cm. in diameter and 33.993 cm. in length. The specimen was annealed for two and one-half hours at a temperature between 800° and 900°C.

TABLE IX

Resistivity Values of Thorium Specimen 2[a] on Heating

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
28	22.8	473	45.5
46	24.0	492	46.3
65	25.0	525	47.6
83	26.0	543	48.5
102	27.0	560	49.3
120	28.1	579	50.0
140	29.1	598	50.9
161	30.2	616	51.7
180	31.2	634	52.5
200	32.4	676	54.2
219	33.4	692	54.9
238	34.4	710	55.6
257	35.4	729	56.3
277	36.3	749	57.1
295	37.3	768	57.7
314	38.2	787	58.5
332	39.1	803	59.1
345	39.7	838	60.4
370	40.9	856	61.1
391	41.9	874	61.8
411	42.8	894	62.5
429	43.6	902	62.9
455	44.8		

[a]Dimensions of the specimen were 0.0853 cm. in diameter and 28.766 cm. in length. The specimen was annealed for nine hours at a temperature between 650° and 700°C.

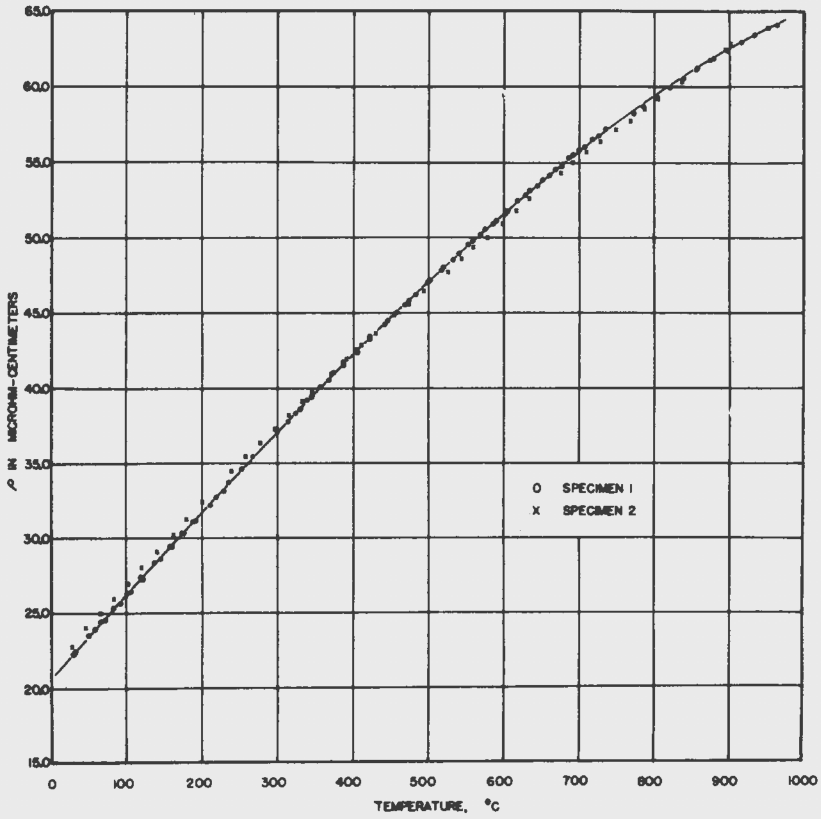

Figure 8. Temperature dependence of electrical resistivity of thorium specimens 1 and 2.

TABLE X

Resistivity Values of Thorium Specimen 3[a] on Heating

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
30	21.0	368	41.9
46	22.4	385	42.8
63	23.4	402	43.8
80	24.5	414	44.5
94	25.3	430	45.4
124	27.1	445	46.2
144	28.3	470	47.6
160	29.4	498	49.1
180	30.6	510	49.8
196	31.6	525	50.5
217	32.8	541	51.4
233	33.8	558	52.2
251	34.9	578	53.3
268	36.0	599	54.4
290	37.3	616	55.3
306	38.2	631	56.0
336	40.0	647	56.7
350	40.8	662	57.4

[a]Dimensions of the specimen were 0.0959 cm. in diameter and 33.060 cm. in length. The specimen was annealed for one hour at a temperature of 650°C.

TABLE XI

Resistivity Values of Thorium Specimen 4[a] on Heating

T°C	ρ microhm-centimeters	T°C	ρ microhm-centimeters
41	21.6	499	48.7
60	23.3	517	49.7
80	24.3	536	50.6
98	25.5	552	51.4
117	26.7	577	52.6
137	27.9	593	53.4
157	29.1	619	54.7
177	30.3	635	55.5
194	31.3	654	56.4
229	33.4	672	57.2
256	35.0	690	58.1
273	36.1	709	59.0
292	37.2	727	59.8
310	38.3	744	60.6
330	39.4	769	61.7
348	40.4	788	62.5
366	41.4	807	63.4
389	42.7	826	64.3
406	43.7	844	65.1
425	44.8	863	65.9
444	45.7	883	66.7
462	46.8	903	67.6
480	47.8		

[a]Dimensions of the specimen were 0.0942 cm. in diameter and 22.860 cm. in length. The specimen was annealed for four hours at a temperature between 650° and 700°C.

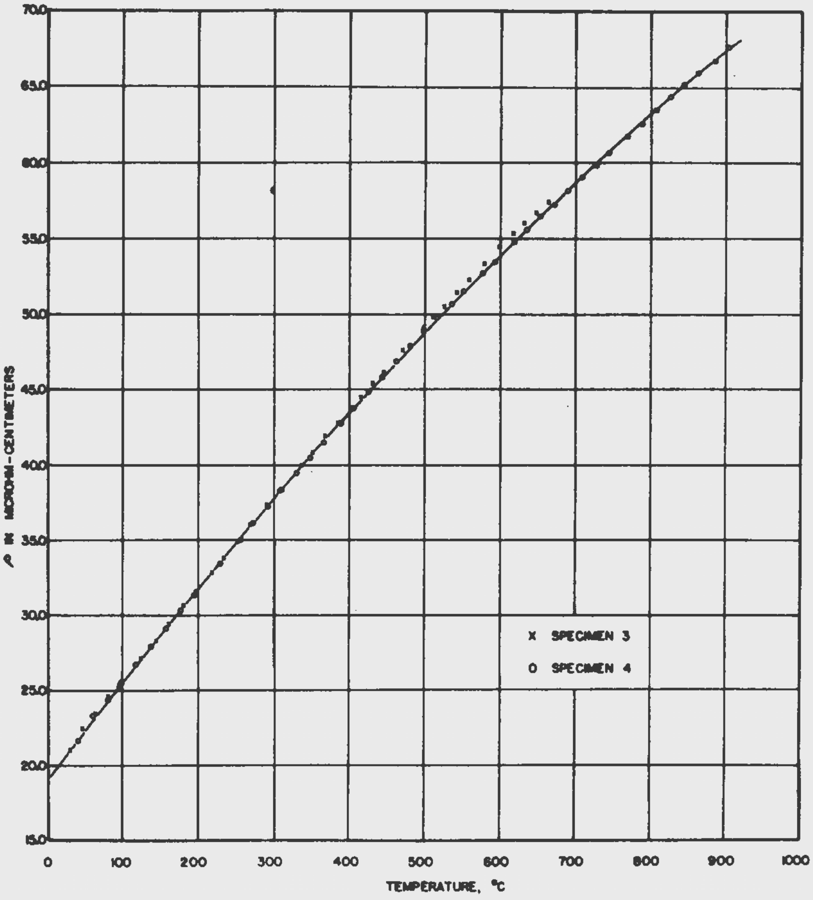

Figure 9. Temperature dependence of electrical resistivity of thorium specimens 3 and 4.

The differences in resistivity and the shape of the resistivity curves may be explained on the basis of the difference in the purity of the specimens. Specimens 1 and 2 contained almost three times the amount of carbon as did specimens 3 and 4. Therefore, one would expect the α for specimens 1 and 2 to be less than that for specimens 3 and 4. Likewise, due to the difference in purity, initial resistivities for specimens 1 and 2 would be expected to be higher than the corresponding ones for specimens 3 and 4. Both these expectations are observed when the resistivity versus temperature curves of the specimens are compared.

Results obtained in this investigation are well within the limits of previously reported values. Bender (19) using thorium of 99.7% purity, ThO_2 the main impurity, obtained resistivity values at room temperature that were higher and temperature coefficients of electrical resistance that were lower than those obtained in this research. Bender's resistivity curve remains above the resistivity curve obtained for the metal with 0.11% carbon over the entire temperature range. However, his curve will cross the resistivity curve obtained for the metal with 0.04% carbon at a temperature between 600° and 700°C. These differences in the behavior of the temperature dependence of electrical resistivity are probably due to the difference in the purity of the metal used. Chiotti (35) using thorium containing 0.09% carbon obtained resistivities of 51.6, 55.7, 60.0 and 63.7 microhm-centimeters at 600°, 700°, 800° and 900°C respectively. Resistivity values for the thorium containing 0.11% carbon for these temperatures agreed quite well, namely, 51.5, 55.6, 59.1 and 62.5 microhm-centimeters. Ames thorium containing 0.04% carbon gave resistivity values of 53.7, 58.5, 63.0 and 67.4 microhm-centimeters at 600°, 700°, 800° and 900°C respectively. These values are higher than the corresponding ones obtained on thorium containing 0.09% and 0.11% carbon.

B. Determination of Discontinuities

1. Graphical

By carrying out resistance measurements on platinum and titanium simultaneously, as was previously described, values of $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ were obtained. Tables 12, 13, 14 and 15 give the $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ values of specimen 1 on heating, specimen 1 on cooling, specimen 2 on heating, and specimen 3 on heating respectively. These values are plotted against temperature as shown in Figures 10, 11, 12 and 13. It is seen that the values for all the specimens can be represented by smooth curves, and there is no pronounced indication of any anomalous discontinuities. The vertical

TABLE XII

$\frac{\Delta R_{ti}}{\Delta R_{pt}}$ Values of Titanium Specimen 1 on Heating

T°C	$\frac{\Delta R_{ti}}{\Delta R_{pt}}$	T°C	$\frac{\Delta R_{ti}}{\Delta R_{pt}}$
53	1.74	431	1.58
78	1.71	445	1.56
94	1.69	460	1.55
106	1.66	479	1.52
120	1.77	497	1.52
134	1.66	513	1.47
147	1.65	541	1.43
159	1.68	557	1.36
176	1.66	574	1.35
189	1.70	594	1.31
221	1.64	609	1.31
233	1.73	630	1.21
247	1.70	646	1.22
258	1.68	669	1.16
270	1.69	683	1.10
287	1.70	702	1.06
301	1.69	720	1.02
323	1.66	739	0.99
339	1.68	753	0.91
353	1.64	770	0.92
364	1.67	794	0.85
374	1.61	812	0.82
392	1.64	830	0.77
404	1.62	843	0.71
419	1.61		

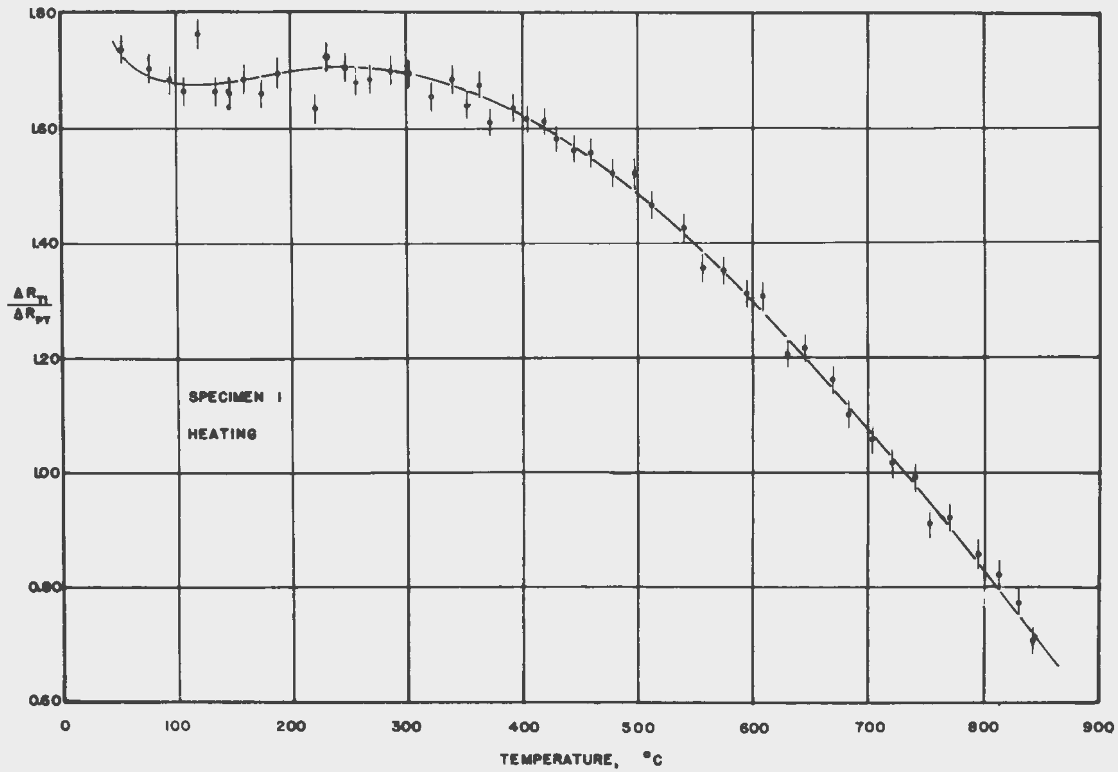

Figure 10. $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ versus temperature curve of titanium specimen 1 on heating.

TABLE XIII

$\frac{\Delta R_{ti}}{\Delta R_{pt}}$ Values of Titanium Specimen 1 on Cooling

T°C	$\frac{\Delta R_{ti}}{\Delta R_{pt}}$	T°C	$\frac{\Delta R_{ti}}{\Delta R_{pt}}$
58	1.71	451	1.58
70	1.70	466	1.53
97	1.65	478	1.51
113	1.66	498	1.48
128	1.70	514	1.46
153	1.74	531	1.42
169	1.78	544	1.43
201	1.68	557	1.37
215	1.66	567	1.42
231	1.70	581	1.32
249	1.73	644	1.25
263	1.70	652	1.19
282	1.67	668	1.14
304	1.70	686	1.07
318	1.72	704	1.02
331	1.66	720	0.99
344	1.68	739	0.98
366	1.66	751	0.89
379	1.65	775	0.84
392	1.62	786	0.87
406	1.61	799	0.78
420	1.62	813	0.74
434	1.59	825	0.70

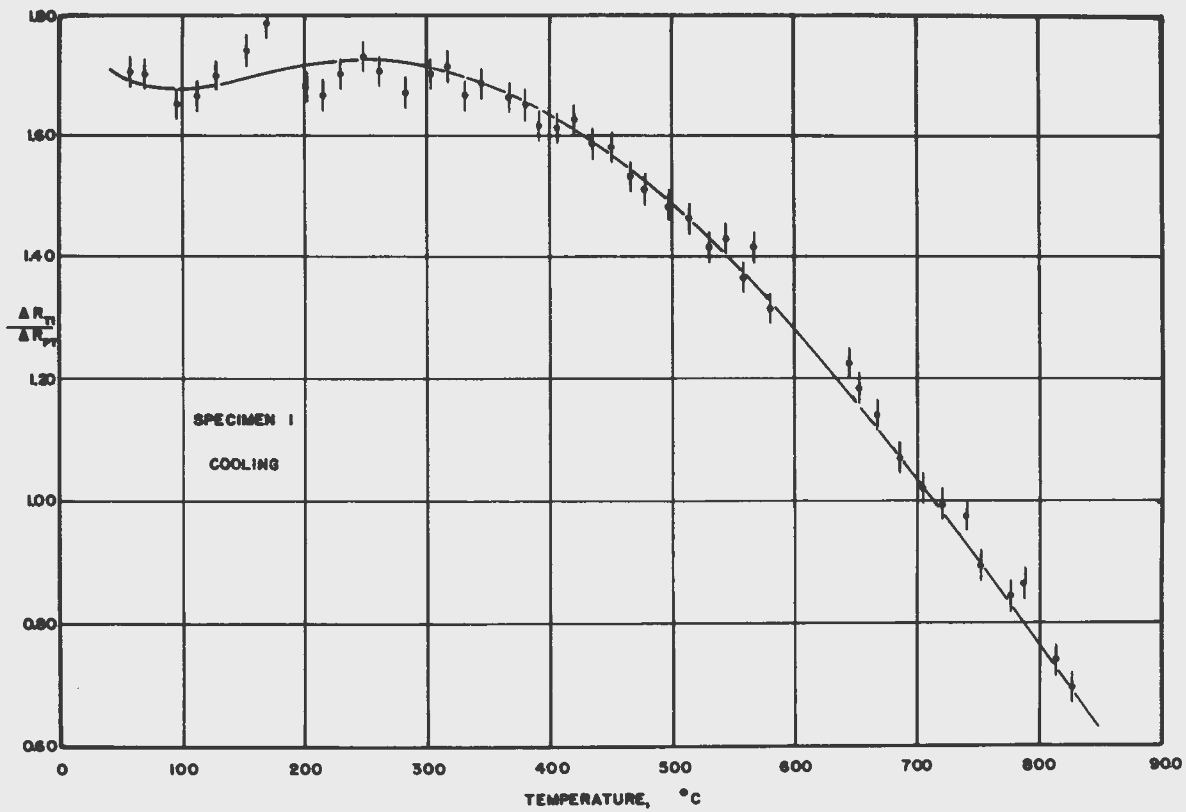

Figure 11. $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ versus temperature curve of titanium specimen 1 on cooling.

TABLE XIV

$\frac{\Delta R_{ti}}{\Delta R_{pt}}$ Values of Titanium Specimen 2 on Heating

T°C	$\frac{\Delta R_{ti}}{\Delta R_{pt}}$	T°C	$\frac{\Delta R_{ti}}{\Delta R_{pt}}$
44	1.72	430	1.68
61	1.78	456	1.55
76	1.84	475	1.57
95	1.82	495	1.51
109	1.85	521	1.47
128	1.79	535	1.44
146	1.84	555	1.26
163	1.84	569	1.37
181	1.84	591	1.43
199	1.82	606	1.37
217	1.83	628	1.30
233	1.83	641	1.27
247	1.76	658	1.21
266	1.74	672	1.19
287	1.82	693	1.13
311	1.77	719	1.06
328	1.81	743	0.98
343	1.79	755	0.99
360	1.72	772	0.88
387	1.66	800	0.92
413	1.68	836	0.79

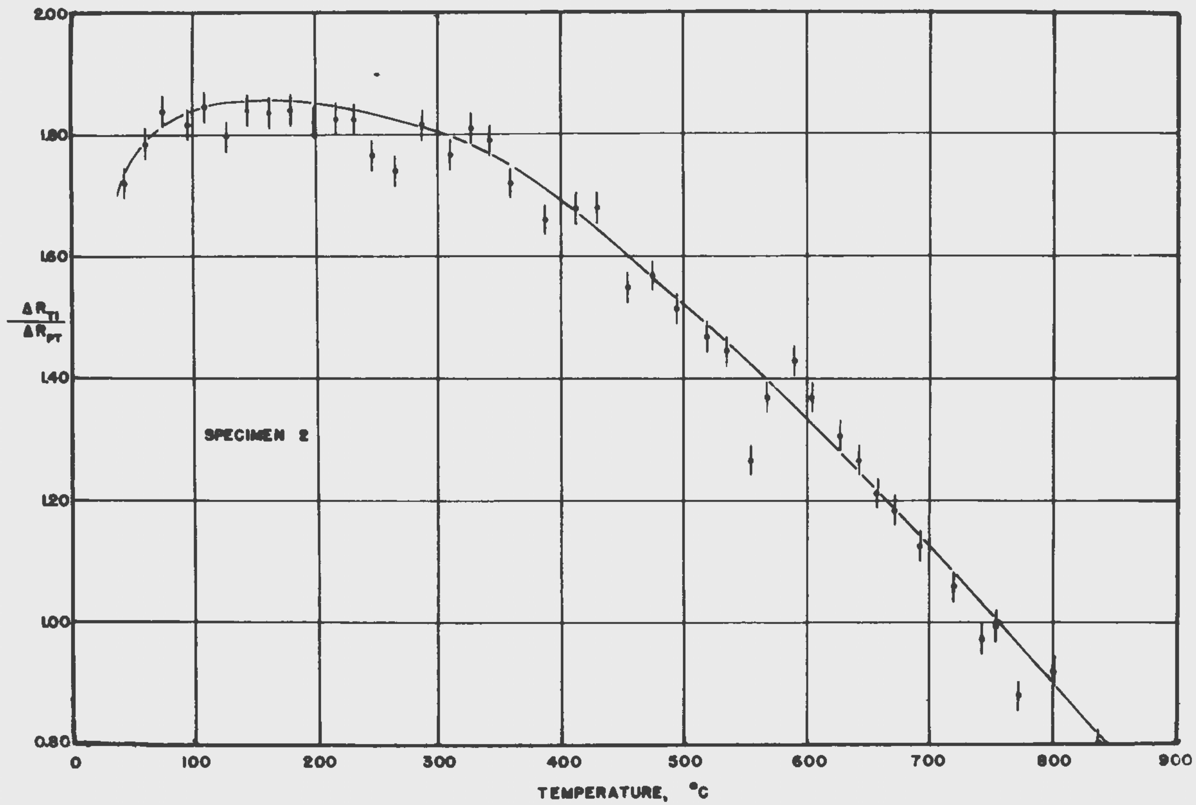

Figure 12. $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ versus temperature curve of titanium specimen 2.

TABLE XV

$\frac{\Delta R_{ti}}{\Delta R_{pt}}$ Values of Titanium Specimen 3 on Heating

T°C	$\frac{\Delta R_{ti}}{\Delta R_{pt}}$	T°C	$\frac{\Delta R_{ti}}{\Delta R_{pt}}$
47	1.87	471	1.39
58	1.82	486	1.40
69	1.74	500	1.37
98	1.76	517	1.30
123	1.76	532	1.30
138	1.77	549	1.28
158	1.73	565	1.20
173	1.72	584	1.16
192	1.70	598	1.15
214	1.71	613	1.10
233	1.70	629	1.07
255	1.69	645	1.01
271	1.67	667	1.00
287	1.67	686	0.91
301	1.64	698	0.89
317	1.63	712	0.84
336	1.62	734	0.80
355	1.58	751	0.81
372	1.57	768	0.73
393	1.55	788	0.72
404	1.52	806	0.66
423	1.49	828	0.66
458	1.43		

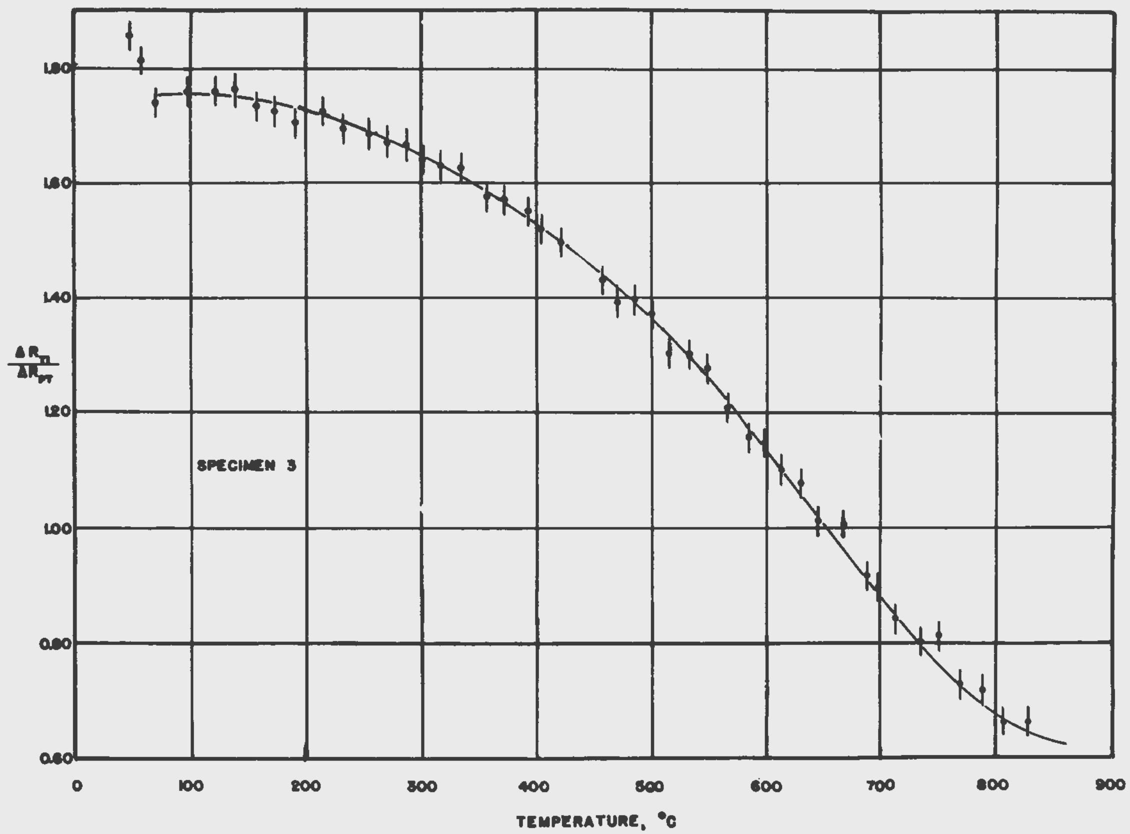

Figure 13. $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ versus temperature curve of titanium specimen 3.

lines, extending above and below each point, represent the precision of the ratio values. The precision of the $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ values was found to be ± 0.03. The precision was calculated according to the method described by Worthing and Geffner (32, p.208). Their equation was employed as follows:

The precision (σ_u) of a function U, where U = f(x,y), is given by the equation

$$\sigma_u = \pm \sqrt{(\partial U/\partial X)^2 \sigma_x^2 + (\partial U/\partial Y)^2 \sigma_y^2}$$

where σ_x and σ_y are the precisions of X and Y respectively. Since the potential drops across the titanium and platinum wires were taken within ten seconds of each other, the variation of current was considered negligible over this period of time and $\frac{\Delta R_{ti}}{\Delta R_{pt}} = \frac{\Delta R_{ti}}{\Delta R_{pt}}$. Performing the partial differentiation, the precision of the ratio is given by the equation

$$\sigma_{\frac{\Delta R_{ti}}{\Delta R_{pt}}} = \pm \sqrt{\frac{(\Delta E_{pt})^2 \sigma_{E_{ti}}^2 + (\Delta E_{ti})^2 \sigma_{E_{pt}}^2}{(\Delta E_{pt})^4}}$$

The precisions of the potential drops across the titanium and platinum were the same and equal to ± 0.00005 volts. ΔE_{ti} was of the order of magnitude of 0.007 volts and ΔE_{pt} was of the order of magnitude of 0.004 volts. Substituting these values, we obtain the precision of $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ to be ± 0.03.

It is seen from Figures 10 and 11, that the $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ versus temperature curve for specimen 1 on heating was reproduced on cooling. There are, however, differences in the shape of the $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ curves among the three specimens. The actual values of the ratio will of course differ with titanium and platinum specimens of different resistances. Since there were differences in the shape of the resistivity curves for the three specimens, it follows that these differences will again appear in the $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ versus temperature curves. Resistivity values for platinum observed at various temperatures showed some variation during the course of eight separate runs. This variation could be due to physical changes in the platinum wire or due to errors in the temperature indicated by the chromel-alumel thermocouple used. Assuming the resistivity of the platinum remained constant, the error in temperature measurement was within ± 3°C, ± 4°C and ± 5°C over the temperature ranges 25° to 300°C, 300° to 500°C and 500° to 800°C respectively.

Tables 16 and 17 give the $\frac{\Delta R_{fe}}{\Delta R_{pt}}$ values for electrolytic iron during heating and cooling. The $\frac{\Delta R_{fe}}{\Delta R_{pt}}$ versus temperature curve for both heating and cooling is shown in Figure 14. The values fall on one smooth curve with one discontinuity corresponding to the inflection point in the resistivity curve for iron. However, aside from this expected discontinuity, no other discontinuities appear in the $\frac{\Delta R_{fe}}{\Delta R_{pt}}$ versus temperature curve. The precision of the ratio values was found to be ± 0.3 by the same method that was used to determine the precision of the ratio $\frac{\Delta R_{ti}}{\Delta R_{pt}}$. In this case, ΔE_{fe} was of the order of magnitude of 0.07 volts, while the other quantities were the same as for the titanium.

Values of $\frac{\Delta R_{th}}{\Delta R_{pt}}$ for thorium specimen 1 on heating and cooling and for specimen 4 on heating are given in Tables 18, 19 and 20 respectively. In the case of specimen 4, resistance values of platinum were read directly from a resistance versus temperature curve for platinum which was previously obtained. Figures 15, 16 and 17 show the $\frac{\Delta R_{th}}{\Delta R_{pt}}$ versus temperature curves for specimen 1 on heating, specimen 1 on cooling, and specimen 4 on heating respectively. As with the curves for titanium, the vertical lines extending above and below each point indicate the precision of the ratio values. The precision of the $\frac{\Delta R_{th}}{\Delta R_{pt}}$ values was found to be ± 0.02. This was calculated by the method previously described. For thorium, ΔE_{th} was of the order of magnitude of 0.003 volts, which the other quantities had the same values as in the case of titanium.

From the curves, it is seen that although some points appear to fall on a horizontal line, a smooth curve seems to give the best fit when the precision of the ratio values is taken into consideration. From room temperature to 900°C, the slope of the resistivity curve for

TABLE XVI

$\frac{\Delta R_{fe}}{\Delta R_{pt}}$ Values of Iron on Heating

T°C	$\frac{\Delta R_{fe}}{\Delta R_{pt}}$	T°C	$\frac{\Delta R_{fe}}{\Delta R_{pt}}$
59	6.2	480	17.0
79	6.7	500	17.9
106	7.1	526	18.3
128	7.7	542	17.6
139	8.4	575	17.7
153	8.7	597	20.7
172	8.9	617	21.3
183	9.4	642	22.9
206	9.8	696	28.8
222	9.9	717	31.8
258	10.3	732	18.9
269	11.2	765	12.6
309	12.4	796	7.5
378	14.1	824	5.9
393	13.8	855	5.2
409	14.2	872	5.0
422	15.0	894	4.7
464	16.5		

TABLE XVII

$\frac{\Delta R_{fe}}{\Delta R_{pt}}$ Values of Iron on Cooling

T°C	$\frac{\Delta R_{fe}}{\Delta R_{pt}}$	T°C	$\frac{\Delta R_{fe}}{\Delta R_{pt}}$
41	5.7	424	14.3
55	6.2	443	15.7
80	6.6	462	15.9
94	7.4	478	16.2
116	7.5	492	17.8
132	7.8	568	19.0
151	8.3	586	19.5
169	8.6	601	20.5
191	9.1	681	27.5
228	9.7	713	31.8
246	10.5	730	23.5
264	10.8	752	14.4
284	11.2	773	12.1
304	11.6	793	8.3
325	12.6	813	7.0
344	13.1	829	6.4
362	13.0	848	5.1
382	14.1	887	4.7

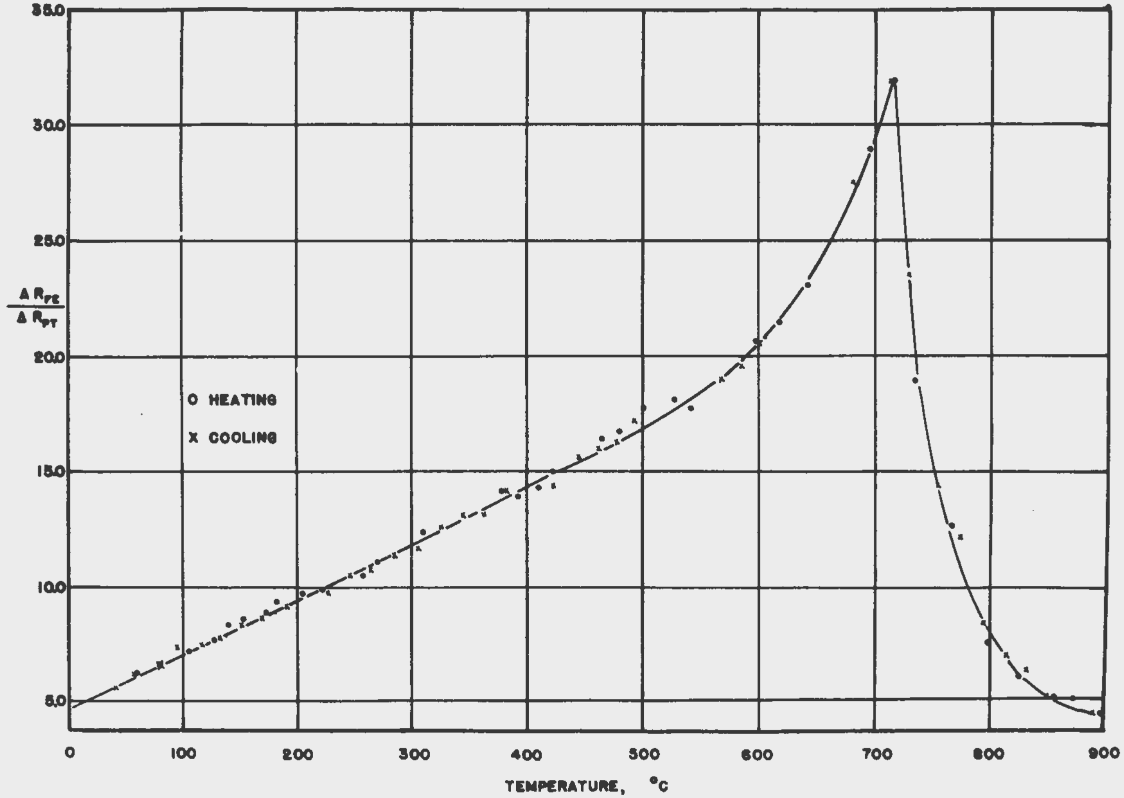

Figure 14. $\frac{\Delta R_{fe}}{\Delta R_{pt}}$ versus temperature curve of iron.

TABLE XVIII

$\frac{\Delta R_{th}}{\Delta R_{pt}}$ Values of Thorium Specimen 1 on Heating

T°C	$\frac{\Delta R_{th}}{\Delta R_{pt}}$	T°C	$\frac{\Delta R_{th}}{\Delta R_{pt}}$
66	0.856	422	0.751
82	0.817	445	0.741
100	0.803	468	0.747
119	0.799	483	0.740
137	0.788	499	0.742
158	0.785	517	0.747
173	0.780	532	0.743
188	0.778	552	0.757
219	0.785	569	0.738
235	0.790	586	0.706
252	0.795	619	0.729
267	0.787	634	0.745
299	0.775	652	0.712
323	0.771	668	0.702
338	0.776	686	0.678
356	0.782	700	0.678
374	0.770	718	0.682
387	0.759	735	0.660
404	0.757	752	0.637

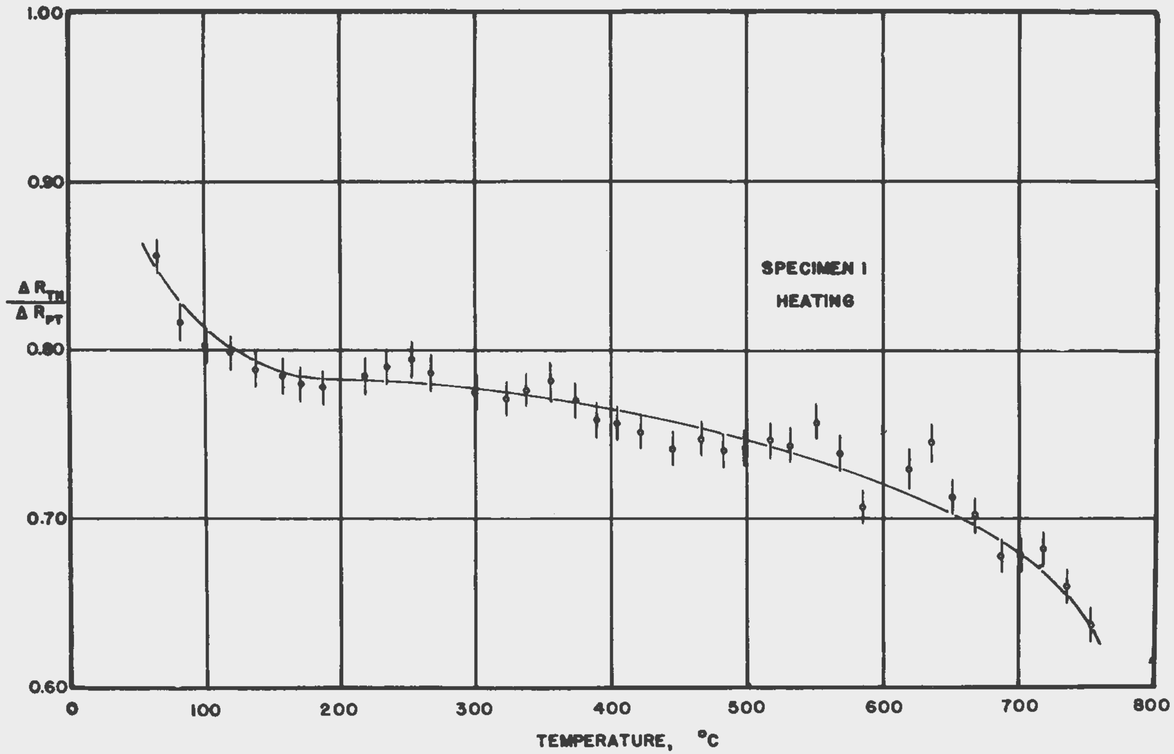

Figure 15. $\frac{\Delta R_{th}}{\Delta R_{pt}}$ versus temperature of thorium specimen 1 on heating.

TABLE XIX

$\frac{\Delta R_{th}}{\Delta R_{pt}}$ Values of Thorium Specimen 1 on Cooling

T°C	$\frac{\Delta R_{th}}{\Delta R_{pt}}$	T°C	$\frac{\Delta R_{th}}{\Delta R_{pt}}$
58	0.794	540	0.719
72	0.793	559	0.716
91	0.809	575	0.716
106	0.796	589	0.701
121	0.791	604	0.701
146	0.792	629	0.681
160	0.791	645	0.685
176	0.786	661	0.696
192	0.800	677	0.683
211	0.798	692	0.688
228	0.778	708	0.685
314	0.776	726	0.682
330	0.773	773	0.648
345	0.770	786	0.639
368	0.763	804	0.620
388	0.760	821	0.629
405	0.754	839	0.641
422	0.755	858	0.591
442	0.764	880	0.565
458	0.754	899	0.568
474	0.742	917	0.543
502	0.742	935	0.484
520	0.741	953	0.384

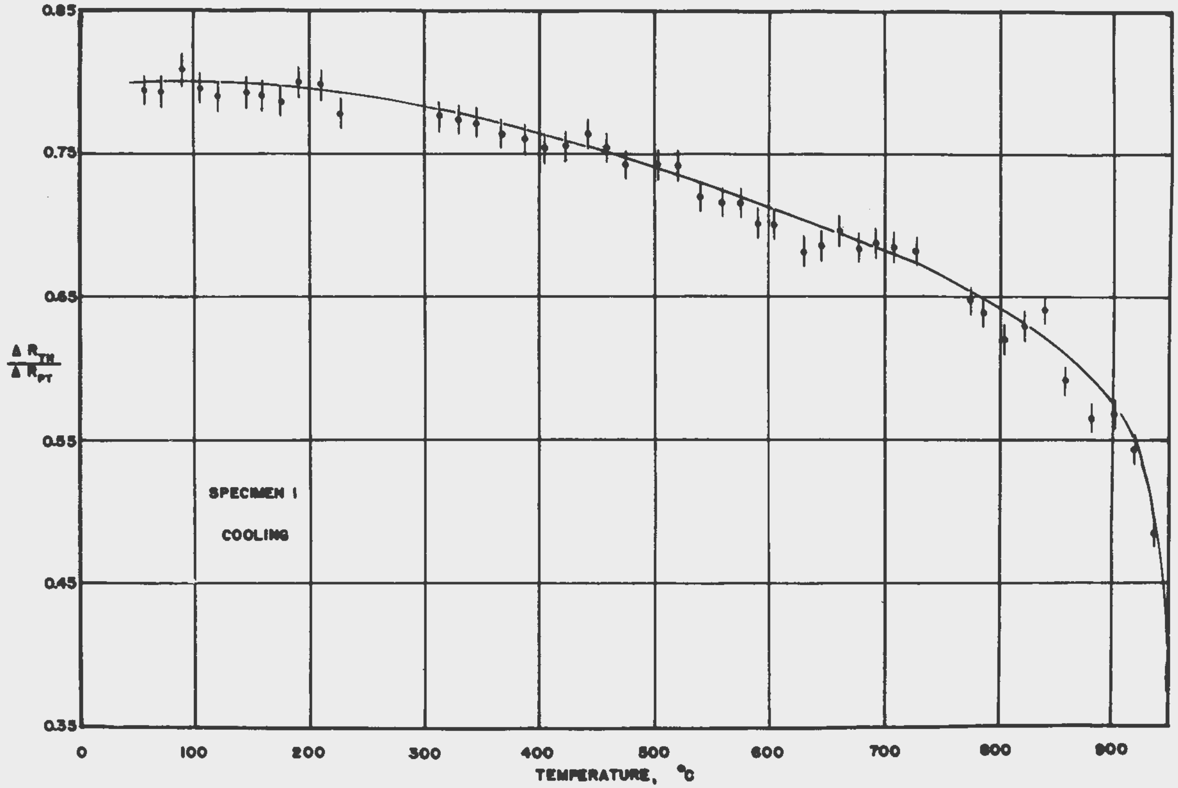

Figure 16. $\frac{\Delta R_{th}}{\Delta R_{pt}}$ versus temperature of thorium specimen 1 on cooling.

TABLE XX

$\frac{\Delta R_{th}}{\Delta R_{pt}}$ Values of Thorium Specimen 4 on Heating

T°C	$\frac{\Delta R_{th}}{\Delta R_{pt}}$	T°C	$\frac{\Delta R_{th}}{\Delta R_{pt}}$
98	0.632	517	0.525
117	0.605	536	0.514
137	0.573	552	0.516
157	0.554	577	0.515
177	0.548	593	0.512
194	0.557	619	0.517
229	0.556	635	0.518
256	0.549	654	0.501
273	0.547	672	0.492
292	0.540	690	0.503
310	0.541	709	0.501
330	0.533	727	0.497
348	0.525	744	0.496
366	0.529	769	0.496
389	0.518	788	0.501
406	0.521	807	0.496
425	0.534	826	0.481
444	0.535	844	0.479
462	0.535	863	0.482
480	0.537	883	0.468
499	0.539	903	0.460

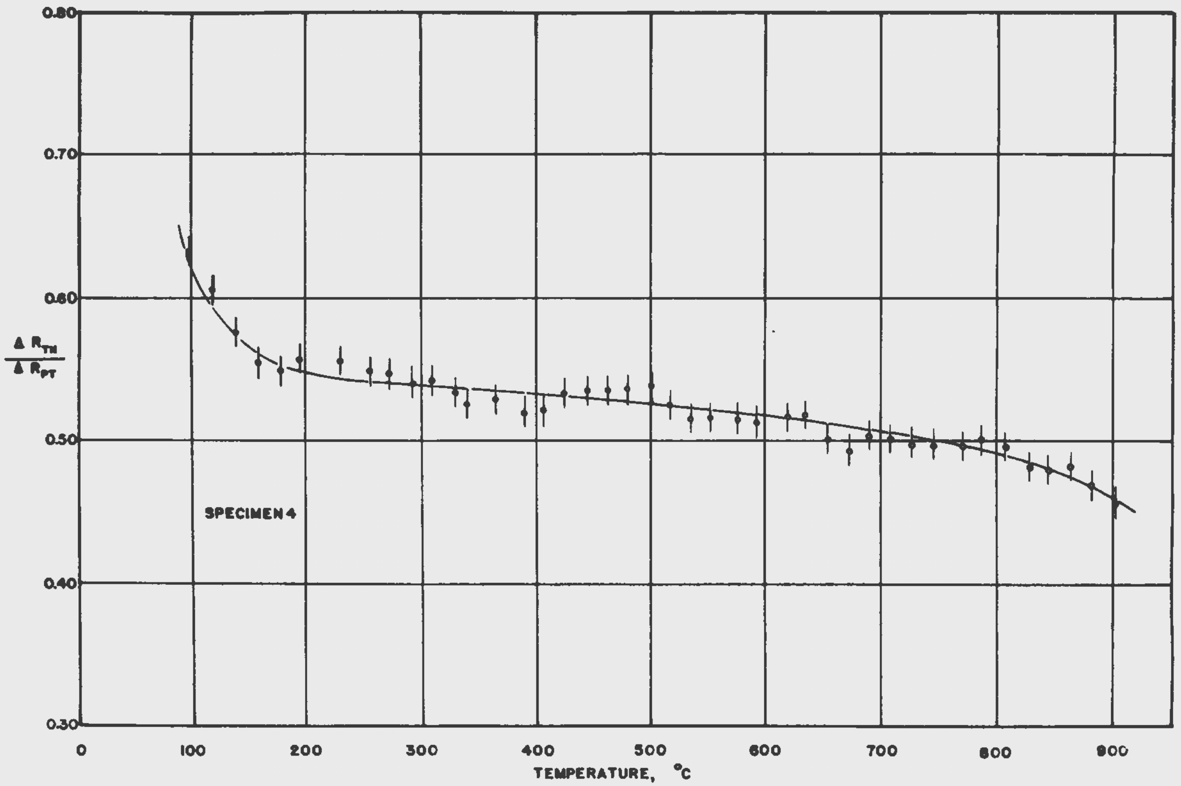

Figure 17. $\frac{\Delta R_{th}}{\Delta R_{pt}}$ versus temperature of thorium specimen 4.

thorium changes comparatively very little and therefore, $\frac{\Delta R_{th}}{\Delta R_{pt}}$ will change only very slightly over small increments of temperature. Since these straight line segments are not reproducible even with the same specimen, it was conlcuded that no apparent anomalous discontinuities exist and the curves were smooth curves. As with titanium, the differences in shape between the curves for the two specimens of thorium may be accounted for by the differences in shape in their resistivity curves.

2. Analytical

When the resistance versus temperature curve for titanium was plotted on a rather enlarged scale, 50 cm. by 100 cm. graph paper, covering the range up to 850°C, it appears that the data might best be approximated by segments of straight lines. In other words, it appears that the resistance is a linear function of the temperature over a number of temperature ranges, namely, 0° to 335°C, 335° to 516°C, 516° to 634°C, 634° to 726°C, 726° to 802°C and 802° up to 850°C. The change in slope at 335°C and 516°C is quite small; those occurring at higher temperatures are somewhat more pronounced, but the number of points between the discontinuities is small so that there is reason to doubt the reality of these discontinuities in slope. However, it was found that these discontinuities could be reproduced to within ± 20°C on different runs. It was also noted that the temperatures of the occurrance of these anomalous changes in slope corresponded quite closely to temperatures at which Jaeger and his co-workers (17) found evidence of minor transitions in their specific heat measurements on titanium. As was previously stated, they also found some evidence for these anomalous effects in their results on the temperature dependence of electrical resistance for titanium.

No obvious discontinuities other than at the Curie point were observed in the resistance versus temperature curve for iron. Some indications of such discontinuities were observed in the case of thorium.

In order to help resolve the question of the reality of these discontinuities relative to the results obtained in this investigation, the least squares method and the criteria for closeness of fit were applied to the data. The closeness of fit of the relations $\frac{R}{R_o} = a + bt$ and $\frac{R}{R_o} = a + bt + ct^2$ to the data over regions in question were determined.

Before applying the least squares method and the criteria for closeness of fit to resistance data, it was decided to determine the limitations of this analytical method to a similar problem. In order to carry out this analysis, this method was applied to a curve that is definitely a smooth curve, namely, the sine curve from 0° to 90°. Sine values were obtained directly from a table; however, errors of the same order of magnitude as those in the potential measurements, up to ±0.00005 volts, were introduced at random. By plotting sine θ versus θ on an enlarged scale, it was possible to draw the sine curve as six straight line segments. These segments covered the ranges 0° to 33°, 33° to 60°, 60° to 74°, 74° to 84° and 84° to 90°. The least squares method was applied to the sine curve both as one smooth curve having the assumed equation sine $\theta = 0.0 + a\theta + b\theta^2$ and as six straight line segments represented by equations of the type sine $\theta = a + \theta b$. After evaluating the constants in the assumed equations, and obtaining the deviations and the squares of the deviations between the observed and calculated values of sine θ, the criteria for closeness of fit was applied. It was found that over the entire range of 0° to 90°, the six straight line segments gave a better fit to the data than one smooth curve by a factor of 5.4. Over the range 0° to 60°, in which the slope of the sine curve does not change with great rapidity, one smooth curve gave a better fit to the data than two straight line segments by the factor of 1.7. Therefore, it was concluded that this analytical method, in which the equation for a smooth curve contains only three constants, can only be applied over a limited range in which the curve does not show marked changes in slope.

The least squares method was applied to both the heating and cooling data obtained for titanium specimen 1. From the resistance curve, it appeared that two discontinuities existed over the range 0° to 600°C, one at 335°C and the other at 516°C. Therefore the resistance curve was drawn as three straight line segments over this range. The change in slope between these two segments meeting at 335°C was not great and therefore the analytical method could be applied. The segment covering the range 0° to 335°C had as its assumed equation $\frac{R}{R_o} = 1.0000 + at$ where $R_o = 0.15220$. The constant <u>a</u> evaluated by the least squares method was found to be 0.003941. The segment from 335° to 516°C had the assumed equation $\frac{R}{R_o} = a + bt$, and the constants as evaluated by the least squares method were found to be a = 1.1831 and b = 0.003406.

Tables 21 and 22 give the deviations and the squares of the deviations between the observed and calculated values of $\frac{R}{R_o}$ for the two straight line segments under discussion. The equation $\frac{R}{R_o} = 1.0000 +$

TABLE XXI

Deviations between the observed and calculated values of $\frac{R}{R_o}$ of titanium specimen 1 from 0° to 335°C assuming the equation of the resistance versus temperature curve is $\frac{R}{R_o} = 1.0000 + 0.003941\ t$, where R_o equals 0.15220.

T°C	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_c$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]^2$
0	1.0000	1.0000	0.0000	0.00000000
24	1.1014	1.0946	0.0070	0.00004900
30	1.1171	1.1182	-0.0011	0.00000121
37	1.1499	1.1458	0.0041	0.00001681
40	1.1665	1.1576	0.0089	0.00007921
53	1.2083	1.2089	-0.0006	0.00000036
58	1.2287	1.2286	0.0001	0.00000001
66	1.2592	1.2601	-0.0009	0.00000081
70	1.2786	1.2759	0.0027	0.00000729
78	1.3056	1.3074	-0.0018	0.00000324
94	1.3692	1.3705	-0.0013	0.00000169
97	1.3830	1.3823	0.0007	0.00000049
106	1.4126	1.4177	-0.0051	0.00002601
113	1.4443	1.4453	-0.0010	0.00000100
120	1.4718	1.4729	-0.0011	0.00000121
127	1.5032	1.5005	0.0027	0.00000729
134	1.5241	1.5281	-0.0040	0.00001600
147	1.5739	1.5793	-0.0054	0.00002916
153	1.6031	1.6030	-0.0001	0.00000001
159	1.6242	1.6266	-0.0024	0.00000576
169	1.6659	1.6660	-0.0001	0.00000001
176	1.6927	1.6936	-0.0009	0.00000081
189	1.7469	1.7448	0.0021	0.00000441
201	1.7964	1.7921	0.0043	0.00001849
215	1.8546	1.8473	0.0073	0.00005239
221	1.8722	1.8710	0.0012	0.00000144
231	1.9142	1.9104	0.0038	0.00001444
233	1.9179	1.9183	-0.0004	0.00000016
247	1.9763	1.9734	0.0029	0.00000841
249	1.9847	1.9813	0.0034	0.00001156

TABLE XXI (Continued)

T^oC	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_c$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]^2$
258	2.0159	2.0168	-0.0009	0.00000081
263	2.0442	2.0365	0.0077	0.00005929
270	2.0635	2.0641	-0.0006	0.00000036
282	2.1140	2.1114	0.0026	0.00000676
287	2.1311	2.1311	0.0000	0.00000000
301	2.1850	2.1862	-0.0012	0.00000144
304	2.1972	2.1981	-0.0009	0.00000081
318	2.2500	2.2532	-0.0032	0.00001024
323	2.2684	2.2729	-0.0045	0.00002025
331	2.2986	2.3045	-0.0059	0.00003481
				0.00049735

TABLE XXII

Deviations between the observed and calculated values of $\frac{R}{R_o}$ of titanium specimen 1 from 335° to 516°C assuming the equation of the resistance versus temperature curve is $\frac{R}{R_o} = 1.1831 + 0.003406\ t$, where R_o equals 0.15220.

T°C	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_c$	$\left[(\frac{R}{R_o})_c - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_o\right]^2$
339	2.2392	2.3377	-0.0085	0.00007225
344	2.3502	2.3548	-0.0046	0.00002116
353	2.3782	2.3854	-0.0072	0.00005184
364	2.4185	2.4229	-0.0044	0.00001936
366	2.4286	2.4297	-0.0009	0.00000081
374	2.4538	2.4569	-0.0031	0.00000961
379	2.4758	2.4740	0.0018	0.00000324
392	2.5181	2.5183	-0.0002	0.00000004
392	2.5200	2.5183	0.0017	0.00000289
404	2.5639	2.5591	0.0048	0.00002304
406	2.5713	2.5659	0.0054	0.00002916
419	2.6143	2.6102	0.0041	0.00001681
420	2.6212	2.6136	0.0076	0.00005776
431	2.6557	2.6511	0.0046	0.00002116
434	2.6672	2.6613	0.0059	0.00003481
445	2.7037	2.6988	0.0049	0.00002401
451	2.7263	2.7192	0.0071	0.00005041
460	2.7534	2.7499	0.0035	0.00001225
466	2.7754	2.7703	0.0051	0.00002601
478	2.8130	2.8112	0.0018	0.00000324
479	2.8166	2.8146	0.0020	0.00000400
497	2.8706	2.8759	-0.0053	0.00002809
498	2.8751	2.8793	-0.0042	0.00001764
513	2.9189	2.9304	-0.0115	0.00013225
514	2.9235	2.9338	-0.0103	0.00010609
				0.00076793

at + bt^2 was also assumed to fit the data between 0°C and 516°C. By applying the least squares method, a was found to equal 0.004102 and b was found to equal -0.0000006226. Table 23 gives the deviations and the squares of the deviations between the observed and calculated values of $\frac{R}{R_o}$ for this smooth curve over the range 0° to 516°C.

The closeness of fit criteria was applied and gave the following results.

$$\Omega \text{ two straight line segments} = \frac{\Sigma(y_o - y)^2}{n - m} =$$

$$\frac{0.0049735 - 0.00076793}{65 - 3} = 0.00002041.$$

$$\Omega \text{ smooth curve} = \frac{\Sigma(y_o - y)^2}{n - m} = \frac{0.0049838}{65 - 2} = 0.00007911.$$

$$\frac{\Omega \text{ smooth curve}}{\Omega \text{ two straight line segments}} = 3.9.$$

Therefore, two straight line segments gave a better fit to the observed data than one smooth curve of the assumed form by a factor of 3.9 over the temperature range 0° to 516°C.

The least squares method was applied to both the heating and cooling data obtained for thorium specimen 1. From the resistance versus temperature curve, it appeared that two discontinuities existed over the temperature range 0° to 700°C, one at 356°C and the other at 540°C. As a result, the resistance curve was drawn as three straight line segments over this range. The change in slope between the two segments meeting at 356°C was small and therefore the analytical method could be applied. The equation covering the range 0° to 356°C was found to be $\frac{R}{R_o} = 1.0000 + 0.002665t$ where $R_o = 0.10540$. The equation for the range 356° to 540°C was found to be $\frac{R}{R_o} = 1.1164 + 0.002324t$. Tables 24 and 25 give the deviations and the squares of the deviations between the observed and calculated values of $\frac{R}{R_o}$ for the two straight line segments. The equation $\frac{R}{R_o} = 1.0000 + 0.002780t - 0.0000004355t^2$ was also assumed to fit the data. Table 26 shows the deviations and the squares of the deviations between the observed and calculated values of $\frac{R}{R_o}$ for this smooth curve over the temperature range 0° to 540°C.

Applying the equation for closeness of fit gave the following results.

TABLE XXIII

Deviations between the observed and calculated values of $\frac{R}{R_o}$ of titanium specimen 1 from 0° to 516°C assuming the equation of the resistance versus temperature curve is $\frac{R}{R_o} = 1.0000 + 0.004102\ t - 0.0000006226\ t^2$, where R_o equals 0.15220.

T°C	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_c$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]^2$
0	1.0000	1.0000	0.0000	0.00000000
24	1.1014	1.0981	0.0033	0.00001089
30	1.1171	1.1225	-0.0054	0.00002916
37	1.1499	1.1509	-0.0010	0.00000100
40	1.1665	1.1631	0.0034	0.00001156
53	1.2083	1.2157	-0.0074	0.00005476
58	1.2287	1.2358	-0.0071	0.00005041
66	1.2592	1.2680	-0.0088	0.00007744
70	1.2786	1.2840	-0.0054	0.00002916
78	1.3056	1.3164	-0.0108	0.00011664
94	1.3692	1.3801	-0.0109	0.00011881
97	1.3830	1.3920	-0.0090	0.00008100
106	1.4126	1.4278	-0.0152	0.00023104
113	1.4443	1.4555	-0.0112	0.00012544
120	1.4718	1.4832	-0.0114	0.00012996
128	1.5032	1.5149	-0.0117	0.00013689
134	1.5241	1.5385	-0.0144	0.00020736
147	1.5739	1.5895	-0.0156	0.00024336
153	1.6031	1.6130	-0.0099	0.00009801
159	1.6242	1.6365	-0.0123	0.00015129
169	1.6659	1.6754	-0.0095	0.00009025
176	1.6927	1.7027	-0.0100	0.00010000
189	1.7469	1.7531	-0.0062	0.00003844
201	1.7964	1.7993	-0.0029	0.00000841
215	1.8546	1.8531	0.0015	0.00000225
221	1.8722	1.8761	-0.0039	0.00001521
231	1.9142	1.9144	-0.0002	0.00000004
233	1.9179	1.9180	-0.0001	0.00000001
247	1.9763	1.9752	0.0011	0.00000121
249	1.9847	1.9828	0.0019	0.00000361

TABLE XXIII (Continued)

T^oC	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_c$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]^2$
258	2.0159	2.0168	-0.0009	0.00000081
263	2.0442	2.0357	0.0085	0.00007225
270	2.0635	2.0621	0.0014	0.00000196
282	2.1140	2.1073	0.0067	0.00004489
287	2.1311	2.1260	0.0051	0.00002601
301	2.1850	2.1783	0.0067	0.00004489
304	2.1972	2.1895	0.0077	0.00005929
318	2.2500	2.2414	0.0086	0.00007396
323	2.2684	2.2600	0.0084	0.00007056
331	2.2986	2.2896	0.0090	0.00008100
339	2.3292	2.3191	0.0101	0.00010201
344	2.3502	2.3374	0.0128	0.00016384
353	2.3782	2.3704	0.0078	0.00006084
364	2.4185	2.4106	0.0079	0.00006241
366	2.4286	2.4179	0.0105	0.00011025
374	2.4538	2.4470	0.0058	0.00003364
379	2.4758	2.4653	0.0105	0.00011025
392	2.5181	2.5123	0.0058	0.00003364
392	2.5200	2.5123	0.0077	0.00005929
404	2.5639	2.5556	0.0083	0.00006889
406	2.5713	2.5628	0.0085	0.00007225
419	2.6143	2.6094	0.0049	0.00002401
420	2.6212	2.6130	0.0082	0.00006724
431	2.6557	2.6523	0.0034	0.00001156
434	2.6672	2.6630	0.0042	0.00001764
445	2.7037	2.7021	0.0016	0.00000256
451	2.7263	2.7234	0.0029	0.00000841
460	2.7532	2.7552	-0.0020	0.00000400
466	2.7754	2.7763	-0.0009	0.00000081
478	2.8130	2.8185	-0.0055	0.00003025
479	2.8166	2.8230	-0.0064	0.00004096
497	2.8706	2.8849	-0.0143	0.00020449
498	2.8706	2.8884	-0.0133	0.00017689
513	2.9189	2.9404	-0.0215	0.00046225
514	2.9235	2.9439	-0.0204	0.00041616
				0.00498377

TABLE XXIV

Deviations between the observed and calculated values of $\frac{R}{R_o}$ of thorium specimen 1 from 0° to 356°C assuming the equation of the resistance versus temperature curve is $\frac{R}{R_o} = 1.0000 + 0.002665\ t$, where R_o equals 0.10540.

T°C	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_c$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]^2$
0	1.0000	1.0000	0.0000	0.00000000
30	1.0809	1.0800	0.0009	0.00000081
31	1.0835	1.0826	0.0009	0.00000081
50	1.1369	1.1323	0.0036	0.00001296
58	1.1568	1.1546	0.0022	0.00000484
66	1.1825	1.1759	0.0066	0.00004356
72	1.1857	1.1919	-0.0062	0.00003844
82	1.2262	1.2185	0.0077	0.00005929
91	1.2387	1.2425	-0.0038	0.00001444
100	1.2753	1.2665	0.0088	0.00007444
106	1.2801	1.2825	-0.0024	0.00000576
119	1.3273	1.3171	0.0102	0.00010404
121	1.3201	1.3225	-0.0024	0.00000576
137	1.3757	1.3651	0.0106	0.00011236
146	1.3857	1.3891	-0.0034	0.00001156
158	1.4294	1.4211	0.0083	0.00006889
160	1.4253	1.4264	-0.0011	0.00000121
173	1.4676	1.4610	0.0066	0.00004356
176	1.4671	1.4690	-0.0019	0.00000361
188	1.5082	1.5010	0.0072	0.00005184
192	1.5090	1.5117	-0.0027	0.00000729
211	1.5609	1.5623	-0.0014	0.00000196
219	1.5849	1.5836	0.0013	0.00000169
228	1.6071	1.6076	-0.0005	0.00000025
235	1.6308	1.6263	0.0045	0.00002025
252	1.6789	1.6716	0.0073	0.00005329
267	1.7176	1.7116	0.0060	0.00003600
299	1.8001	1.7968	0.0033	0.00001089
314	1.8306	1.8368	-0.0062	0.00003844
323	1.8568	1.8610	-0.0042	0.00001764

TABLE XXIV (Continued)

$T^{o}C$	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_c$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]^2$
330	1.8712	1.8795	-0.0073	0.00005329
338	1.8994	1.9008	-0.0014	0.00000196
345	1.9098	1.9194	-0.0096	0.00009216
356	1.9433	1.9487	-0.0054	0.00002916
				0.00102545

TABLE XXV

Deviations between the observed and calculated values of $\frac{R}{R_o}$ of thorium specimen 1 from 356° to 540°C assuming the equation of the resistance versus temperature curve is $\frac{R}{R_o} = 1.1164 + 0.002324\ t$, where Ro equals 0.10540.

T°C	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_c$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]^2$
368	1.9661	1.9716	-0.0055	0.00003025
374	1.9856	1.9856	0.0000	0.00000000
387	2.0193	2.0158	0.0035	0.00001225
388	2.0148	2.0181	-0.0033	0.00001089
404	2.0574	2.0553	0.0021	0.00000441
405	2.0556	2.0576	-0.0020	0.00000400
422	2.1009	2.0971	0.0038	0.00001444
422	2.0966	2.0971	-0.0005	0.00000025
442	2.1441	2.1436	0.0005	0.00000025
445	2.1528	2.1506	0.0022	0.00000484
458	2.1808	2.1776	0.0022	0.00000484
468	2.2058	2.2040	0.0018	0.00000324
474	2.2203	2.2180	0.0023	0.00000529
483	2.2392	2.2389	0.0003	0.00000009
499	2.2776	2.2761	0.0015	0.00000225
502	2.2838	2.2830	0.0008	0.00000064
517	2.3155	2.3179	-0.0024	0.00000576
520	2.3251	2.3249	0.0002	0.00000004
532	2.3504	2.3528	-0.0024	0.00000576
540	2.3693	2.3714	-0.0021	0.00000441
				0.00011390

TABLE XXVI

Deviations between the observed and calculated values of $\frac{R}{R_o}$ of thorium specimen 1 from 0° to 540°C assuming the equation of the resistance versus temperature curve is $\frac{R}{R_o} = 1.0000 + 0.002780\ t - 0.0000004335\ t^2$, where R_o equals 0.10540Ω.

T°C	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_e$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]^2$
0	1.0000	1.0000	0.0000	0.00000000
30	1.0809	1.0830	-0.0021	0.00000441
31	1.0835	1.0858	-0.0023	0.00000529
50	1.1369	1.1379	-0.0010	0.00000100
58	1.1568	1.1597	-0.0029	0.00000841
66	1.1825	1.1816	0.0009	0.00000081
72	1.1857	1.1980	-0.0123	0.00015129
82	1.2262	1.2251	0.0011	0.00000121
91	1.2387	1.2494	-0.0107	0.00011449
100	1.2753	1.2737	0.0016	0.00000256
106	1.2801	1.2898	-0.0097	0.00009409
119	1.3273	1.3247	0.0026	0.00000676
121	1.3201	1.3301	-0.0100	0.00010000
137	1.3757	1.3728	0.0029	0.00000841
146	1.3857	1.3967	-0.0110	0.00012100
158	1.4294	1.4284	0.0010	0.00000100
160	1.4253	1.4337	-0.0084	0.00007056
173	1.4676	1.4679	-0.0003	0.00000009
176	1.4671	1.4759	-0.0088	0.00007744
188	1.5082	1.5073	0.0009	0.00000081
192	1.5090	1.5178	-0.0088	0.00007744
211	1.5609	1.5673	-0.0064	0.00004096
219	1.5849	1.5880	-0.0031	0.00000961
228	1.6071	1.6113	-0.0042	0.00001764
235	1.6308	1.6294	0.0014	0.00000196
252	1.6789	1.6731	0.0058	0.00003364
267	1.7176	1.7114	0.0062	0.00003844
299	1.8001	1.7924	0.0077	0.00005929
314	1.8306	1.8302	0.0004	0.00000016
323	1.8568	1.8527	0.0041	0.00001681

TABLE XXVI (Continued)

T^oC	$(\frac{R}{R_o})_o$	$(\frac{R}{R_o})_c$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]$	$\left[(\frac{R}{R_o})_o - (\frac{R}{R_o})_c\right]^2$
330	1.8712	1.8702	0.0010	0.00000100
338	1.8994	1.8901	0.0093	0.00008649
345	1.9098	1.9075	0.0023	0.00000529
356	1.9433	1.9348	0.0085	0.00007225
368	1.9661	1.9643	0.0018	0.00000324
374	1.9856	1.9791	0.0065	0.00004225
387	2.0193	2.0110	0.0083	0.00006889
388	2.0148	2.0133	0.0015	0.00000225
404	2.0574	2.0523	0.0051	0.00002601
405	2.0556	2.0548	0.0008	0.00000064
422	2.0966	2.0960	0.0006	0.00000036
422	2.1009	2.0960	0.0049	0.00002401
442	2.1441	2.1441	0.0000	0.00000000
445	2.1528	2.1513	0.0015	0.00000225
458	2.1808	2.1823	-0.0015	0.00000225
468	2.2058	2.2061	-0.0003	0.00000009
474	2.2203	2.2203	0.0000	0.00000000
483	2.2392	2.2416	-0.0024	0.00000576
499	2.2776	2.2793	-0.0017	0.00000289
502	2.2838	2.2864	-0.0026	0.00000676
517	2.3155	2.3214	-0.0059	0.00003481
520	2.3251	2.3284	-0.0033	0.00001089
532	2.3504	2.3563	-0.0059	0.00003481
540	2.3693	2.3748	-0.0055	0.00003025
				0.00152902

$$\Omega_{\text{two straight line segments}} = \frac{\Sigma (y_o - y)^2}{n - m} =$$

$$\frac{0.0010255 - 0.00011390}{54 - 3} = 0.00002234.$$

$$\Omega_{\text{smooth curve}} = \frac{\Sigma (y_o - y)^2}{n - m} = \frac{0.0015290}{54 - 2} = 0.00002940.$$

$$\frac{\Omega_{\text{smooth curve}}}{\Omega_{\text{two straight line segments}}} = 1.3.$$

Therefore, two straight line segments gave a better fit to the observed data than one smooth curve of the assumed form by the factor of 1.3.

These results indicate that straight line segments are a better fit than a smooth curve of the assumed form in both these cases. In the case of thorium, however, the precision of Ω is ± 0.00000600 which lends little significance to the factor 1.3. In the case of titanium, the factor is sufficiently large that it cannot be disregarded. However, addition of more terms to the equation for the smooth curve would probably reduce the factor. Furthermore, since the $\frac{\Delta R_x}{\Delta R_{pt}}$ curves give no good indication of discontinuities, it cannot be concluded from these data that these discontinuities are real.

VII. SUMMARY AND CONCLUSIONS

The purpose of this investigation was to study the temperature dependence of electrical resistivity of thorium and titanium and to determine whether or not the slope of the resistance versus temperature curve of these metals exhibit anomalous discontinuities. Iron was also studied in an attempt to reproduce previously reported results on discontinuities in the slope of the resistance versus temperature curve for this metal.

The results of this investigation indicate that the best value for the resistivity of iodide titanium at 20°C is 49.6 microhm-centimeters, and is 167.5 microhm-centimeters at 850°C. The temperature coefficient of electrical resistance from 0° to 100°C was found to be 0.00397. The room temperature resistivity is somewhat higher than the values of 46.7 and 47.5 microhm-centimeters reported in the literature by Jaffee and Campbell and Van Arkel. The slightly higher results obtained in this investigation were probably due to contamination of the metal by minute amounts of oxygen and nitrogen.

The resistivity of electrolytic iron at 20°C was found to be 9.7 microhm-centimeters, and is 105.5 microhm-centimeters at 900°C. The Curie point was observed to be at 756°C, reproducing the result which Burgess and Kellberg obtained for electrolytic iron.

The resistivity of thorium containing 0.03% beryllium, 0.01% aluminum, 0.11% carbon, and < 0.01% nitrogen was found to be 21.7 microhm-centimeters at 20°C. At 965°C, the resistivity of this metal is 64.1 microhm-centimeters. The temperature coefficient of electrical resistance from 0° to 100°C is 0.00277. The resistivity of thorium containing 0.06% beryllium, < 0.01% aluminum, 0.04% carbon, and 0.02% nitrogen was found to be 20.4 microhm-centimeters at 20°C, and is 67.5 microhm-centimeters at 900°C. The temperature coefficient of electrical resistance of this metal from 0° to 100°C was found to be 0.00333. The reason why at elevated temperatures the resistivities of the 0.11% carbon sample should be lower than those of the sample containing only 0.0445% carbon is uncertain.

In addition to inspection of resistance versus temperature plots, two rigorous methods were utilized to study the existence of discontinuities in slope of the resistance versus temperature curves of thorium, titanium, and iron. These methods entailed plotting $\frac{\Delta R_x}{\Delta R_{pt}}$ versus temperature and the application of the least squares method with the closeness of fit criteria to the data.

Visual examination of the resistance data for titanium when plotted on an enlarged scale indicated that the data could best be represented by segments of straight lines and that discontinuities in slope existed. The temperatures of these discontinuities were reporduced to within ± 20°C on separate runs. No obvious discontinuities were observed in the resistance versus temperature curve for iron. Some indication of such discontinuities was observed in the case of thorium.

The graphical method that involved practically simultaneous isothermal measurements of the electrical resistance of both platinum and the metal being studied offered a new approach to the problem. By taking small increments of temperature, $\frac{\Delta R_x}{\Delta R_{pt}}$ versus temperature curves were plotted. The $\frac{\Delta R_x}{\Delta R_{pt}}$ versus temperature curves in all cases were smooth curves, thereby giving no indication of the existence of anomalous discontinuities.

An analytical method, which employed the least squares method and the criteria for closeness of fit both to segments of straight lines represented by equations of the type $\frac{R}{R_o} = a + bt$ and to a smooth curve having as its assumed equation $\frac{R}{R_o} = 1.0000 + at + bt^2$, was applied to the resistance data for titanium and for thorium over the temperature ranges in which discontinuities in slope of resistance versus temperature curves were indicated by visual observations. In the case of thorium over the range 0° to 540°C, two straight line segments gave a better fit to the data than one smooth curve of the assumed form by a factor of 1.3. However, the precision index was such that this factor has little significance. In the case of titanium over the range 0° to 516°C, two straight line segments gave a better fit to the data by a factor of 3.9. This factor is sufficiently large that it cannot be disregarded. However, addition of more terms to the equation for the smooth curve would probably reduce this factor.

The results obtained in this investigation in general do not show the existence of discontinuities in the slope of the resistance versus temperature curves of these metals over the temperature ranges studied, except possibly in the case of titanium. For titanium, visual observation of the plotted data and the analytical method indicate the existence of discontinuities, while the $\frac{\Delta R_{ti}}{\Delta R_{pt}}$ versus temperature curves gave no indication for their existence. Further, the addition of more terms to the equation for the smooth curve would probably reduce the significance of the results obtained by the analytical method.

Further studies on this problem should be conducted. A number of experimental difficulties encountered in this study must be taken into consideration in future investigations. The elimination of temperature gradients in the specimen and between the two specimens is of great importance. Besides using a furnace which at elevated temperatures has temperature gradients as small as possible, a metal sleeve, preferably of titanium or zirconium, should be placed around the specimens inside the furnace tube. Since these metals are both good conductors of heat as well as efficient getters, they would serve to reduce temperature gradients and to remove active residual gases. The use of a silver tube on the outside of the furnace tube would further help to eliminate temperature gradients. Also, the use of small diameter potential and current lead wires will minimize the heat that is conducted away from the specimens. Another major problem to contend with is that of the possible physical changes of the specimen during the run. Crystal growth and the precipitation of impurities, such as oxides and nitrides, in the grain boundaries may give rise to erroneous results. Although these changes are characteristic of the metal, fully annealing the specimens and maintaining a high vacuum will help minimize their effect. Another source of difficulty is the possible presence of metallic vapors due to volatile impurities in the specimens or due to the vapor pressure of the particular metal used. These vapors tend to condense on the current and potential lead-wire-insulators in the colder areas of the furnace and cause shorting between the wires, particularly if the insulating material is porous or at points where the wire extends through an otherwise impervious insulator. Such vapors would also tend to contaminate the platinum specimen. Also, all apparatus should be shielded to prevent any pickup of stray currents.

The precision in measuring $\frac{\Delta R_x}{\Delta R_{pt}}$ should be increased and more terms should be used in the equation for the smooth curve. Taking all these factors into consideration and utilizing the methods employed in this investigation, more conclusive evidence should then be available to determine the reality of anomalous discontinuities in the slope of the resistance versus temperature curves of metals.

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