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A RESISTIVITY STUDY OF HOT SHORT FERROUS ALLOYS

BY

HAROLD ALFRED KOELLING

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A

THESIS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

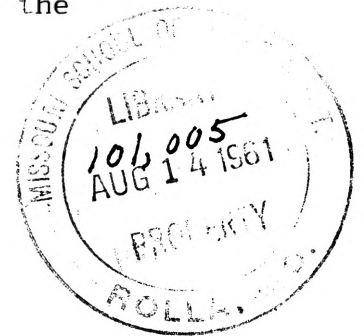
in partial fulfillment of the work required for the

Degree of

MASTER OF SCIENCE, METALLURGICAL MAJOR

Rolla, Missouri

1961



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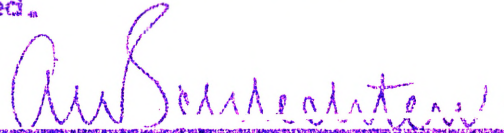
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TABLE OF CONTENTS

	Page
LIST OF ILLUSTRATIONS.....	4
LIST OF TABLES.....	6
I. ABSTRACT.....	7
II. ACKNOWLEDGEMENTS.....	8
III. INTRODUCTION.....	9
1. Statement of Problem.....	9
2. Importance of the Study.....	9
3. Reason for the Study.....	9
IV. REVIEW OF LITERATURE.....	11
V. METHODS OF EVALUATING RESISTIVITY.....	14
1. Deflection Method.....	14
2. Bridge Method.....	15
3. Potentiometer Method.....	15
VI. RED SHORTNESS.....	18
1. Effect of Sulfur.....	18
2. Effect of Copper.....	19
VII. PREPARATION OF SPECIMENS.....	22
VIII. APPARATUS AND PROCEDURE.....	27
1. Apparatus.....	27
2. Combustion Tube Furnace Set Up.....	27
3. Electrical Set Up.....	28
4. Procedure.....	29
IX. EXPERIMENTAL RESULTS.....	35
1. Results of Hot Forging Tests.....	35
2. Results of High Temperature Resistivity Test...	35
3. Evaluation of Resistivity Data.....	36
4. Metallographic Examination.....	38
X. CONCLUSIONS.....	80
SUGGESTIONS FOR FURTHER STUDY.....	81

	Page
BIBLIOGRAPHY.....	82
APPENDIX A.....	84
APPENDIX B.....	85
APPENDIX C.....	86
APPENDIX D.....	87
VITA.....	88

LIST OF ILLUSTRATIONS

Figures	Page
1. The Iron-Sulfur Equilibrium Diagram.....	13
2. Electrical Circuits for Evaluating Resistivity.....	17
3. The Iron-Copper Equilibrium Diagram.....	21
4. The Resistivity Specimen.....	26
5. The Apparatus.....	31
6. The Electrical Set Up.....	32
7. Combustion Tube Set Up.....	33
8. Schematic of Electrical Set Up.....	34
9. Resistivity of Fe versus Temperature.....	42
10. Resistivity of Fe versus Temperature.....	45
11. Resistivity of Fe + .023 S versus Temperature.....	48
12. Resistivity of Fe + .023 S versus Temperature.....	51
13. Resistivity of Fe + .33 Cu versus Temperature.....	54
14. Resistivity of Fe + .33 Cu versus Temperature.....	57
15. Resistivity of Fe + .024 S + .32 Cu versus Temperature.....	60
16. Resistivity of Fe + .024 S + .32 Cu versus Temperature.....	63
17. Resistivity of Armco Iron versus Temperature.....	66
18. Resistivity of Armco Iron versus Temperature.....	69
19. Change in Resistivity per 10 <sup>0</sup> C versus Temperature for Fe.....	70
20. Change in Resistivity per 10 <sup>0</sup> C versus Temperature for Fe + .023 S.....	71
21. Change in Resistivity per 10 <sup>0</sup> C versus Temperature for Fe + .33 Cu.....	72

Figures	Page
22. Change in Resistivity per $10^{\circ}\text{C}$ versus Temperature for Fe + .024 S + .32 Cu.....	73
23. Change in Resistivity per $10^{\circ}\text{C}$ versus Temperature for Armco Iron.....	74
24. Microstructure of Pure Iron.....	75
25. Microstructure of Iron + .023 Sulfur.....	76
26. Microstructure of Iron + .33 Copper.....	77
27. Microstructure of Iron + .024 Sulfur + .32 Copper....	78



LIST OF TABLES

Table	Page
1. ACTUAL CHEMICAL COMPOSITIONS OF ALLOYS INVESTIGATED.....	25
2. FORGING TEMPERATURES FOR CAST ALLOYS.....	25
3. HOT FORGING DATA.....	39
4. DATA SHEET, Specimen 1, Pure Iron.....	40
5. DATA SHEET, Specimen 2, Pure Iron.....	43
6. DATA SHEET, Specimen 3, Iron + .023 Sulfur.....	46
7. DATA SHEET, Specimen 4, Iron + .023 Sulfur.....	49
8. DATA SHEET, Specimen 5, Iron + .33 Copper.....	52
9. DATA SHEET, Specimen 6, Iron + .33 Copper.....	55
10. DATA SHEET, Specimen 7, Iron + .024 Sulfur + .32 Copper.....	58
11. DATA SHEET, Specimen 8, Iron + .024 Sulfur + .32 Copper.....	61
12. DATA SHEET, Specimen 9, Armco Iron.....	64
13. DATA SHEET, Specimen 10, Armco Iron.....	67
14. DIMENSIONS OF SPECIMENS.....	79

## I. ABSTRACT

Resistivity measurements were performed at elevated temperatures on four special ferrous alloys and one commercial iron. The alloys were pure iron, iron with .023 per cent sulfur, iron with .33 copper, iron with .32 copper and .024 sulfur. Other elements were held to the minimum amounts possible in order to determine the actual effects of sulfur and copper. The commercial alloy was Armco Iron.

Measurements of resistivity versus temperature were taken at slow heating rates to determine if the iron sulphide liquid film formed in the grain boundaries of red-short steels had any effect on the electrical resistivity.

## II. ACKNOWLEDGEMENTS

Thanks are due many persons who assisted in various ways to bring about the culmination of this investigation.

Thanks especially to the U.S. Bureau of Mines, Rolla, Missouri, and to its director Mr. J. A. Rowland in particular, for providing the special alloys required for this investigation. Without these alloys this project would not have been possible.

Also thanks are due Dr. D. S. Eppelsheimer who initiated the idea upon which this project was based, acted as advisor, and contributed many thoughtful suggestions throughout the work.

The assistance and contributions of other faculty members and graduate students are also acknowledged.

### III. INTRODUCTION

#### 1. Statement of the Problem.

The purpose of this experimental investigation was (1) to design an apparatus for the measurement of electrical resistivity at elevated temperatures (2) to determine if electrical resistivity measurements can be used to detect the "red shortness" range in steel (3) to determine by electrical resistivity measurements if copper has any effect in causing a steel to be red short.

#### 2. Importance of the Study.

When a red-short steel is worked or forged in the temperature range approximately  $50^{\circ}\text{C}$  to  $150^{\circ}\text{C}$  above the transformation temperature ( $910^{\circ}\text{C}$ ) of iron failure will occur by fracture along the grain boundaries. This failure is not attributable to normal weakening of the grain boundaries at elevated temperatures. Thus this particular steel must be heated to a much higher temperature to successfully accomplish the hot forging operation before cooling into the undesirable temperature range.

Consequently, any knowledge of the "red shortness" range which could lead to a clearer understanding about the nature of the problem would be important in that it could lead to a forging process which might be used in this temperature range.

#### 3. Reason for the Study.

For many years investigators have stated the cause of "red shortness" in steel to be the formation of an iron sulphide liquid film in the grain boundaries when the steel is heated into the temperature range  $950^{\circ}$ - $1100^{\circ}\text{C}$ .

It was thought by this investigator there should be a

substantial change in the electrical conductivity of a steel if a liquid film were formed around the grains.

A survey of literature was performed to determine if any previous investigations had been carried out concerning the resistivity of the metal when the liquid film was formed. No previous references could be found in the survey so it was thought an investigation of resistivity in this temperature range might yield some interesting results.

#### IV. REVIEW OF LITERATURE

Practically all the previous work on resistivity at elevated temperature for ferrous materials has been carried out in two main areas. 1) correlation of resistivity and thermal conductivity of irons and steels and 2) the determination of the absolute resistivity, one of the basic properties of materials. In no case could any investigation be found which dealt with determining resistivity in red-short steels and irons. It has only been in the last fifteen years, the period just after the second World War, in which investigators have attempted to determine resistivity values of steel at temperatures above 900°C.

In 1917, Honda and Simidu<sup>(1)</sup> studied a series of carbon steel up to 900°C. This was probably the first investigation in which thermal conductivity and electrical conductivity were measured at the same time at temperatures above 100°C.

Powell<sup>(2)</sup> in 1934 determined the resistivity of ingot iron at temperatures up to 800°C. The maximum temperature was below the start of the red-short range of ingot irons which in some cases has been reported as low as 860°C.

An extension<sup>(3)</sup> of Powell's work was performed in 1936 on four additional steels. Powell found in both investigations, the tendency of electrical resistivity of irons and steels to converge to a narrow range of values. All of Powell's work was carried out with the intent of correlating thermal and electrical conductivity.

Powell and Hickman<sup>(4)</sup> determined the resistivities of twenty two carbon and alloy steels up to 1300°C in 1946. They

again found the convergence of the resistivities for a majority of the alloys investigated. Noteworthy among their results is a depression in the resistivity curves between  $470^{\circ}$  and  $670^{\circ}$  C. This could be attributed to a defect known in alloy steels as "blue brittleness".

Pallister<sup>(5)</sup> in 1957, performed investigations on low carbon steel up to  $1000^{\circ}$  C by the direct current potentiometer method. His work consisted of 1200 observation points between  $0^{\circ}$  and  $1000^{\circ}$  C. The work was performed on a  $\frac{1}{4}$  inch steel rod 5 cm. long using a current of one ampere. A plot of resistivity shows a smooth curve increasing up to  $800^{\circ}$  C and leveling off above this temperature. The absence of discontinuities in the resistivity curve was noteworthy. This investigator believes the potential drop across the specimen caused by a one ampere current is too low to determine phase changes in steels.

Recent work which is related to the red shortness problem has been performed by Rosenqvist and Dunicz<sup>(6)</sup>, Turkdogan, Ignatowicz, and Pearson<sup>(7)</sup>, and Barloga, Bock, and Parlee<sup>(8)</sup>. Each of these investigations dealt with the problem of sulfur solubility in iron at elevated temperatures. The equilibrium diagrams which these investigators have determined clearly show the red-short temperature range. The composite equilibrium diagram of their investigations is shown in Figure 1.

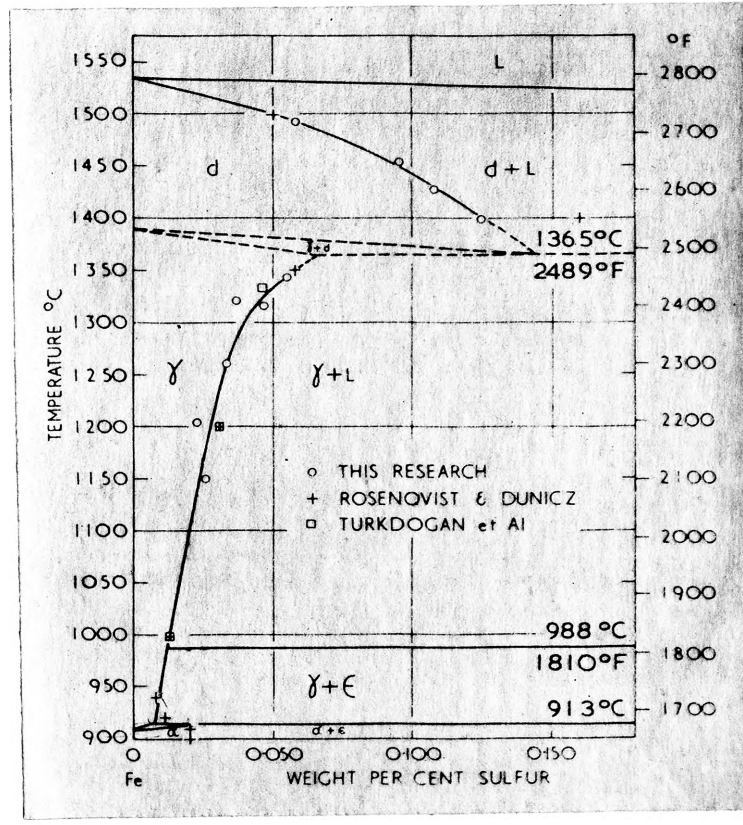


Figure 1  
The Fe-S Equilibrium Diagram (8)



## V. METHODS OF EVALUATING RESISTIVITY

In most metallurgical investigations an absolute determination of the resistivity of a metal is not required. The procedure normally used involves the necessity of comparing resistivity values of samples differing in composition or following changes in the resistivity as a sample undergoes some type of treatment. In this investigation the sample was heated at a slow rate and the change of resistivity with temperature was determined.

Resistivities may be determined by methods involving the use of alternating current or direct current. This discussion will be limited to direct current measuring methods which are the simplest to employ and involve far less rigorous mathematical treatment. Direct current measurements fall into three general classes. They are 1) deflection methods, 2) bridge methods, and 3) potentiometer methods.

### 1. Deflection Method.

By definition the resistance of a specimen is the ratio of the potential difference between its ends to the current passing through it. The simplest and most direct method of determining the resistance would be to measure these two quantities. A simple electrical circuit would consist of a battery, ammeter, and the specimen connected in series. This circuit is shown in Figure 2a. A voltmeter is connected across the specimen. The ratio of the resistance of the voltmeter to the specimen would be high to eliminate excessive current draw. The ratio of the readings on the voltmeter to the ammeter would then give the value of the specimen resistance.

However, methods involving the measurement of instrument deflections have been found to be lacking in accuracy for many metallurgical investigations. For high accuracy a null method such as the bridge method or a d.c. potentiometer method must be used.

## 2. Bridge Method.

The Wheatstone bridge schematic diagram is shown in Figure 2b. If we consider the arrangement of the resistances shown in the circuit it is evident that no current will flow in the galvanometer G if point C and D are at the same potential. If no potential difference exists between C and D the current  $i_1$  must equal the current  $i_2$  and similarly current  $i_3$  equals current  $i_4$ . From these facts it follows the ratio of  $R_1/R_2$  equals  $R_3/R_4$ . Thus if in such a network the ratio of the resistances  $R_3/R_4$  is known and  $R_2$  is a calibrated variable resistance, a specimen may be inserted into the circuit in place of  $R_1$  and its resistance determined when the galvanometer indicates no potential between points C and D, in other words when no current is flowing through the galvanometer.

The bridge methods are not especially applicable to measurements of resistance at elevated temperatures because of the resistance changes in the wire connections to the specimen.

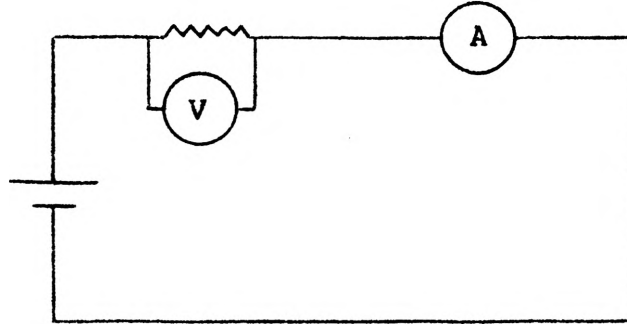
## 3. Potentiometer Method.

Figure 2c gives the schematic diagram for a simple d.c. potentiometer method of determining resistivity. A constant current is provided by the battery which is in series with a standard resistor and the specimen. Essentially the determination

of the specimen resistance is as follows. The potential is determined across the standard resistor. Substituting resistance and potential into Ohms law gives the value of the current flowing in the circuit. The potential drop across the specimen is determined. Again using Ohms law the resistance of the specimen can be determined since the potential and current are known.

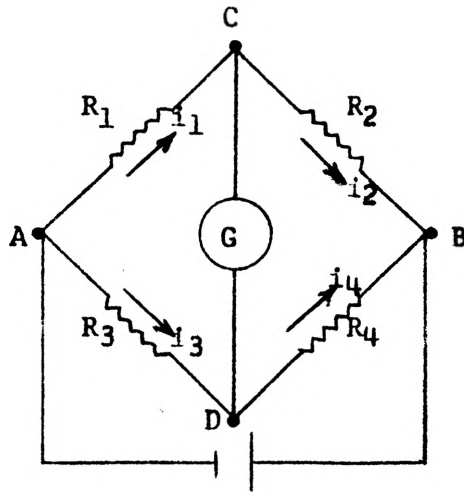
Certain advantages besides accuracy are immediately obvious when employing a potentiometer instead of a voltmeter as in the deflection method which was discussed previously. When the potentiometer is properly adjusted it presents an infinite resistance to the potential difference it is measuring and therefore does not draw any current from the primary circuit. Another advantage is that the resistance of the leads connecting the potentiometer and specimen does not effect the results. This last fact is of primary importance when measuring resistances at elevated temperature. However, potential errors caused by generated thermal voltages do arise when working at elevated temperatures. These errors cannot be eliminated and must be taken into account in the final results.

Methods of Measuring Resistivity



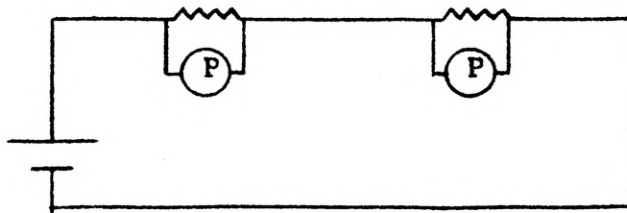
A-ammeter  
V-voltmeter

a) Deflection Method



G-galvanometer

b) Bridge Method



P-potentiometer

c) Potentiometer Method

Figure 2

## VI. RED SHORTNESS

"Red shortness" is a term given to a specific phenomena occurring in some ferrous alloys at elevated temperatures. Sometimes the term hot shortness is used interchangeably. The specific phenomena involve the embrittlement of the iron or steel when worked above red heat. Sulfur has been known for many years to be a major contributor to "red shortness". The role of copper singly or combined with sulfur has been the subject of many investigations.

### 1. Effect of Sulfur.

Sulfur has been known for over a hundred years to cause "red shortness" in irons and steels. Howe<sup>(9)</sup> in 1890, made the first comprehensive review of the effects of elements on "red shortness". He found a considerable amount of variance in reported results which he attributed to poor chemical analysis. Howe probably was one of the first investigators to state that the red-short range could be circumvented by heating to a higher temperature. Howe also found that manganese would counteract "red shortness" by removing the sulfur from the metal. We know today manganese does eliminate "red shortness" but by a different mechanism than proposed by Howe.

The theory of the role of sulfur in red-short steels held today is as follows: When a red-short steel is heated into a certain temperature range, a liquid film is formed around the grain boundaries of the metal. The liquid film is believed to be a low melting Fe-S compound. The Fe-S equilibrium diagram is shown in Figure 1. The melting point of the eutectic composition

is given as  $988^{\circ}\text{C}$ . When the metal is worked in the red-short region, failure occurs by cracking along the grain boundaries. "Red shortness" occurs only in a temperature range. The upper limit of the red-short range is defined in the Fe-S diagram by the phase boundary between gamma iron and gamma iron plus liquid.

One other theory of "red shortness" which never received any acclaim was postulated by Wohrman<sup>(10)</sup>. He believed the solubility of sulfur in gamma iron at the  $A_3$  transformation point was of sufficient magnitude to cause embrittlement to occur. Part of his theory was based upon an investigation by Sauveur and Lee<sup>(11)</sup> in which they discovered the phenomenon of "critical plasticity". Sauveur and Lee found that when a bar of low carbon steel was heated in the center to a temperature above the  $A_3$  point and then twisted, the twisting did not occur at the center but at two points equidistant from the center. At these two points the bar was at the  $A_3$  transformation temperature. Wohrman's explanation for the disappearance of "red shortness" at higher temperatures was that the gamma iron regained its plasticity.

## 2. Effect of Copper.

The interest in copper as a cause of "red shortness" has become important because of the increased copper contents in iron and steel brought about by recycled scrap metal. The scrap metal is continually increasing in copper content due to non-removal during iron and steel refining processes.

Howe<sup>(9)</sup> in his compilation of early data claimed that copper was a cause of "red shortness".

Stead and Evans<sup>(12)</sup> found that copper was of minor significance

in causing "red shortness" although in combination with high sulfur contents there was a possibility of the phenomenon occurring.

Cain<sup>(13)</sup> believed that copper contents of 0.05 to 0.5 percent had no effect in causing "red shortness". He even found at low sulfur concentrations (.01-.02%) a beneficial effect of copper on "red shortness".

The Fe-Cu phase diagram is given in Figure 3. It might be worthwhile to note that in the Fe-Cu system no liquid phases occur in the temperature range normally considered to be red-short. If "red shortness" is attributed to copper alone some other mechanism other than liquid formation must be postulated.

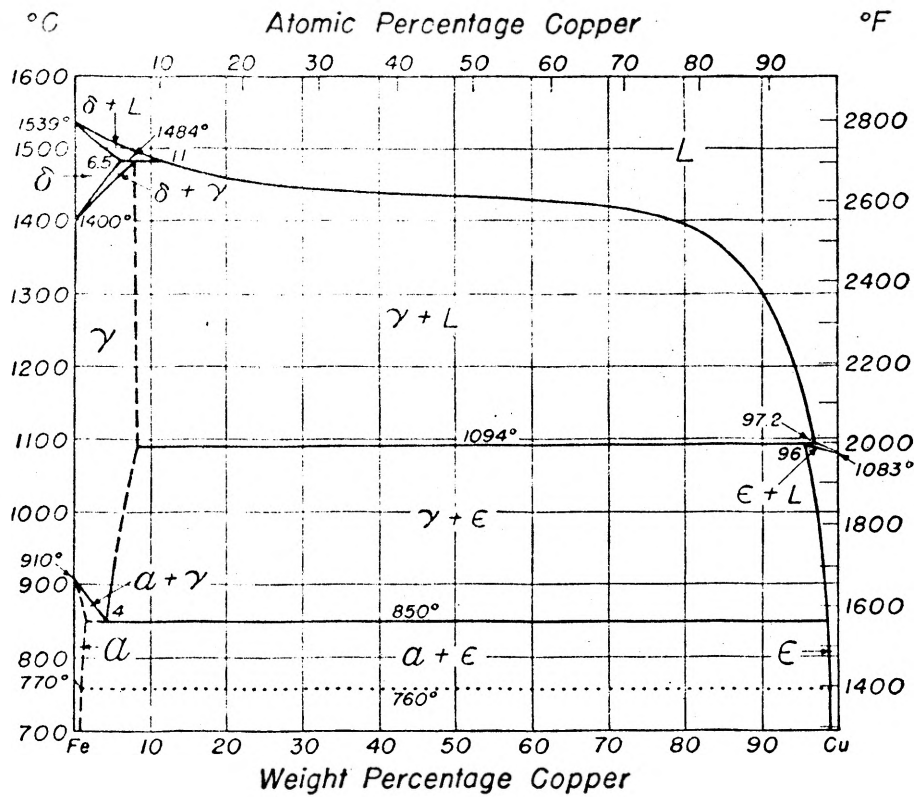


Figure 3  
The Fe-Cu Equilibrium Diagram (14)



## VII. PREPARATION OF SPECIMENS

In order to measure the effect of sulfur and copper, singly or combined, on the resistivity of steel at elevated temperatures, it was necessary to make representative alloys since steels having the desired chemical analysis are not available on the commercial market.

A decision was made to investigate alloys having the following approximate nominal compositions.

1. Iron - 100%
2. Iron plus .025% sulfur
3. Iron plus .25% copper
4. Iron plus .25% Cu plus .025% S.
5. Armco Iron

The actual chemical compositions of the heats are given in Table 1.

The first four alloys listed above were prepared by melting electrolytic iron with the appropriate additions in an Ecco Vacuum Furnace. The chemical composition of the electrolytic iron used as the primary melting stock is listed in appendix A. After melting and thorough mixing of the alloying constituents, the molten metal was cast into ingots  $3\frac{1}{2}$ " X  $3\frac{1}{2}$ " X 14" under helium pressure. The cast ingots were forged into one inch rods. Forging temperatures are listed in Table 2. Prior to forging all ingots were radiographed and found to be free of defects.

During the initial melting operations it was necessary to deoxidize the molten metal with carbon when it was found the ingots were not forgeable due to the presence of oxides. The

carbon remaining after deoxidation was believed to have no effect upon the property being investigated since carbon does not change the solid solubility of sulfur in gamma iron. Carbon does however change the solid solubility of sulfur in delta iron, but the temperature at which delta iron exists has no effect in this investigation. Effects of carbon on the solid solubility of sulfur in iron have been investigated by Barloga, Bock, and Parlee.<sup>(8)</sup>

After forging, two pieces 5" long were cut from the rods and machined into resistivity specimens as shown in Figure 4. Two .042" diameter holes, approximately two inches apart, were drilled through the necked down section of the specimens. A pure iron wire (.032" diameter) was threaded through the drilled holes. Small beads were formed on the ends of the wires by melting with an oxy-acetylene welding torch. Each bead was then peened back into the drilled holes and welded to the specimen. This operation assured good contact of the potential leads to the specimen. Pure iron wire was selected as potential leads to decrease the thermocouple effect of the welded junctions at elevated temperature.

The resistivity specimens were austenitized at 1100°C for twenty four hours in argon gas to alleviate the effects of machining and welding on the microstructure. The exceptionally long length of time at temperature was used to insure two factors, 1) homogeneity of the alloying elements, and 2) a grain size which would not grow larger during testing and thereby be a source of resistivity change.

A  $\frac{1}{4}$ " slice was cut from the one inch bar stock and given a metallographic examination. The procedure for polishing and etching specimens is given in appendix B.

TABLE 1ACTUAL CHEMICAL COMPOSITIONS OF ALLOYS INVESTIGATED

Heat #	Bureau of Mines Heat #	Composition
1	597	.008C; .005S; .005Cu
2	512	.04C; .023S
3	513	.03C; .004S; .33Cu
4	520	.08C; .024S; .32Cu
Armco Iron*	-	.012C; .017Mn; .005P; .025S

\* Typical composition of commercial Armco Ingot Iron. (15)

TABLE 2FORGING TEMPERATURES FOR CAST ALLOYS

Heat #	Bureau of Mines Heat #	Forging Temperature
1	597	1176 <sup>o</sup> C*
2	512	953 <sup>o</sup> C
3	513	1010 <sup>o</sup> C
4	520	954 <sup>o</sup> C

\* Reforged to 3/4" dia at 1093<sup>o</sup>C.

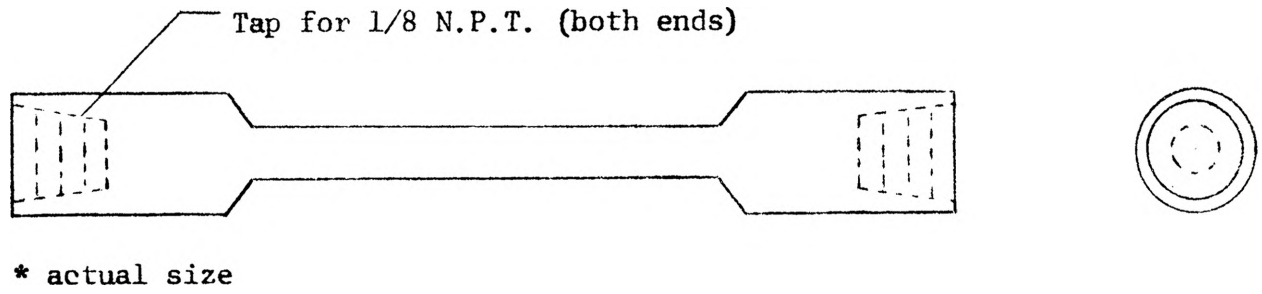


Figure 4  
The Resistivity Specimen

## VIII. APPARATUS AND PROCEDURE

### 1. Apparatus.

The following is a list of the apparatus that was required for this investigation.

- a. Lindberg Furnace, type CF-2
- b. Leeds & Northrup Portable Precision Potentiometer, Model 8662
- c. Bristol Temperature Recorder, type 6PG 560-21
- d. Bottle of Argon
- e. Flowmeter
- f. D.C. Ammeter - Simpson, 0-25 amperes
- g. Standard resistance
- h. Knife switches -3 double pole, double throw
- i. Variable resistance - .84 ohm/50 amperes
- j. Acid storage battery -6 volt

The experimental set up for the apparatus is shown in Figure 5 and 6.

### 2. Combustion Tube Furnace Set Up.

The positioning of the specimen in the combustion tube furnace is shown in Figure 7.

Two 3/8" low carbon steel lead in rods were threaded into the ends of the specimen. The iron potential lead wires which had previously been welded to the specimen were threaded through a two hole insulating tube. The insulating tube was placed parallel to the specimen and secured to the lead in rods. A chrome-alumel thermocouple with its tip at the center of the specimen was attached to the lead in rods. At each end of the specimen insulating fire brick was cut and contoured to the shape of the lead in rods and the inside diameter of the combustion tube. The use of the insulating brick on each end of the specimen helped considerably to cut down the loss of heat to the ends of the combustion tube. The above sub-section was inserted into the

combustion tube and then into the furnace. Rubber stoppers were placed in both ends of the combustion tube. The potential leads and thermocouple wire emerged from opposite ends of the tube through holes in the rubber stopper. The glass tube attachments used for admitting and ejecting the protective gas were inserted through the rubber stoppers. After all connections had been made, the ends of the combustion tube were made gas tight by sealing with Fisher Sealit. The sealant was prevented from melting by copper cooling coils wrapped around the ends of the combustion tube.

### 3. Electrical Set Up.

A photographic close-up of the electrical portion of the apparatus set up is shown in Figure 6. A schematic of the electrical set up is given in Figure 8. A six volt acid storage battery, standard resistor, D-C ammeter, specimen, and switch are connected in series.

The potentiometer is connected across the standard resistor and the specimen. A switch in each of these secondary circuits allows the standard resistor and the specimen resistance to be read individually. Switch #3 is hooked up so the potential across the specimen may be reversed by a simple throw of the switch. This last feature is needed when the thermal emf generated is in the opposite direction from the potential drop across the specimen.

The standard resistor was made by silver soldering two pieces of 18 gage copper wire to a piece of 10 gage copper wire at two points approximately 12 inches apart. The resistance of the standard resistor was assumed to be .001 ohm. This value was

used in all calculations. The standard resistor was immersed in an ice bath and maintained at 32-35°F during the course of each experimental run.

One of the problems that arose in the electrical set up was the increase in resistance of the variable resistor. The increase in resistance was caused by the heating of the resistor when the current was flowing in the circuit. During initial preliminary runs it became necessary to keep the current flowing in the circuit and allowing the variable resistor to come to an equilibrium temperature. This required an amperage draw from the battery of about 6.5 amperes for 6 hours for each run. The battery was recharged slowly for twenty four hours after each run to bring it to optimum operating condition. The current running continuously in the circuit also gave another beneficial result. It eliminated the polarization effect which occurred in the battery when the current was turned on each time.

All potential measuring connections were soldered to prevent excessive contact resistances developing due to corrosion.

#### 4. Procedure.

The argon gas was turned on and adjusted to a flow rate of approximately 50 cc per minute.

The furnace was turned on and heated to a temperature of 500-600°C. The heating rate was adjusted in this temperature range so the heating curve would level off and allow the furnace to come to equilibrium.

About ten minutes before readings were started, the current was started in the circuit by closing switch #1. (See Figure 8)



This allowed the variable resistor to heat up to a maximum value.

The furnace was adjusted to a maximum heating rate of  $3.5^{\circ}\text{C}$  per minute. This heating rate was the average value between  $600$  and  $900^{\circ}\text{C}$ . At  $900^{\circ}\text{C}$  the heating rate began to level off and it became necessary to adjust the maximum power input continually to obtain a heating rate of  $1^{\circ}\text{C}$  per minute.

Readings of resistance were initiated when the temperature of the specimen reached  $600$ - $650^{\circ}\text{C}$ . The readings were made as follows: (See Figure 8)

- Step 1. Switch #2 was closed and the potential drop across the standard resistor was determined.
- Step 2. Switch #2 was opened and switch #3 was closed. The potential drop was determined across the specimen.
- Step 3. Switch #1 was opened and the potential drop across the specimen was determined again. This potential was due to a residual thermal emf caused by uneven heating and thermocouple effects. If the residual emf was in the opposite direction a fourth step was necessary.
- Step 4. Switch #3 was reversed and the thermal emf determined. In this case the emf was added to the specimen IR drop while in step 3 the thermal emf was subtracted from the specimen IR drop.
- Step 5. Switch #3 is opened and switch #1 closed for subsequent readings. The above steps took approximately one minute elapsed time to complete.



Figure 5 - The Apparatus



Figure 6 - The Electrical Set-up

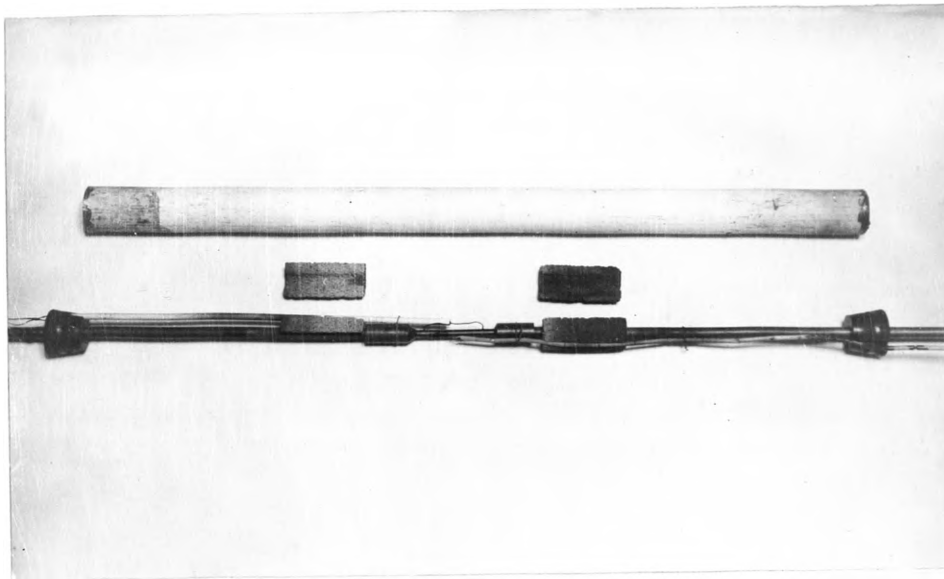


Figure 7 - Combustion Tube Set-up

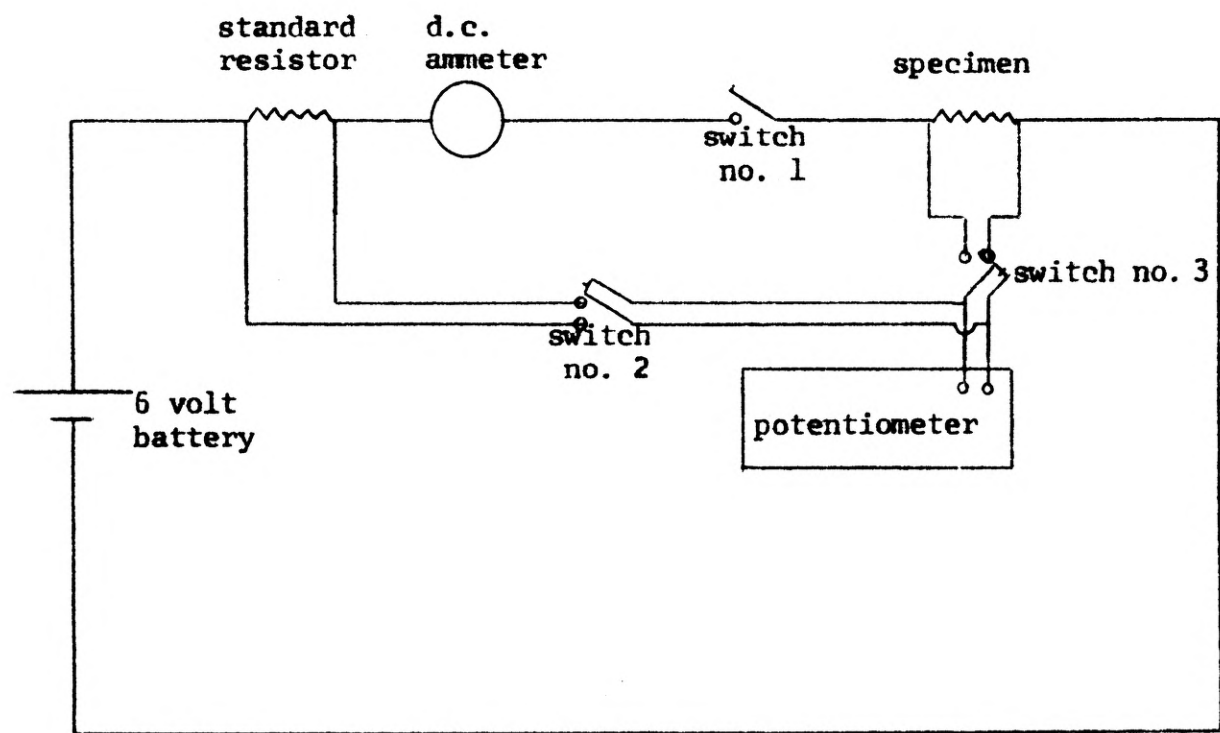


Figure 8

Schematic of Electrical Set Up

## IX. EXPERIMENTAL RESULTS

### 1. Results of Hot Forging Tests.

The results of Chao's work<sup>(16)</sup> are tabulated in Table 3 in order to compare the resistivity measurements with hot forging data. The heats used by Chao in his investigation were the same as these used in this resistivity study.

### 2. Results of High Temperature Resistivity Test.

The results of the high temperature resistivity test are tabulated in Tables 4 through 13 and shown graphically in Figures 9 through 18. Figures 19-23 show a special method of plotting the results to give greater sensitivity. These graphs are made by plotting the change in resistivity per  $10^{\circ}\text{C}$  taken from Figures 9-18. This method of plotting data was employed by Powell and Hickman<sup>(4)</sup>. All graphs and calculations give values of apparent resistivity, that is, the dimensions of each specimen at room temperature are assumed to remain constant over the entire temperature range.

A breakdown of the figures and tables for each material investigated is as follows:

1. Pure iron - Tables 4 and 5  
Figures 9, 10, and 19
2. Pure iron + sulfur - Tables 6 and 7  
Figures 11, 12, and 20
3. Pure iron + copper - Tables 8 and 9  
Figures 13, 14, and 21
4. Pure iron + copper + sulfur - Tables 10 and 11  
Figures 15, 16, and 22
5. Armco Iron - Tables 12 and 13  
Figures 17, 18, and 23

### 3. Evaluation of Resistivity Data.

A tabulation of the dimensions of the specimens is given in Table 14. These values are required in calculating the resistivity. A sample calculation of resistivity is given in appendix C.

Figures 9, 10, and 19 are graphical representations of the results on pure iron. They show a discontinuity occurring in the resistivity curve of specimens 1 and 2 at  $925^{\circ}\text{C}$  and  $955^{\circ}\text{C}$  respectively. The normally accepted value for the alpha to gamma transformation of pure iron is  $910^{\circ}\text{C}$ , a figure determined by a number of investigators.<sup>(17)</sup> The discrepancy between the observed values and the published value has probably been caused by the tip of the thermocouple not being in close proximity to the specimen.

Figures 11, 12, and 20 show the resistivity curves for the iron + .023 sulfur heat. Specimen 3 indicated discontinuities in the resistivity curve at  $900^{\circ}\text{C}$ ,  $975^{\circ}\text{C}$ , and  $1065^{\circ}\text{C}$ . Specimen 4 indicates resistivity changes at  $900^{\circ}\text{C}$ ,  $990^{\circ}\text{C}$ , and  $1100^{\circ}\text{C}$ . If we look at the composite phase boundaries of the Fe-S diagram shown in Figure 1, we see a .023% sulfur material has phase transformations at  $913^{\circ}\text{C}$ ,  $988^{\circ}\text{C}$ , and about  $1090^{\circ}\text{C}$ . The results of this investigation are seen to be in fair agreement with the Fe-S equilibrium diagram. The differences in the values at the upper temperature may be due to minor differences in sulfur composition throughout the forged rods. A very small difference in sulfur content would cause a considerable change in the temperature at which liquid would be present. An effect of this nature would be caused by the extreme slope of the phase boundary between gamma

solid solution and gamma plus liquid. Chao's forging results listed in Table 3 indicate the liquid phase should exist up to  $1280^{\circ}\text{C}$ . This value seems to be abnormally high since it would require a sulfur content of approximately .035% to produce a liquid film at this temperature.

Figures 13, 14, and 21 are the graphical results of the resistivity measurements on the iron-copper alloy. Both curves indicate single discontinuities in the resistivity curve. Specimen 5 showed the change at  $925^{\circ}\text{C}$  and specimen 6 at  $900^{\circ}\text{C}$ . Again this discrepancy in the two values indicates a displacement of the thermocouple from the specimens.

A look at the Fe-Cu diagram (Figure 3) indicates two changes taking place in a .33 Cu alloy as it is heated from  $600^{\circ}\text{C}$  to  $1150^{\circ}\text{C}$ . The two changes would be at  $900^{\circ}\text{C}$  and  $910^{\circ}\text{C}$ . Only one sharp discontinuity was found in the resistivity tests. Because these two temperatures are very close, it was quite possible to miss one of the phase changes since readings were taken at  $10^{\circ}\text{C}$  intervals.

Figures 15, 16, and 22 show the results of the Fe-Cu-S resistivity curves. In both specimens the resistivity curves were essentially smooth over the entire range of temperature. It might be noted in Figures 11 and 12 sulfur increased the resistivity of iron while in Figures 13 and 14 copper decreased the resistivity. Thus a possibility for the lack of resistivity change could be the cancellation of resistivity effects of one element by the other.

The resistivity of Armco Iron is shown in Figures 17, 18, and 23. The resistivity curves of both specimens indicate a phase



change at  $915^{\circ}\text{C}$ . This value and the absence of other discontinuities in the resistivity curve indicate this particular heat of Armco Iron to be free of the Fe-S liquid phase.

#### 4. Metallographic Examination.

Figures 24, 25, 26, and 27 show the results of the metallogical examination. Metallographic procedures are given in Appendix B.

TABLE 3Hot Forging Data (15)

<u>Specimen</u>	<u>Forging Quality</u>
Pure Iron	Good through entire temperature range
Iron + S	Failure occurs between 1000°C - 1280°C
Iron + Cu	Good through entire temperature range
Iron + Cu + S	Failure* occurs between 1000°C - 1280°C

\* The severity of the failure is much less for the Fe-Cu-S specimens than the Fe-S specimens.

TABLE 4  
DATA SHEET

Specimen: (1) Pure iron

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
630	6.095	5.825	-	5.825	955.7	67.6
655	6.020	6.245	-	6.245	1037	73.4
670	6.045	6.650	-	6.650	1100	77.9
685	6.010	7.035	-	7.035	1171	82.9
705	5.135	6.265	-	6.265	1220	86.4
715	5.140	6.550	-.005	6.545	1273	90.1
740	5.155	6.850	-	6.850	1329	94.1
755	5.175	7.130	-.005	7.125	1377	97.5
765	5.165	7.390	-.005	7.385	1430	101.2
780	5.170	7.650	-	7.650	1480	104.8
790	5.150	7.945	-	7.945	1543	109.2
800	5.145	8.080	-	8.080	1570	111.2
810	5.145	8.190	+.015	8.205	1595	112.9
820	5.140	8.255	+.015	8.270	1609	113.9
840	5.145	8.390	+.030	8.420	1637	115.9
855	5.150	8.475	+.040	8.515	1653	117.0
875	5.140	8.545	+.055	8.600	1673	118.4
885	5.135	8.610	+.065	8.675	1689	119.6
900	5.135	8.620	+.075	8.695	1693	119.9
910	5.125	8.655	+.075	8.730	1703	120.6
920	5.140	8.710	+.045	8.755	1703	120.6
925	5.145	8.780	+.020	8.800	1710	121.1

Specimen: (1) Pure iron (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
935	5.140	8.730	+.050	8.780	1708	120.9
945	5.145	8.720	+.065	8.785	1707	120.9
960	5.145	8.735	+.080	8.815	1713	121.3
975	5.130	8.765	+.085	8.850	1725	122.1
990	5.130	8.790	+.100	8.890	1733	122.7
1000	5.135	8.810	+.100	8.910	1735	122.8
1010	5.140	8.840	+.100	8.940	1739	123.1
1020	5.140	8.865	+.100	8.965	1744	123.5
1030	5.135	8.880	+.100	8.980	1749	123.8
1050	5.140	8.905	+.100	9.005	1752	124.0
1070	5.140	8.955	+.100	9.055	1762	124.7
1090	5.130	9.005	+.085	9.090	1772	125.5
1100	5.140	9.070	+.085	9.155	1781	126.1
1110	5.150	9.065	+.100	9.165	1780	126.0
1120	5.150	9.065	+.090	9.155	1778	125.9
1130	5.135	9.070	+.100	9.170	1786	126.4
1140	5.120	9.060	+.100	9.160	1789	126.7
1150	5.140	9.085	+.095	9.180	1786	126.4
1175	5.150	9.075	+.140	9.215	1789	126.7

Figure 9  
Resistivity of Fe versus Temperature  
Specimen 1

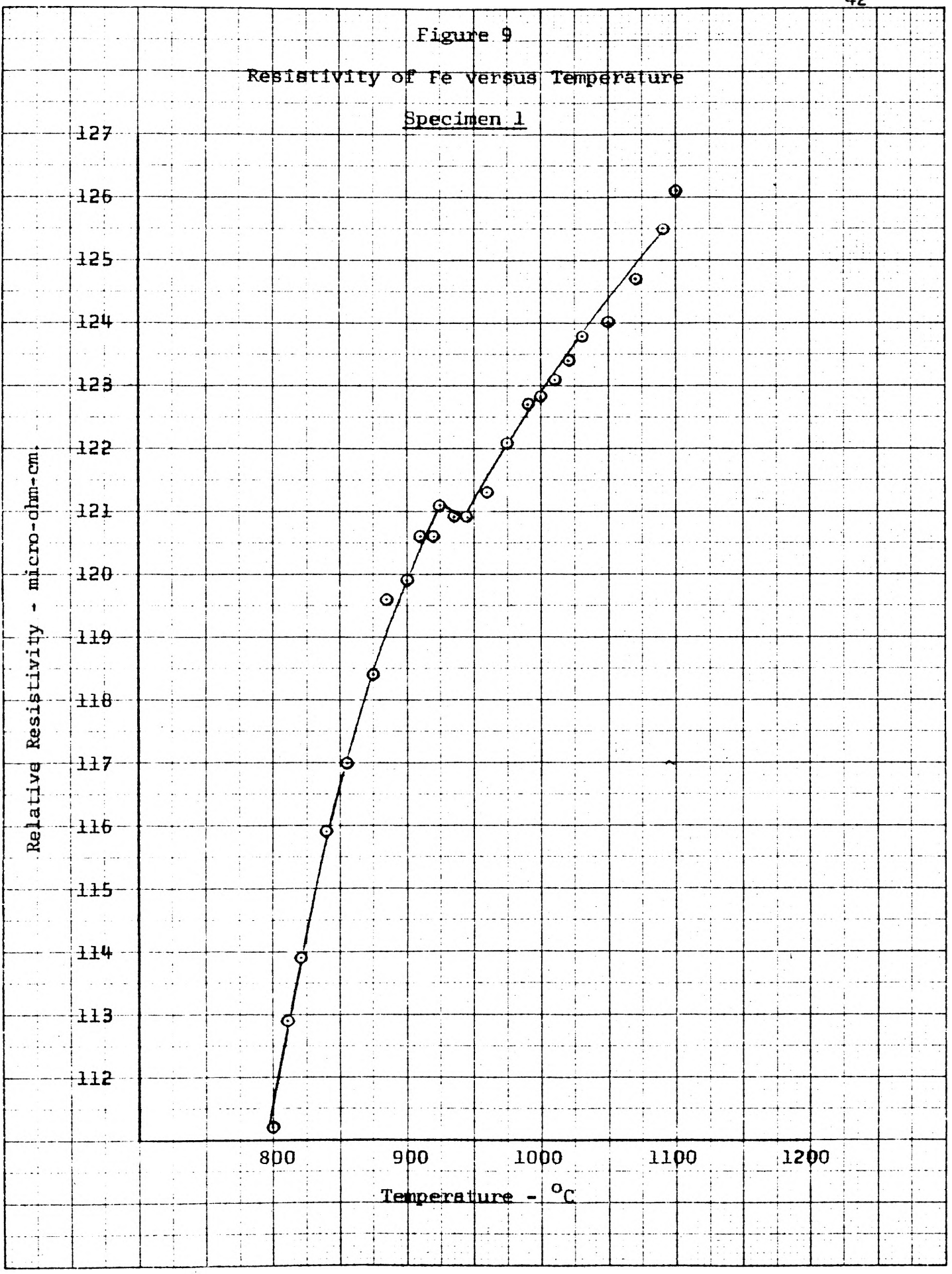


TABLE 5  
DATA SHEET

Specimen: (2) Pure iron

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
620	6.325	6.720	+0.020	6.740	1066	76.3
655	6.330	7.430	-	7.430	1174	84.1
680	6.320	7.860	-.005	7.855	1243	90.0
705	6.295	8.280	-	8.280	1315	94.2
720	6.295	8.640	-.005	8.635	1372	98.2
740	6.290	8.955	-.005	8.950	1423	101.9
750	6.290	9.265	-.005	9.260	1472	105.4
765	6.290	9.570	-.010	9.560	1520	108.8
775	6.290	9.865	-.005	9.860	1568	112.3
795	6.255	10.235	-.005	10.230	1635	117.1
805	6.250	10.375	-.005	10.370	1659	118.8
810	6.255	10.475	-.005	10.470	1674	119.9
825	6.240	10.580	-.005	10.575	1695	121.4
835	6.255	10.695	-.005	10.690	1709	122.4
850	6.250	10.775	-.005	10.770	1723	123.4
860	6.230	10.825	-.005	10.820	1737	124.4
870	6.235	10.890	-	10.890	1747	125.1
875	6.230	10.935	-	10.935	1755	125.7
885	6.235	10.985	-	10.985	1762	126.2
895	6.235	11.020	+0.010	11.030	1769	126.7
900	6.235	11.050	+0.015	11.065	1775	127.1
910	6.240	11.070	+0.015	11.085	1776	127.2

Specimen: (2) Pure iron (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
915	6.230	11.100	+ .020	11.120	1785	127.8
920	6.225	11.125	+ .015	11.140	1790	128.2
925	6.230	11.140	+ .015	11.155	1791	128.2
930	6.230	11.190	-	11.190	1796	128.6
935	6.235	11.235	- .005	11.230	1801	129.0
940	6.225	11.250	- .015	11.235	1805	129.2
945	6.230	11.280	- .015	11.265	1808	129.5
950	6.220	11.285	- .015	11.270	1812	129.8
955	6.225	11.325	- .020	11.305	1816	130.0
960	6.230	11.305	- .020	11.285	1811	129.7
980	6.225	11.335	- .025	11.310	1817	130.1
1000	6.225	11.385	- .015	11.370	1827	130.8
1030	6.215	11.455	- .010	11.435	1840	131.7
1040	6.185	11.435	- .025	11.410	1845	132.1
1065	6.205	11.570	- .065	11.505	1854	132.7
1095	6.215	11.690	- .085	11.605	1867	133.7
1120	6.220	11.745	- .095	11.630	1870	133.9
1140	6.240	11.810	- .095	11.715	1877	134.4
1150	6.260	11.850	- .105	11.745	1876	134.3

Figure 10

Resistivity of Fe versus Temperature

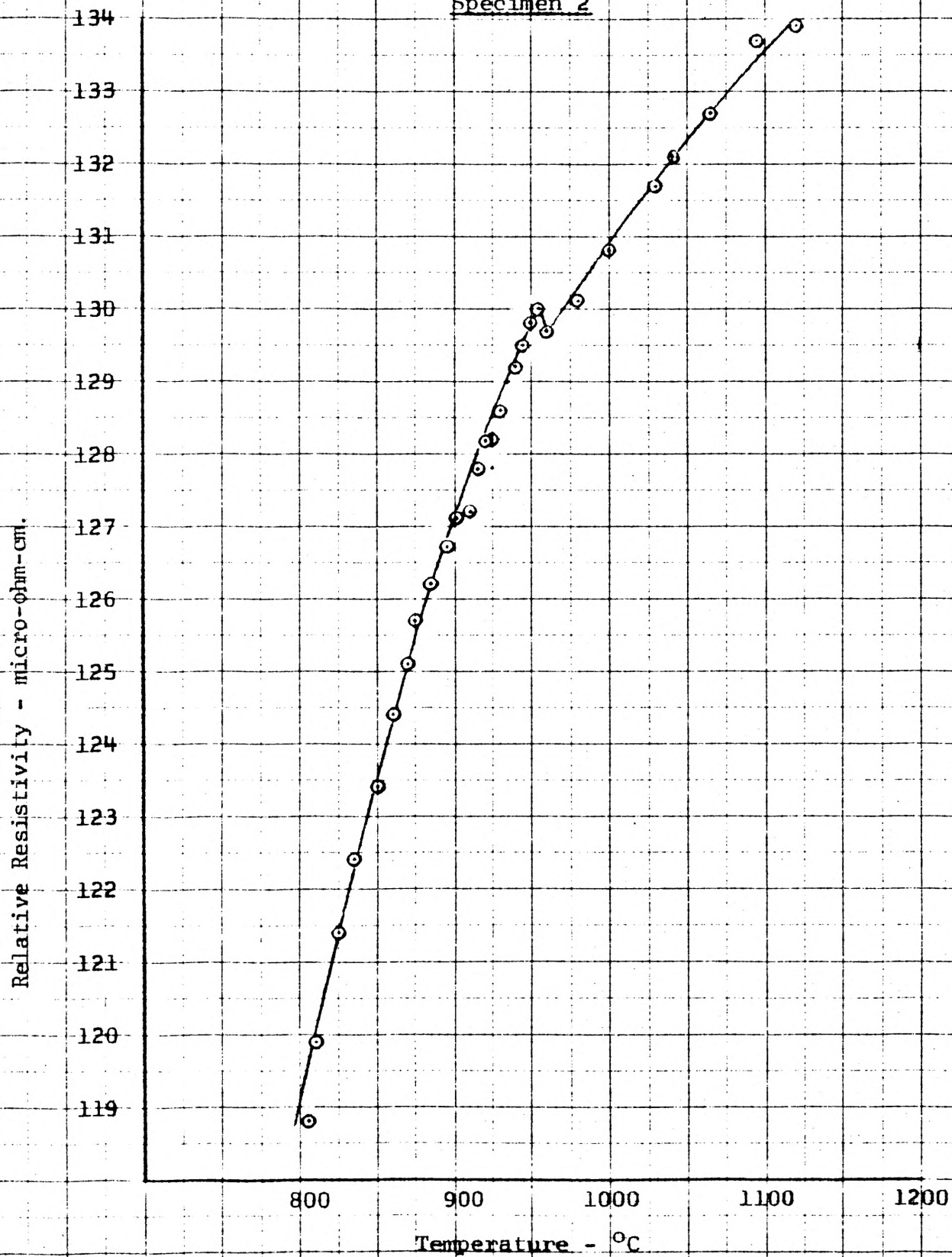
Specimen 2



TABLE 6  
DATA SHEET

Specimen: (3) Pure iron plus .023 sulfur

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
630	6.440	6.780	-	6.780	1053	65.7
650	6.440	7.100	-	7.100	1102	68.8
665	6.440	7.500	-	7.500	1165	72.7
690	6.430	8.110	-	8.110	1261	78.7
715	6.410	8.655	+ .005	8.660	1351	84.3
740	6.395	9.175	+ .005	9.180	1435	89.5
760	6.380	9.570	+ .010	9.580	1502	93.7
770	6.365	9.920	+ .020	9.940	1562	97.5
800	6.600	11.030	+ .030	11.060	1676	104.6
815	6.560	11.135	+ .040	11.175	1704	106.3
835	6.515	11.345	+ .060	11.405	1751	109.3
850	6.515	11.465	+ .085	11.550	1773	110.6
865	6.520	11.570	+ .125	11.695	1794	111.9
880	6.480	11.540	+ .160	11.700	1806	112.7
890	6.480	11.570	+ .190	11.760	1815	113.3
895	6.480	11.575	+ .230	11.805	1822	113.7
900	6.460	11.555	+ .260	11.815	1829	114.1
910	6.460	11.545	+ .285	11.830	1831	114.3
915	6.455	11.535	+ .300	11.835	1833	114.4
920	6.450	11.550	+ .285	11.835	1835	114.5
925	6.445	11.590	+ .250	11.840	1837	114.6
930	6.445	11.640	+ .215	11.855	1839	114.8

Specimen: (3) Pure iron plus .023 sulfur (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
935	6.435	11.660	+.200	11.860	1843	115.0
940	6.430	11.670	+.180	11.850	1843	115.0
945	6.420	11.705	+.100	11.865	1848	115.3
950	6.415	11.720	+.140	11.860	1849	115.4
965	6.380	11.730	+.100	11.830	1854	115.7
970	6.375	11.750	+.080	11.830	1856	115.8
975	6.375	11.790	+.070	11.860	1860	116.1
980	6.375	11.810	+.040	11.850	1859	116.0
990	6.355	11.845	-	11.845	1864	116.3
1000	6.345	11.870	-.030	11.840	1866	116.4
1010	6.350	11.975	-.085	11.890	1872	116.8
1020	6.320	11.975	-.120	11.855	1876	117.1
1030	6.285	11.970	-.150	11.820	1881	117.4
1040	6.265	11.955	-.140	11.815	1886	117.7
1055	6.205	11.910	-.170	11.740	1892	118.1
1060	6.190	11.925	-.175	11.750	1898	118.4
1070	6.130	11.845	-.190	11.635	1898	118.4
1080	6.070	11.780	-.200	11.580	1908	119.1
1100	6.010	11.730	-.190	11.540	1920	119.8
1120	5.970	11.760	-.240	11.520	1930	120.4
1140	5.870	11.660	-.235	11.425	1946	121.4

Figure 11  
Resistivity of Fe + .023 S versus Temperature  
Specimen 3

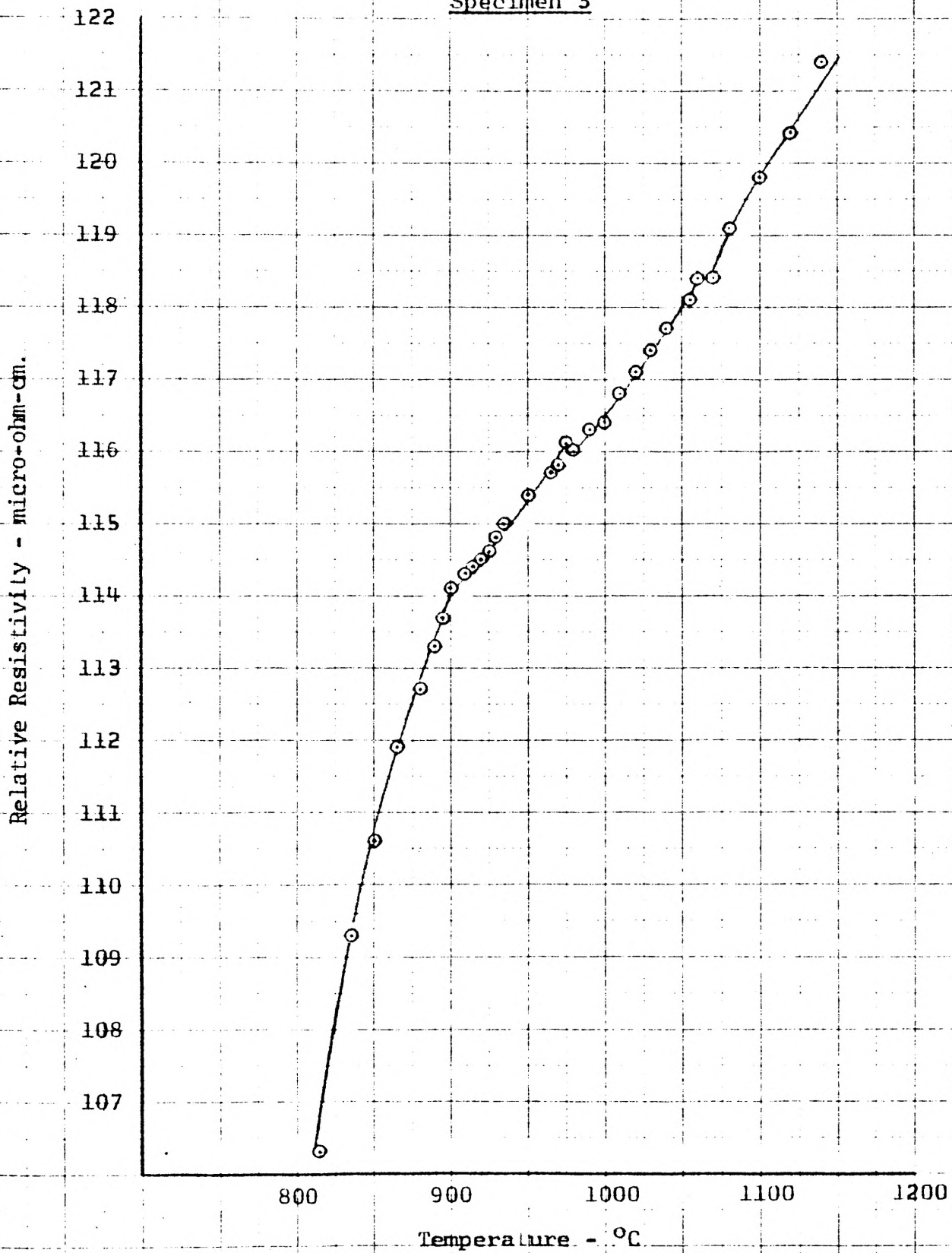


TABLE 7  
DATA SHEET

Specimen: (4) Pure iron plus .023 sulfur

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
615	6.680	7.435	-	7.435	1113	66.8
630	6.640	7.720	-	7.720	1163	69.8
650	6.610	8.235	-	8.235	1246	74.8
680	6.595	8.740	+0.010	8.750	1327	79.6
700	6.590	9.260	+0.035	9.295	1410	84.6
725	6.580	9.840	+0.020	9.860	1498	89.9
750	6.560	10.405	+0.005	10.410	1587	95.2
775	6.510	11.020	-.025	10.995	1689	101.3
800	6.660	12.240	-.310	11.930	1791	107.5
825	6.645	12.680	-.440	12.240	1842	110.5
850	6.630	12.955	-.515	12.440	1876	112.6
860	6.625	13.040	-.530	12.510	1888	113.3
870	6.630	13.125	-.545	12.580	1897	113.8
890	6.615	13.205	-.530	12.675	1916	115.0
900	6.605	13.235	-.515	12.720	1926	115.6
910	6.595	13.215	-.480	12.735	1931	115.9
920	6.585	13.145	-.410	12.735	1934	116.0
930	6.575	13.020	-.270	12.750	1939	116.3
940	6.565	12.990	-.220	12.770	1945	116.7
950	6.550	12.940	-.170	12.770	1950	117.0
960	6.540	12.910	-.125	12.785	1955	117.3
975	6.530	12.925	-.100	12.825	1964	117.8

Specimen: (4) Pure iron plus .023 sulfur (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
980	6.525	12.930	-.080	12.850	1969	118.1
990	6.510	12.950	-.050	12.900	1982	118.9
1000	6.495	12.915	-.055	12.860	1980	118.8
1010	6.465	12.935	-.065	12.870	1991	119.5
1020	6.445	12.960	-.070	12.890	2000	120.0
1035	6.420	13.000	-.085	12.915	2012	120.7
1040	6.400	13.000	-.100	12.900	2016	121.0
1050	6.385	13.045	-.120	12.925	2024	121.4
1060	6.365	13.070	-.140	12.930	2031	121.9
1075	6.350	13.130	-.155	12.975	2043	122.6
1080	6.330	13.155	-.185	12.970	2049	122.9
1090	6.320	13.190	-.190	13.000	2057	123.4
1100	6.315	13.285	-.200	13.085	2072	124.3
1115	6.310	13.285	-.200	13.085	2074	124.4
1125	6.310	13.300	-.205	13.095	2075	124.5
1150	6.295	13.330	-.215	13.115	2083	125.0

Figure 12

Resistivity of Fe + .02% S versus Temperature

Specimen 4

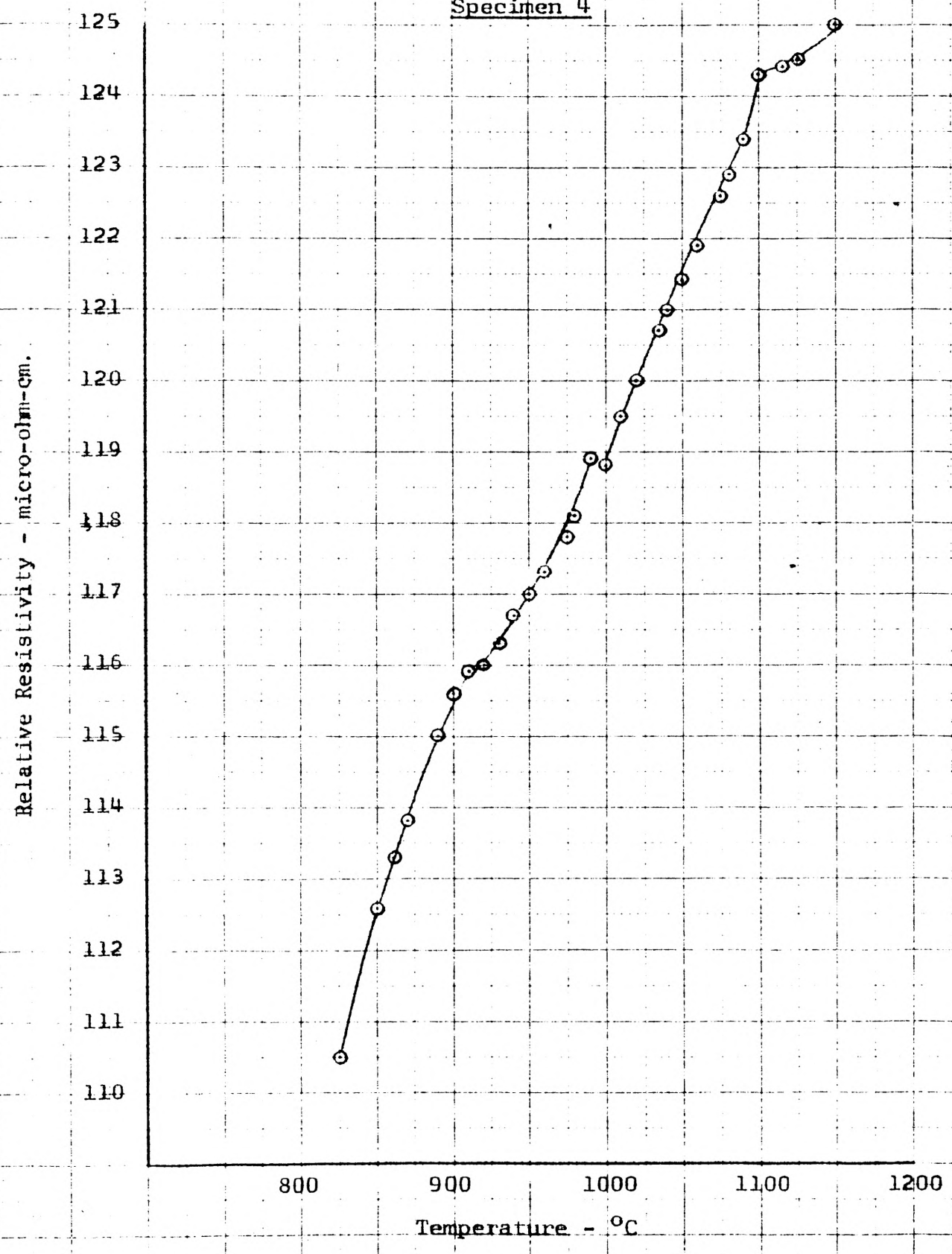


TABLE 8  
DATA SHEET

Specimen: (5) Pure iron plus .33 copper

temp. oC	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
620	6.680	7.690	+.010	7.700	1153	69.6
645	6.695	8.175	+.015	8.190	1223	73.9
665	6.695	8.565	+.015	8.580	1282	77.4
690	6.700	8.970	+.020	8.950	1336	80.7
715	6.700	9.600	+.020	9.620	1436	86.7
735	6.705	10.160	+.015	10.175	1518	91.7
755	6.705	10.570	+.010	10.580	1580	95.4
770	6.705	10.950	+.015	10.965	1635	98.8
785	6.705	11.370	+.015	11.385	1698	102.6
795	6.705	11.575	+.015	11.580	1727	104.3
810	6.700	11.725	+.020	11.745	1733	104.7
825	6.700	11.900	+.025	11.925	1780	107.5
840	6.700	12.030	+.035	12.065	1801	108.8
850	6.700	12.120	+.045	12.165	1816	109.7
860	6.705	12.205	+.050	12.255	1828	110.4
870	6.695	12.245	+.065	12.310	1839	111.1
880	6.695	12.285	+.080	12.365	1847	111.6
890	6.695	12.300	+.095	12.375	1851	111.8
895	6.700	12.310	+.110	12.420	1854	112.0
900	6.695	12.330	+.135	12.465	1862	112.5
910	6.700	12.330	+.165	12.495	1865	112.6
915	6.690	12.305	+.185	12.490	1867	112.8

Specimen: (5) Pure iron plus .33 copper (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
920	6.690	12.305	+.205	12.510	1870	112.9
930	6.695	12.300	+.220	12.520	1870	112.9
935	6.690	12.315	+.225	12.540	1874	113.2
940	6.685	12.340	+.220	12.560	1879	113.5
945	6.690	12.370	+.205	12.575	1880	113.6
950	6.680	12.385	+.200	12.585	1884	113.8
970	6.690	12.510	+.160	12.670	1894	114.4
985	6.685	12.590	+.130	12.720	1903	114.9
1000	6.695	12.675	+.110	12.785	1910	115.4
1010	6.675	12.730	+.080	12.810	1919	115.9
1025	6.685	12.830	+.030	12.860	1924	116.2
1035	6.685	12.890	-	12.890	1928	116.5
1050	6.680	12.910	-	12.910	1933	116.8
1060	6.675	12.965	-.020	12.945	1939	117.1
1080	6.675	12.990	-.020	12.970	1943	117.4
1100	6.675	13.025	-.015	13.010	1949	117.7
1130	6.675	13.095	-.015	13.080	1960	118.4
1150	6.660	13.155	-.015	13.140	1973	119.2
1170	6.660	13.175	-.015	13.160	1976	119.4
1185	6.665	13.220	-.015	13.205	1981	119.7



Figure 13

Resistivity of Fe + .33 Cu versus Temperature

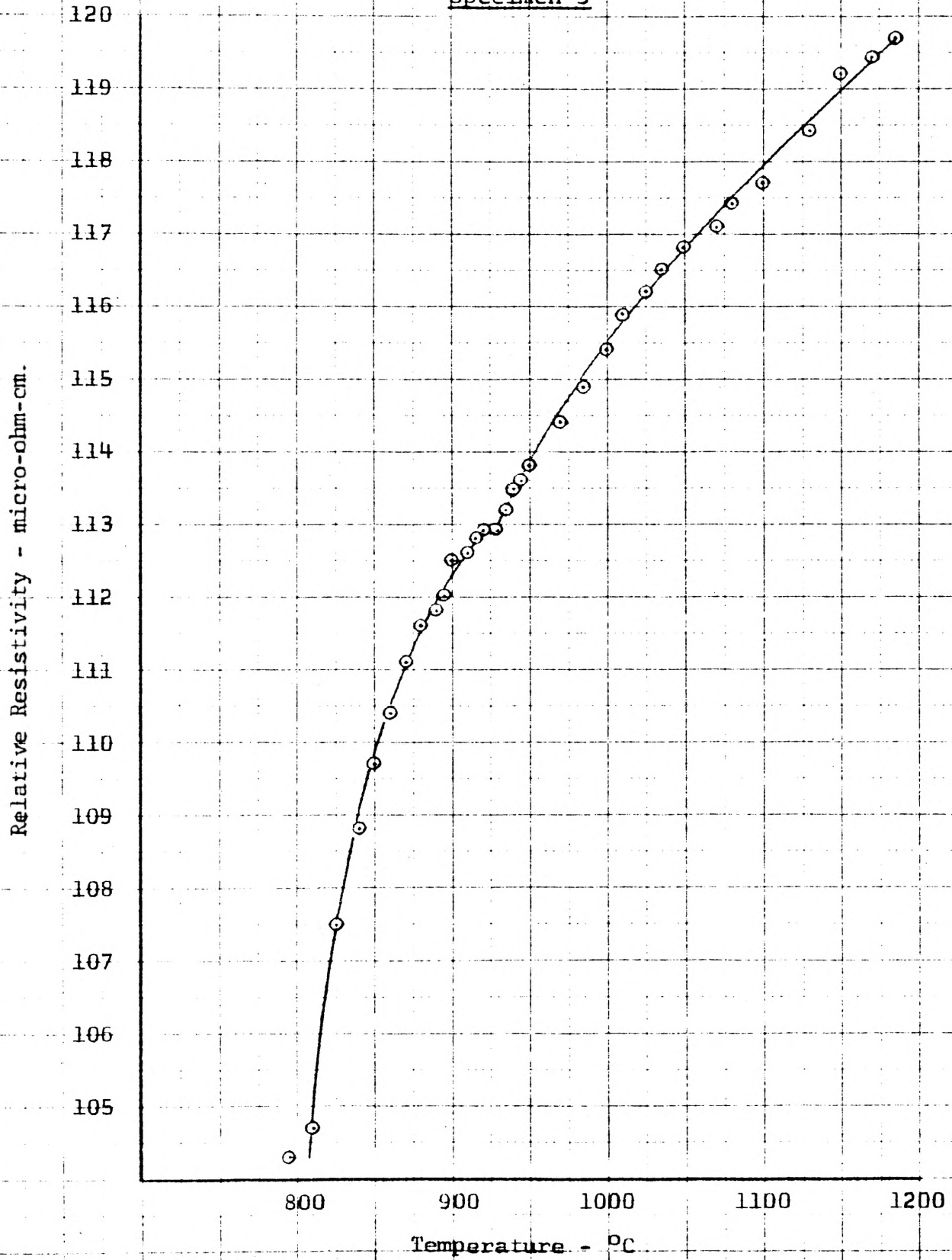
Specimen 5

TABLE 9  
DATA SHEET

Specimen: (6) Pure iron plus .33 copper

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
610	6.645	7.305	-.010	7.295	1098	64.7
630	6.645	7.730	-.010	7.720	1162	68.4
650	6.620	8.160	-.010	8.150	1231	72.5
670	6.630	8.550	+.035	8.585	1295	76.3
695	6.630	9.035	+.010	9.045	1364	80.3
720	6.620	9.620	+.030	9.650	1458	85.9
740	6.620	10.035	+.020	10.055	1519	89.5
760	6.600	10.525	+.020	10.545	1598	94.1
775	6.600	10.980	+.030	11.010	1668	98.2
790	6.590	11.355	+.025	11.380	1727	101.7
805	6.590	11.565	-	11.565	1755	103.4
825	6.580	11.750	-.005	11.745	1785	105.1
850	6.570	11.920	-	11.920	1814	106.8
865	6.565	12.000	-.005	11.995	1827	107.6
870	6.560	12.035	-.005	12.030	1834	108.0
875	6.555	12.055	-.005	12.050	1838	108.3
880	6.555	12.065	-.005	12.060	1840	108.4
890	6.555	12.090	-.005	12.085	1844	108.6
895	6.540	12.110	-.005	12.105	1851	109.0
900	6.540	12.125	-	12.125	1854	109.2
910	6.535	12.130	-.015	12.115	1854	109.2
920	6.525	12.145	-.025	12.120	1857	109.4

Specimen: (6) Pure iron plus copper (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
930	6.515	12.150	-.025	12.125	1861	109.6
940	6.500	12.125	-.005	12.120	1865	109.8
950	6.490	12.150	-.015	12.135	1870	110.1
960	6.490	12.155	-.005	12.150	1872	110.3
975	6.480	12.165	-.005	12.160	1877	110.6
980	6.465	12.160	-.010	12.150	1879	110.7
990	6.465	12.175	-.010	12.165	1882	110.8
1000	6.460	12.180	-.015	12.175	1885	111.0
1015	6.450	12.210	-.025	12.185	1889	111.3
1025	6.450	12.215	-.020	12.195	1891	111.4
1030	6.450	12.235	-.025	12.210	1893	111.5
1040	6.445	12.240	-.020	12.220	1896	111.7
1050	6.440	12.255	-.020	12.235	1900	111.9
1065	6.440	12.280	-.030	12.250	1902	112.0
1075	6.435	12.295	-.035	12.260	1905	112.2
1090	6.435	12.305	-.025	12.280	1908	112.4
1100	6.425	12.300	-.020	12.280	1911	112.6
1125	6.430	12.330	-.015	12.315	1915	112.8
1150	6.425	12.345	-.010	12.335	1920	113.1
1175	6.425	12.365	-.010	12.355	1923	113.3

Figure 14

Resistivity of Fe + .33 Cu versus Temperature

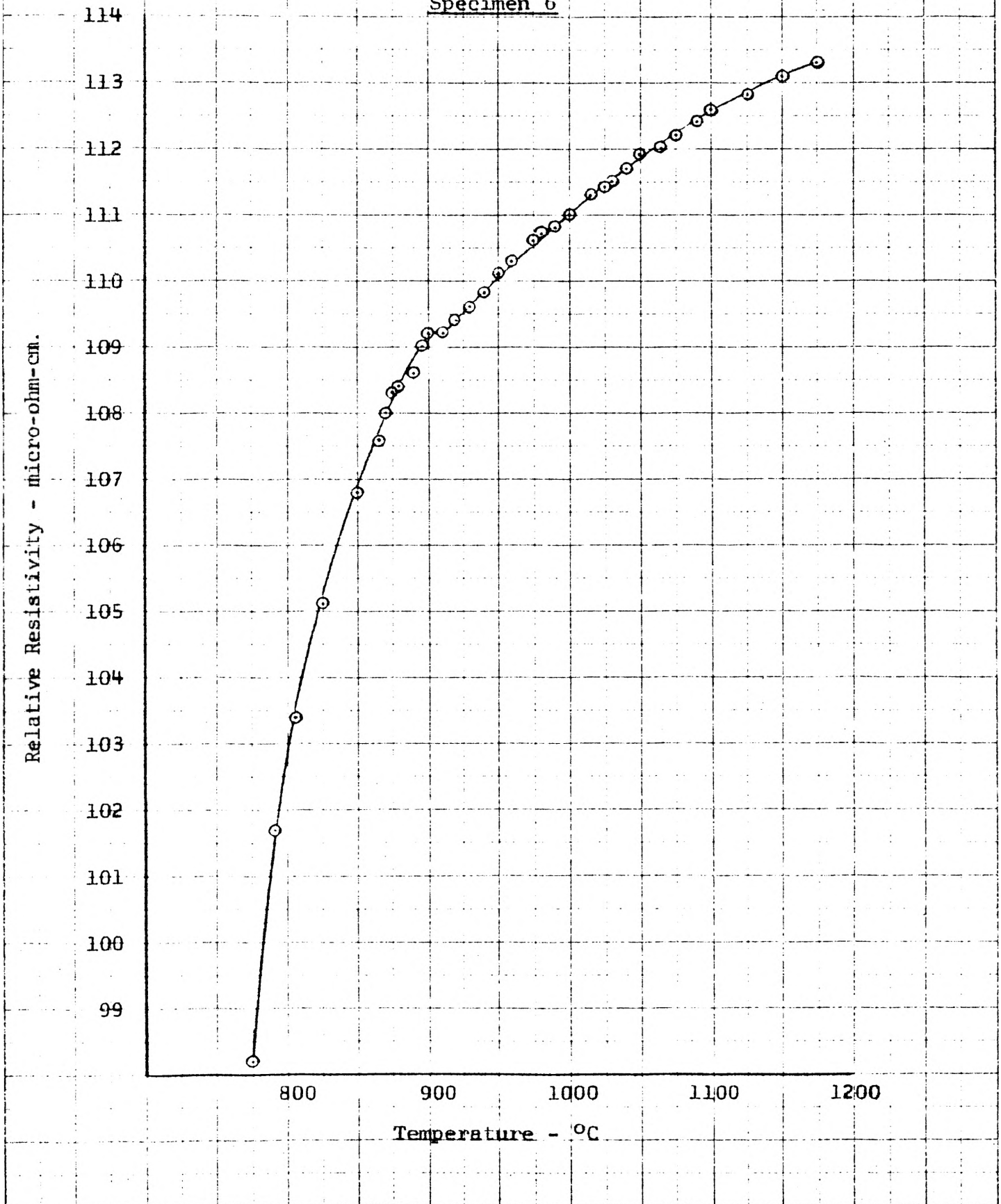
Specimen 6

TABLE 10  
DATA SHEET

Specimen: (7) Pure iron + .024 S + .32 Cu

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
630	6.830	9.170	-	9.170	1343	80.6
645	6.790	9.630	-	9.630	1418	85.1
670	6.800	10.030	-.020	10.010	1472	88.3
730	6.745	11.450	-.010	11.440	1696	101.8
755	6.715	12.115	-	12.115	1804	108.2
770	6.690	12.640	-	12.640	1889	113.3
780	6.650	13.030	-	13.030	1959	117.5
810	6.800	14.300	-	14.300	2103	126.2
820	6.800	14.450	-	14.450	2125	127.5
830	6.790	14.635	-	14.635	2155	129.3
840	6.770	14.750	-	14.750	2179	130.7
855	6.765	14.865	-	14.865	2197	131.8
870	6.755	15.020	-	15.020	2224	133.4
880	6.735	15.070	-.010	15.060	2236	134.2
890	6.715	15.130	-.010	15.110	2250	135.0
900	6.715	15.200	-.025	15.175	2260	135.6
910	6.690	15.190	-.015	15.175	2268	136.1
920	6.675	15.220	-.020	15.200	2277	136.6
930	6.655	15.225	-.040	15.185	2282	136.9
940	6.640	15.230	-.050	15.180	2286	137.2
950	6.650	15.320	-.065	15.255	2294	137.6
960	6.550	15.200	-.115	15.085	2303	138.2

Specimen: (7) Pure iron + .024 S + .32 Cu (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
970	6.515	15.175	-.150	15.025	2306	138.4
980	6.480	15.130	-.150	14.980	2312	138.7
990	6.430	15.060	-.150	14.910	2319	139.1
1005	6.390	15.020	-.160	14.860	2326	139.6
1020	6.340	14.950	-.155	14.795	2334	140.0
1040	6.240	14.710	-.100	14.610	2341	140.5
1065	6.720	15.820	-.040	15.780	2348	140.9
1080	6.665	15.690	-	15.690	2354	141.2
1100	6.645	15.665	+.010	15.675	2359	141.5
1125	6.620	15.650	+.010	15.660	2366	142.0
1150	6.540	15.500	-	15.500	2370	142.2
1175	6.450	15.320	-	15.320	2375	142.5

Figure 15

Resistivity of Fe + .024 S + .32 Cu versus Temperature

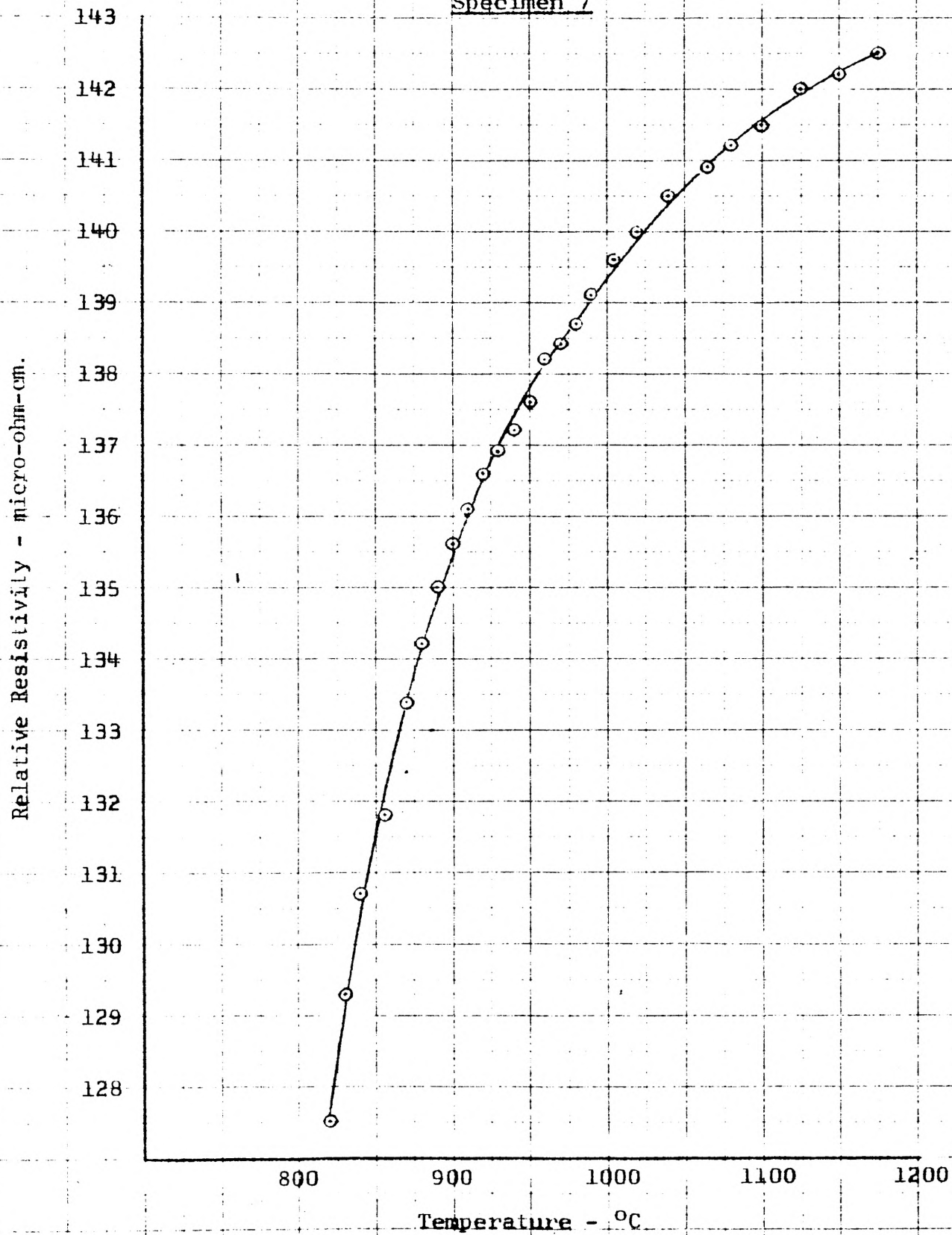
Specimen 7

TABLE 11  
DATA SHEET

Specimen: (8) Pure iron + .024 S + .32 Cu

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
675	6.730	7.845	-	7.845	1166	70.6
690	6.725	8.360	-	8.360	1243	75.3
710	6.725	8.740	-	8.740	1300	78.8
730	6.720	10.275	-.005	10.270	1528	92.6
750	6.710	10.990	-.005	10.985	1637	99.2
775	6.690	11.435	-.010	11.425	1708	103.5
790	6.680	12.060	-.015	12.045	1803	109.2
805	6.680	12.960	-.030	12.930	1936	117.3
825	6.675	13.110	-.040	13.070	1958	118.6
835	6.665	13.345	-.030	13.315	1998	121.0
850	6.655	13.490	-.025	13.465	2023	122.6
860	6.650	13.595	-.025	13.570	2041	123.6
875	6.650	13.740	-.020	13.720	2063	125.0
890	6.640	13.840	-.010	13.830	2083	126.2
900	6.620	13.875	-.020	13.855	2093	126.8
910	6.610	13.920	-.030	13.890	2101	127.3
920	6.600	13.935	-.010	13.925	2110	127.8
930	6.590	13.950	-.010	13.940	2115	128.1
940	6.585	13.985	-.030	13.955	2119	128.4
950	6.580	14.035	-.040	13.995	2127	128.9
960	6.560	14.070	-.060	14.010	2136	129.4
970	6.540	14.055	-.040	14.015	2143	129.8



Specimen: (8) Pure iron + .024 S + .32 Cu (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
980	6.535	14.085	-.040	14.045	2149	130.2
990	6.525	14.080	-.030	14.050	2153	130.4
1000	6.510	14.060	-.030	14.030	2155	130.6
1015	6.500	14.075	-.015	14.060	2163	131.0
1025	6.495	14.085	-.005	14.080	2168	131.3
1040	6.475	14.075	-.005	14.070	2173	131.6
1060	6.470	14.100	-.010	14.090	2178	131.9
1075	6.460	14.125	-.025	14.100	2183	132.3
1090	6.450	14.120	-.010	14.110	2188	132.6
1100	6.430	14.105	-.015	14.090	2191	132.7
1125	6.430	14.140	-.020	14.120	2196	133.0
1150	6.425	14.145	-.010	14.135	2200	133.3
1170	6.425	14.170	-.005	14.165	2205	133.6

Figure 16

Resistivity of Fe + .024 S + .32 Cu versus Temperature

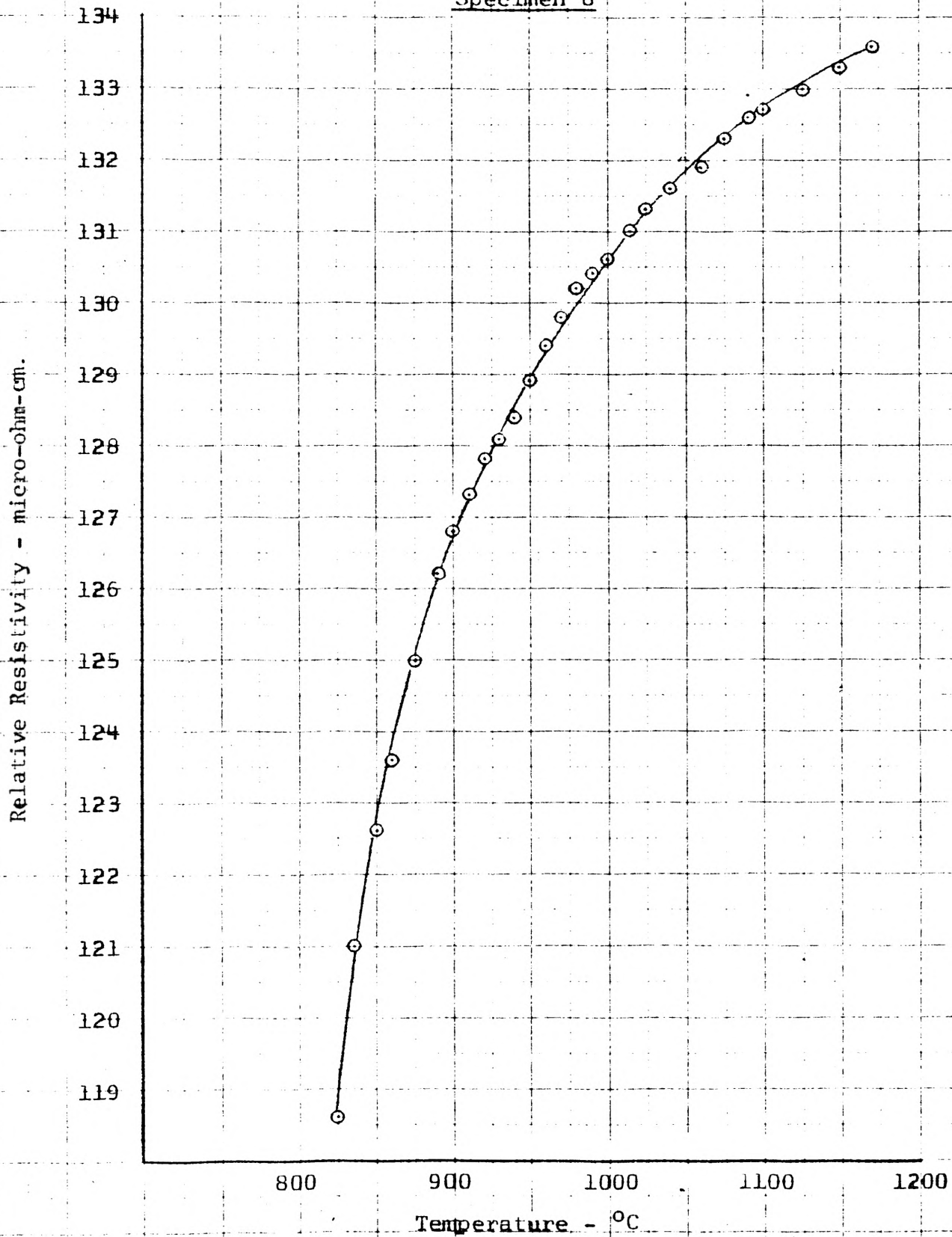
Specimen 8

TABLE 12  
DATA SHEET

Specimen: (9) Armco Iron

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
645	8.220	8.135	-	8.135	990	69.6
690	8.210	9.065	-	9.065	1104	77.6
720	8.200	9.900	-	9.900	1207	84.9
750	8.190	10.635	-.005	10.635	1298	91.3
775	8.180	11.215	-.005	11.210	1370	96.4
790	8.160	11.810	-	11.810	1447	101.8
810	6.850	10.185	-	10.185	1487	104.6
830	8.150	12.380	-	12.380	1519	106.8
840	8.275	12.745	-	12.745	1540	108.3
860	8.285	12.900	+.015	12.915	1559	109.6
870	8.290	13.020	+.025	13.045	1574	110.7
880	8.290	13.060	+.040	13.100	1580	111.1
890	8.265	13.085	+.050	13.135	1589	111.8
900	8.280	13.140	+.070	13.210	1595	112.2
910	8.285	13.170	+.090	13.260	1600	112.5
915	8.290	13.285	+.100	13.385	1615	113.6
925	8.295	13.340	+.090	13.430	1619	113.9
930	8.295	13.375	+.065	13.440	1620	113.9
940	8.295	13.415	+.060	13.475	1624	114.2
945	8.290	13.450	+.055	13.505	1629	114.6
950	8.290	13.470	+.060	13.530	1632	114.8
955	8.290	13.495	+.060	13.555	1635	115.0

Specimen: (9) Armco Iron (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
970	8.290	13.545	+0.060	13.605	1641	115.4
980	8.290	13.600	+0.055	13.655	1647	115.8
990	8.290	13.615	+0.065	13.680	1650	116.0
1000	8.265	13.600	+0.070	13.670	1654	116.3
1015	8.265	13.660	+0.075	13.735	1662	116.9
1025	8.270	13.715	+0.080	13.795	1668	117.3
1040	8.185	13.640	+0.090	13.730	1677	117.9
1055	8.195	13.705	+0.085	13.790	1683	118.4
1070	8.195	13.750	+0.080	13.830	1688	118.7
1080	8.180	13.770	+0.060	13.830	1691	118.9
1090	8.165	13.810	+0.040	13.850	1696	119.3
1100	8.150	13.810	+0.030	13.840	1698	119.4
1115	8.145	13.825	+0.030	13.855	1701	119.6
1130	8.095	13.765	+0.025	13.790	1704	119.8
1150	8.050	13.760	+0.005	13.765	1710	120.3
1165	8.050	13.795	-	13.795	1714	120.5

Figure 17

Resistivity of Armco Iron versus Temperature

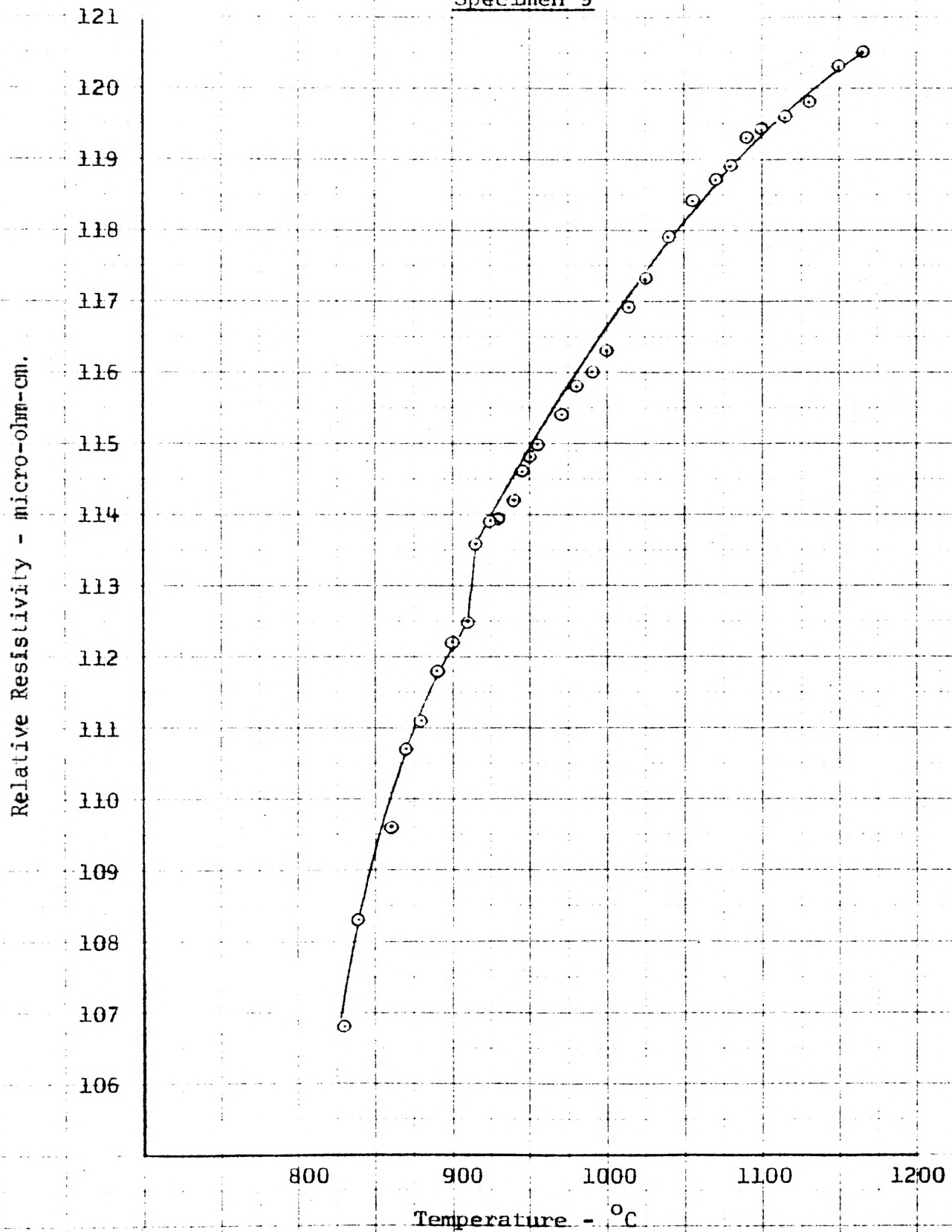
Specimen 9

TABLE 13  
DATA SHEET

Specimen: (10) Armco Iron

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
610	6.430	5.900	+ .030	5.930	922	64.8
630	6.430	6.325	+ .030	6.355	988	69.5
650	6.420	6.700	+ .050	6.750	1051	73.9
670	6.420	7.035	+ .060	7.095	1105	77.7
685	6.410	7.345	+ .055	7.400	1154	81.2
700	6.410	7.630	+ .050	7.680	1198	84.3
715	6.405	7.890	+ .050	7.940	1240	87.2
735	6.400	8.255	+ .045	8.300	1297	91.2
760	6.390	8.690	+ .030	8.720	1365	96.0
780	6.385	9.265	- .033	9.230	1446	101.7
795	6.375	9.680	- .120	9.560	1500	105.5
810	6.370	9.955	- .240	9.715	1525	107.3
830	6.360	10.330	- .485	9.845	1548	108.9
855	6.360	10.810	- .750	10.060	1582	111.3
870	6.350	10.985	- .870	10.115	1593	112.0
880	6.345	11.070	- .925	10.145	1599	112.5
890	6.335	11.150	- .965	10.185	1608	113.1
900	6.335	11.190	- .975	10.215	1612	113.4
905	6.325	11.200	- .960	10.240	1619	113.9
910	6.325	11.200	- .940	10.260	1622	114.1
915	6.325	11.180	- .910	10.270	1624	114.2
920	6.320	11.115	- .845	10.270	1625	114.3

Specimen: (10) Armco Iron (continued)

temp. °C	I amps	spec. mv	residual mv	E true mv	R micro- ohm	micro- ohm-cm
930	6.315	11.040	-.755	10.285	1629	114.6
940	6.300	10.900	-.705	10.285	1633	114.8
955	6.290	10.900	-.585	10.315	1640	115.3
965	6.290	10.885	-.550	10.335	1643	115.6
980	6.285	10.865	-.500	10.365	1649	116.0
990	6.290	10.745	-.355	10.390	1652	116.2
1000	6.280	10.650	-.250	10.400	1656	116.5
1025	6.270	10.640	-.190	10.450	1667	117.2
1040	6.270	10.585	-.100	10.485	1672	117.6
1065	6.265	10.545	-.010	10.535	1682	118.3
1080	6.265	10.560	-.005	10.555	1685	118.5
1100	6.245	10.580	-.030	10.550	1689	118.8

Figure 18  
Resistivity of Armco Iron versus Temperature  
Specimen 10

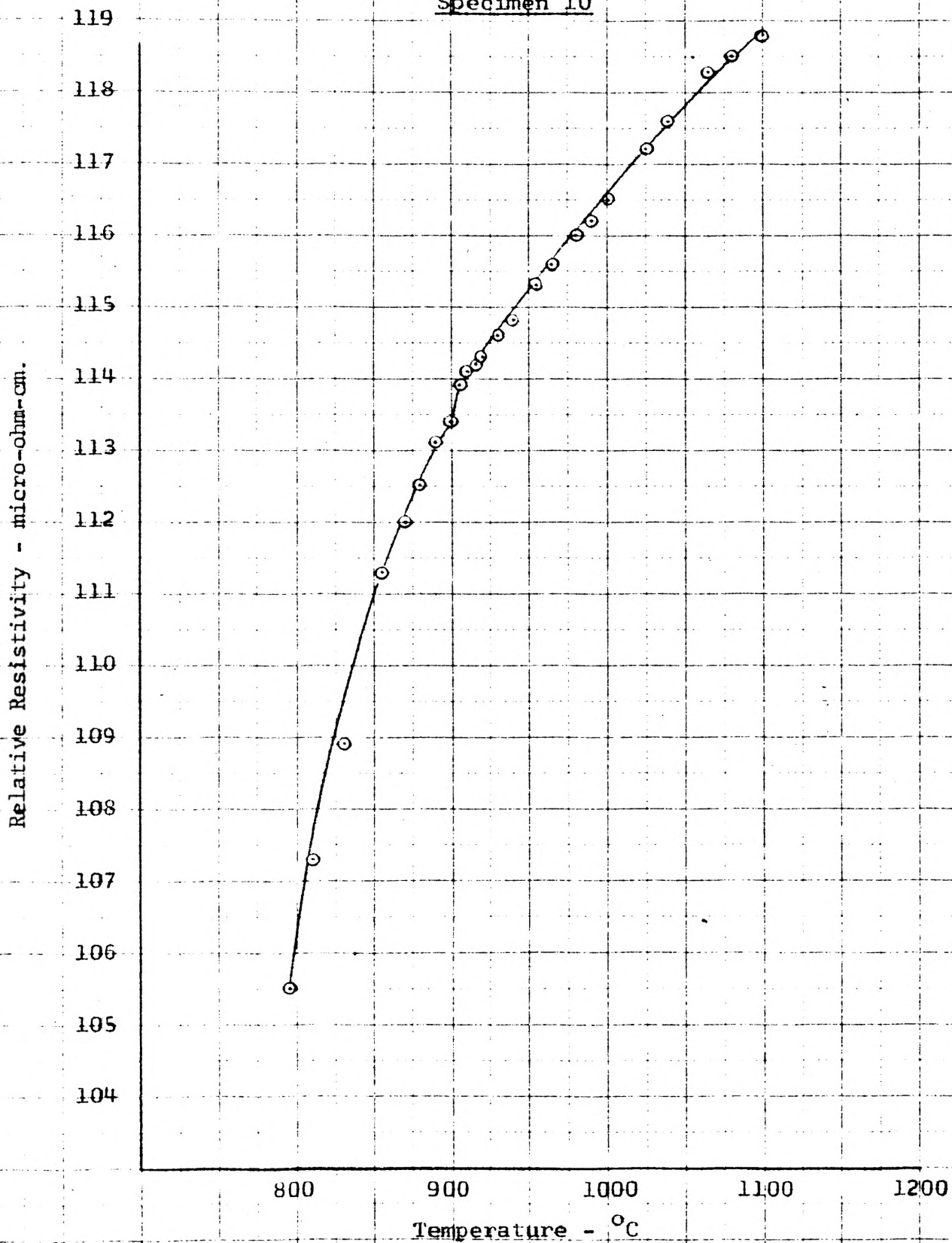




Figure 19  
Change in Resistivity per  $10^{\circ}\text{C}$  versus Temperature  
for Fe

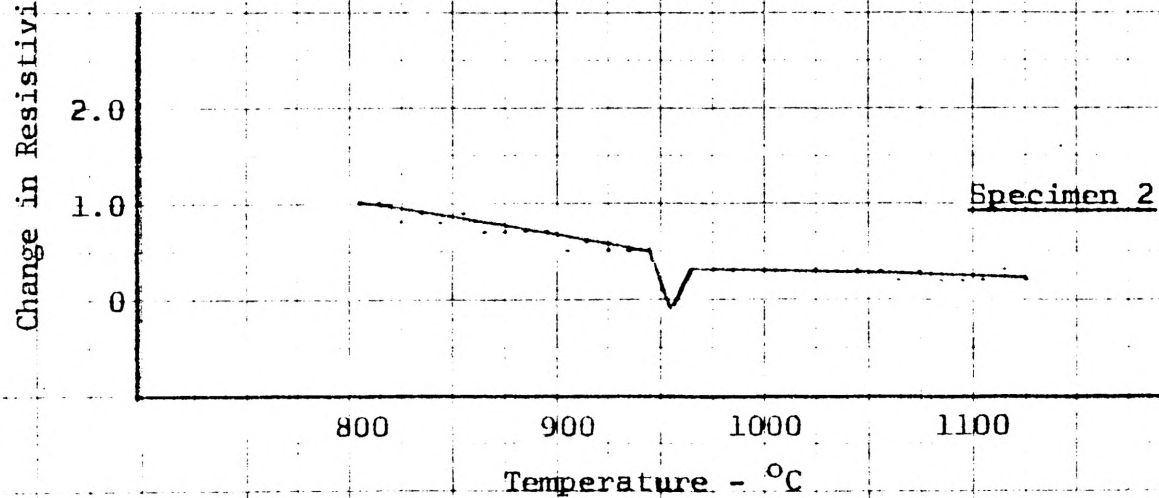
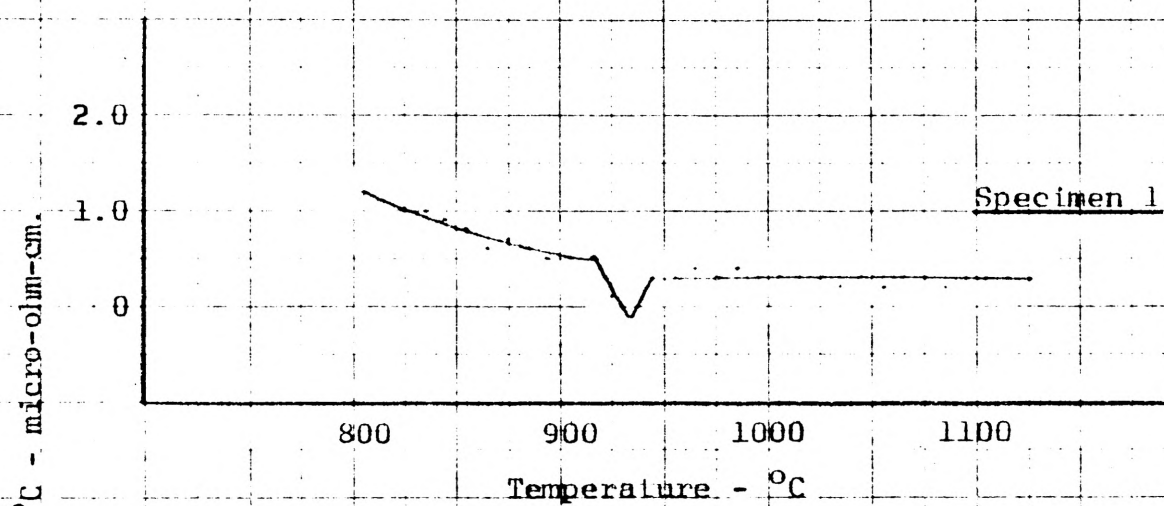


Figure 20  
Change in Resistivity per 10°C versus Temperature  
for Fe + .023 S

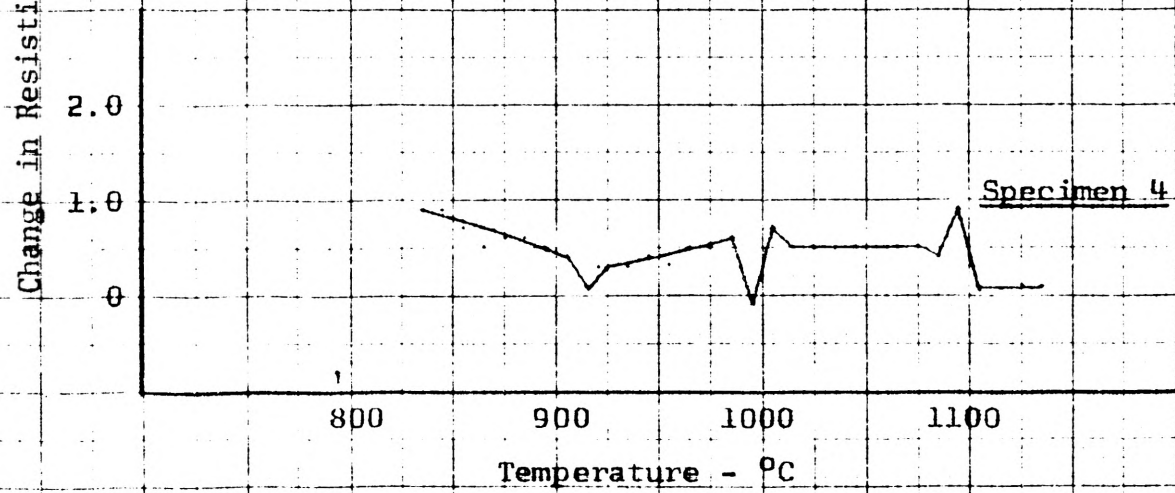
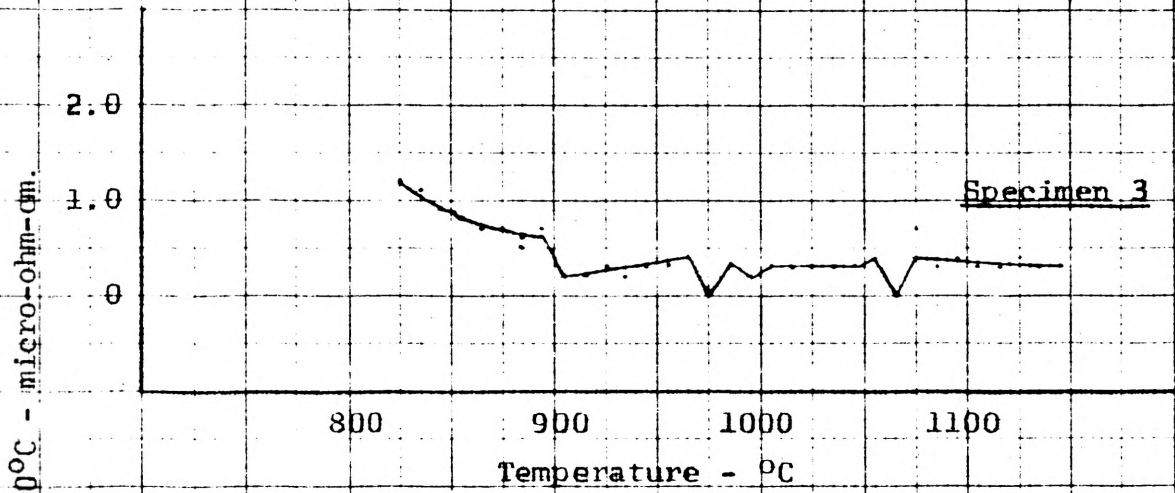


Figure 21

Change in Resistivity per  $10^{\circ}\text{C}$  versus Temperature  
for Fe + .33 Cu

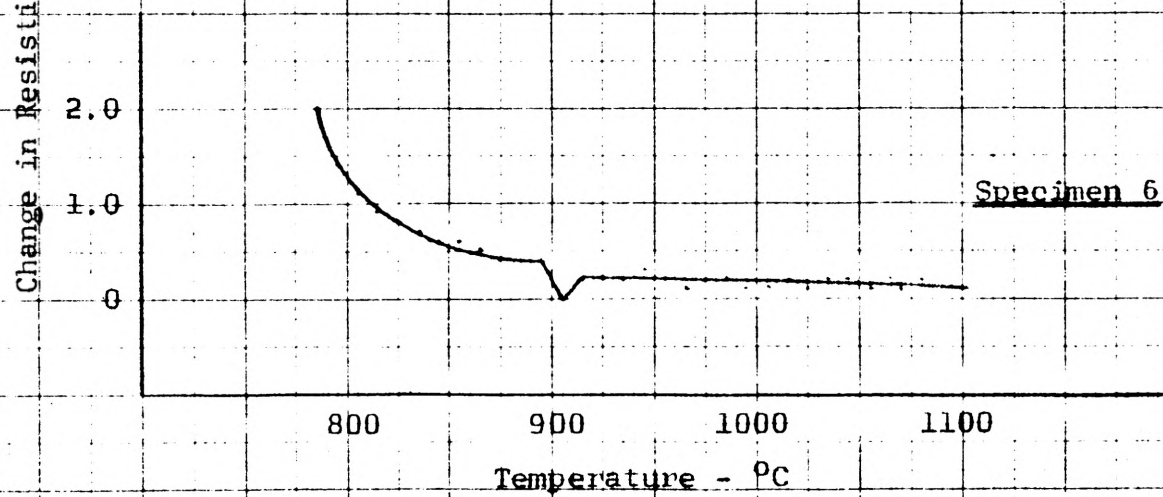
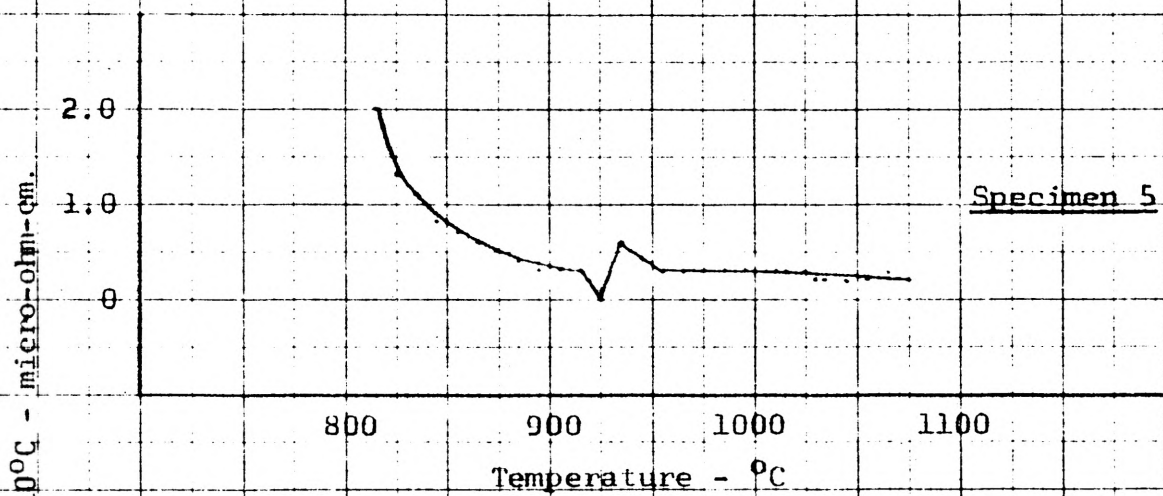


Figure 22

Change in Resistivity per 10°C versus Temperature  
for Fe + .024 S + .32 Cu

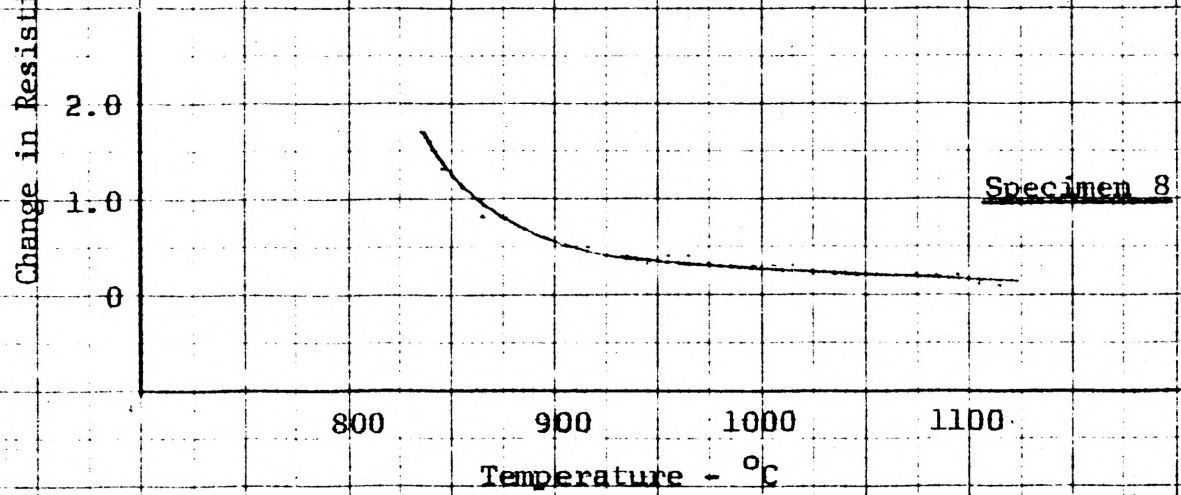
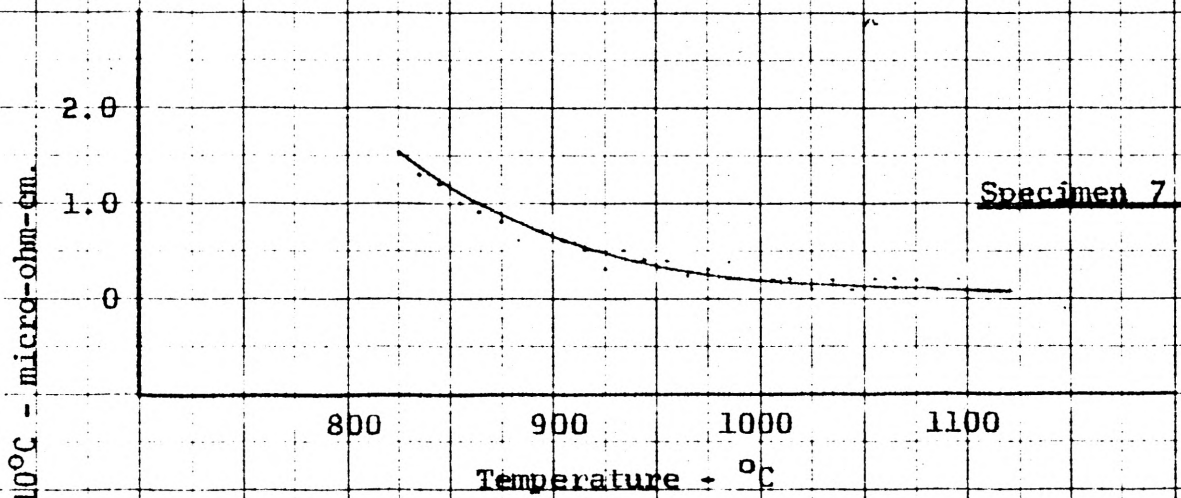
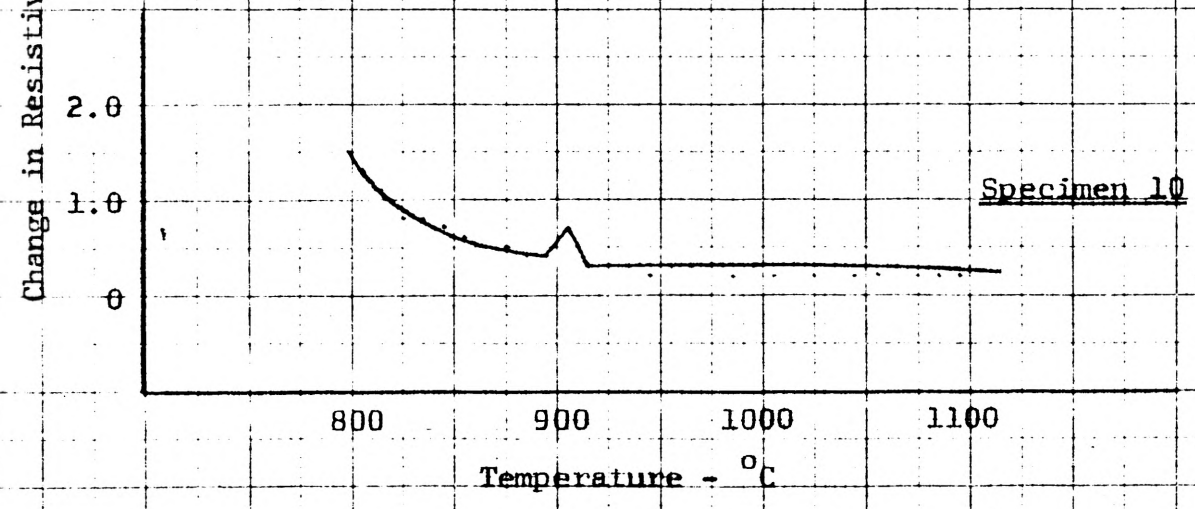
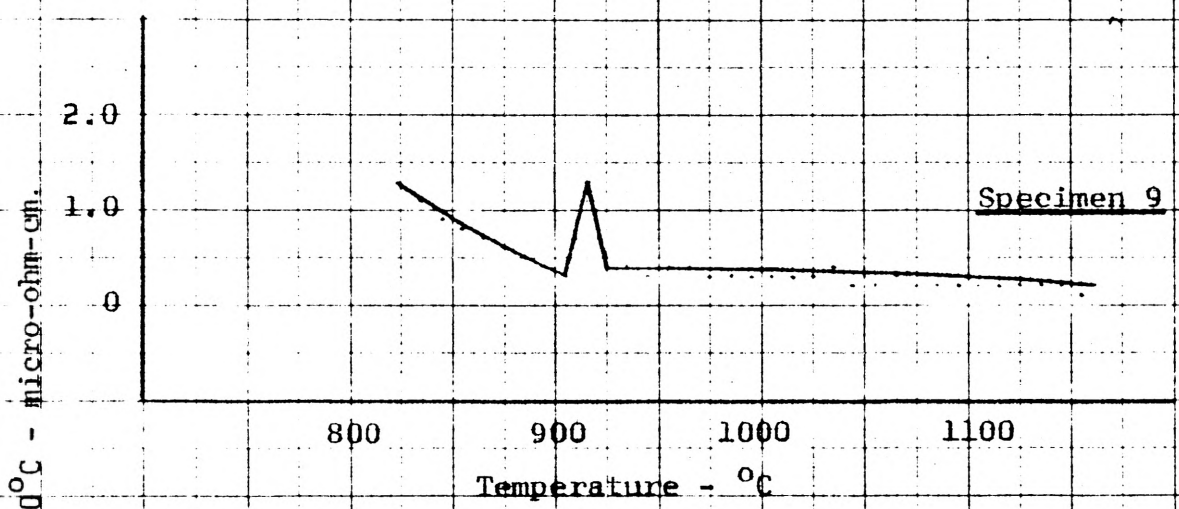


Figure 23  
Change in Resistivity per 10°C versus Temperature  
for Arceo Iron



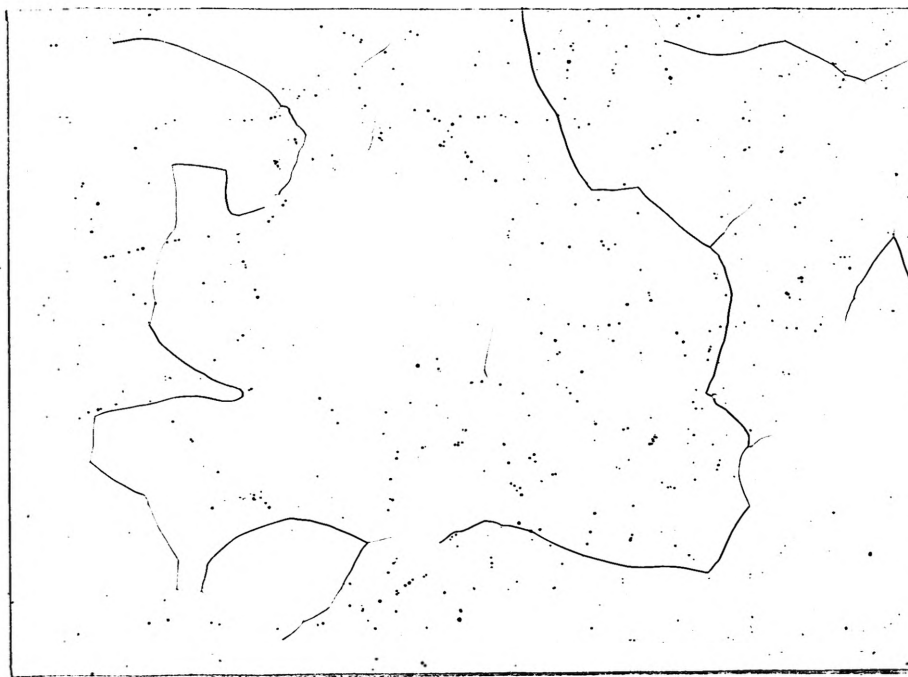


Figure 24. Pure Iron

Mag.-100X

Etch: 4% picral and 3% nital

Microstructure shows ferrite grains of pure iron after forging at 1093°C.

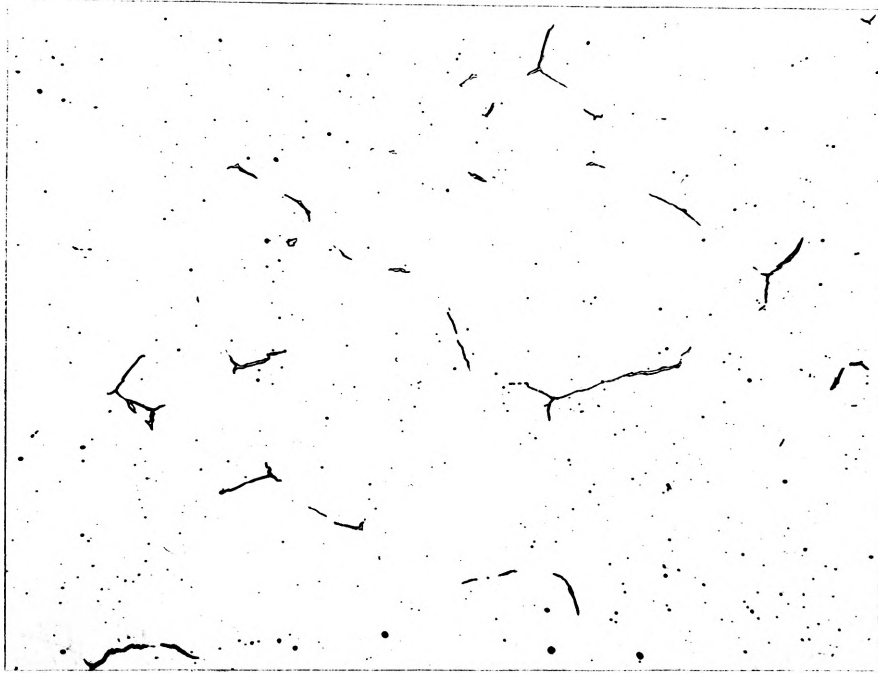


Figure 25. Iron + .023 Sulfur

Mag. -500X

Etch: 4% picral and 3% nital

Microstructure shows the Fe-S eutectic film normally found in red-short steels.

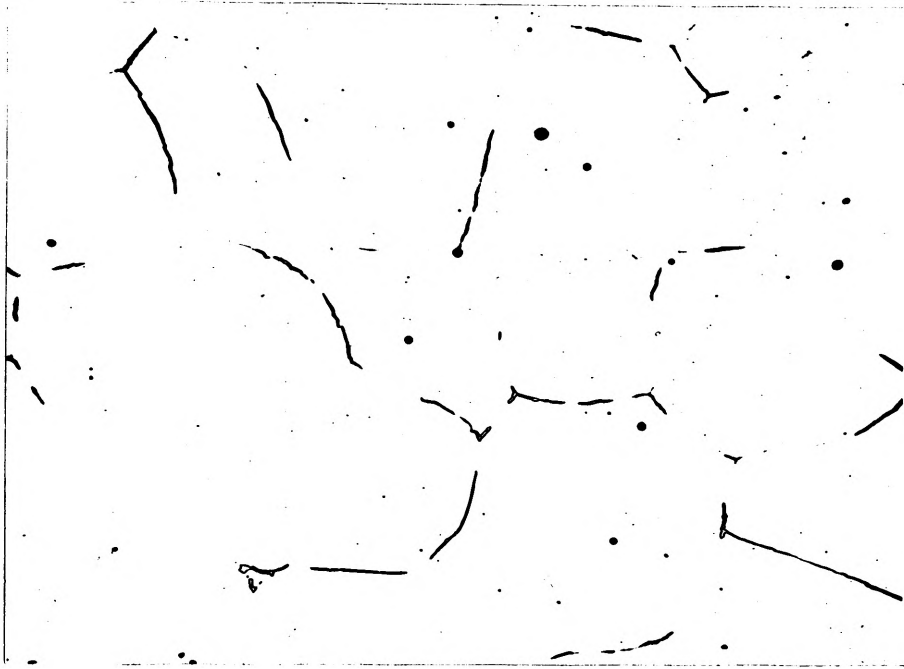


Figure 26. Iron + .33 Copper

Mag. -500X

Etch: 4% picral and 3% nital

This microstructure shows a phase present in the grain boundaries quite similar to Figure 25. This apparently is a copper rich phase precipitated during cooling from the forging temperature.



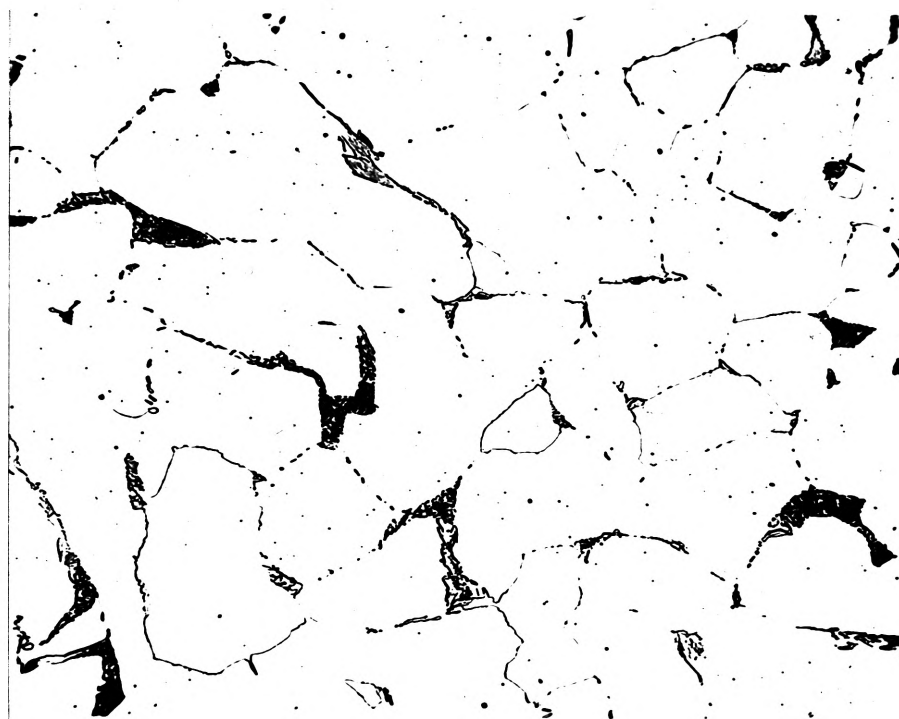


Figure 27. Iron + .024 Sulfur + .32 Copper

Mag.-500X

Etch: 4% picral and 3% nital

Microstructure shows copper-sulfur phase in grain boundaries. Also present are small amounts of pearlite due to .08 carbon in composition of steel.

TABLE 14  
DIMENSIONS OF SPECIMENS

Specimen No.	Material	Length-L cm.	Diameter cm.	Area <sub>2</sub> A cm <sup>2</sup>	A/L cm.
1	Fe	4.55	.640	.322	.0708
2	Fe	4.50	.640	.322	.0716
3	Fe + S	5.00	.630	.312	.0624
4	Fe + S	5.25	.633	.315	.0600
5	Fe + Cu	5.25	.635	.317	.0604
6	Fe + Cu	5.35	.633	.315	.0589
7	Fe + Cu + S	5.25	.633	.315	.0600
8	Fe + Cu + S	5.15	.630	.312	.0606
9	Armco Iron	4.55	.638	.320	.0703
10	Armco Iron	4.55	.638	.320	.0703

## X. CONCLUSIONS

From the results of this investigation the following is concluded.

An apparatus has been designed and developed which can be employed for measuring resistivity values of metals up to 1200°C and higher.

The "red shortness" range which occurs in some steels and irons can be determined by measuring resistivity changes at elevated temperatures. The formation of the liquid film at approximately 990°C changes the resistivity of the metal significantly. Likewise the disappearance of the liquid film at higher temperatures can be detected by an abrupt change in resistivity.

Pure iron with .33 copper added had no resistivity changes at elevated temperature that would indicate the formation of a liquid film as is the case with the Fe-S composition.

Pure iron with .32 copper and .024 sulfur is not red-short according to resistivity measurements.

### SUGGESTIONS FOR FURTHER STUDY

The research conducted in this investigation was of a very general nature and requires much more study to produce substantial results. Probably the major areas which can be investigated as indicated by the resistivity study are:

1. The Fe-S diagram can be studied at the iron rich end over a range of sulfur concentration by the resistivity method. A study of resistivity would substantiate the results of other investigations which all used chemical analysis to determine the Fe-S phase diagram.
2. The ratio of manganese to sulfur that would eliminate "red shortness" can also be an area in which a study could be undertaken. There is considerable disagreement among investigators to the amount of manganese required to offset excessive sulfur contents.

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APPENDICESAPPENDIX A

## Chemical Composition of Electrolytic Iron Used

## For Melting Stock

99.84% Fe  
0.002% Al  
0.002% Cr  
0.007% Co  
0.008% Cu  
0.010% H  
0.002% Mn  
0.002% Mo  
0.008% Ni  
0.004% N  
0.070% O  
0.005% P  
0.005% Si  
0.004% S  
0.005% Sn  
0.015% C  
0.003% V

## APPENDIX B

### Metallographic Procedure

#### Polishing Procedure.

The specimens were rough ground initially to obtain a flat surface. They were then ground from 1 through 4/0 emery paper. After each grinding step the specimen must be thoroughly washed.

After grinding on the 4/0 emery paper, the specimens were lapped on a nylon cloth charged with diamond paste. Final polishing was performed on a Metcloth charged with Linde A polishing compound.

The specimens were given a double etch in picral and nital. The first etch with 4% picral removed most of the worked surface. The second etch with 3% nital produced the final microstructures.

Photomicrographs were made on a Bausch and Lomb Metallograph using a tungsten arc as the light source. Metallographic plates were used for all micrographs.

#### Printing Data:

Specimen	Paper	Time
Fe	F-1	15 Sec.
Fe + S	F-3	20 Sec.
Fe + Cu	F-3	12 Sec.
Fe + S + Cu	F-3	8 Sec.

#### Developing Procedure.

1. Develop one minute in D-72.
2. Short stop for one minute.
3. F-5 fixer for ten minutes .
4. Wash for one hour.



APPENDIX C

## Sample Calculation of Resistivity

Example: Pure iron, specimen 2, temperature 1000°C

Voltage across standard resistor = 0.006225 V

Amperage flowing in circuits (I)

$$I = \frac{\text{voltage across standard resistor}}{\text{resistance of standard resistor}} = \frac{0.006225}{0.001}$$

$$= 6.225 \text{ amperes}$$

Voltage across specimen (E)

$$E = \text{potential across specimen} - \text{residual emf}$$

$$= 0.011385 - 0.000015 = 0.011370\text{V} = 11.370 \text{ MV}$$

Resistance of specimen (R)

$$R = \frac{\text{voltage across specimen}}{\text{amperage flowing}} = \frac{E}{I} = \frac{0.011370}{6.225}$$

$$= 0.001827 \text{ ohms} = 1827 \text{ micro-ohms}$$

Dimension of specimen:

Distance between potential leads = L = 4.50 cm.

Cross sectional area = .322 cm<sup>2</sup>

A/L = 0.0716 cm.

Resistivity = R X A/L = 1827 X 0.0716 = 130.8 micro-ohm-cm.

APPENDIX D

## Improvements of Apparatus

The major improvement which can be made on the resistivity apparatus is a furnace that can be set and controlled to 10°C intervals. The improvement would be realized in two ways. One, the temperatures measured would have considerably more accuracy and two, the series of readings required for each resistivity value would be taken at a constant temperature.

Also, a constant temperature furnace would allow the use of a potentiometer which could measure one micro-volt potential drops. A potentiometer with this accuracy requires an extremely sensitive galvanometer which in turn increases the time for individual readings. The increased time is the reason for requiring a furnace that can be set and controlled. The potentiometer and galvanometer must be selected with care to insure compatibility which gives maximum accuracy with a minimum interval of time for readings.

VITA

Harold Alfred Koelling was born in St. Louis, Missouri on December 16, 1932. He received his high school education at Grover Cleveland High School in St. Louis. He entered the Missouri School of Mines and Metallurgy in September of 1950 and completed the requirements for the Bachelor of Science Degree in Metallurgical Engineering in May, 1954. From June 1954 to June 1956 he served two years in the United States Army. He spent the following three years in industry and then enrolled in the graduate school of the Missouri School of Mines and Metallurgy. He was appointed a graduate assistant in January of 1960.

