

5-1-1942

Electrical Resistivity Measurements of Powder Metal Compacts

John M. Fitzpatrick

Follow this and additional works at: http://digitalcommons.mtech.edu/bach_theses

 Part of the [Ceramic Materials Commons](#), [Environmental Engineering Commons](#), [Geology Commons](#), [Geophysics and Seismology Commons](#), [Metallurgy Commons](#), [Other Engineering Commons](#), and the [Other Materials Science and Engineering Commons](#)

Recommended Citation

Fitzpatrick, John M., "Electrical Resistivity Measurements of Powder Metal Compacts" (1942). *Bachelors Theses and Reports, 1928 - 1970*. Paper 158.

This Bachelors Thesis is brought to you for free and open access by the Student Scholarship at Digital Commons @ Montana Tech. It has been accepted for inclusion in Bachelors Theses and Reports, 1928 - 1970 by an authorized administrator of Digital Commons @ Montana Tech. For more information, please contact ccote@mtech.edu.

Fitzpatrick, John M.

ELECTRICAL RESISTIVITY MEASUREMENTS OF POWDER
METAL COMPACTS

by

John M. Fitzpatrick

A Thesis

Submitted to the Department of Metallurgy
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Metallurgical Engineering

MONTANA SCHOOL OF MINES

Butte, Montana

May 1, 1942

MONTANA SCHOOL OF MINES LIBRARY.

ELECTRICAL RESISTIVITY MEASUREMENTS OF POWDER
METAL COMPACTS

by

John M. Fitzpatrick

14914

A Thesis

Submitted to the Department of Metallurgy
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Metallurgical Engineering

MONTANA SCHOOL OF MINES

Butte, Montana

May 1, 1942



TABLE OF CONTENTS

| | Page |
|---|------|
| INTRODUCTION..... | 1 |
| POWDER METALLURGY..... | 2 |
| General; | |
| Theory of Compacting; | |
| Properties of Powder Compacts; | |
| Shortcomings and Limitations; | |
| Trends and Possibilities. | |
| ELECTRICAL RESISTANCE..... | 8 |
| Problem; | |
| Previous Work on Electrical Resistivity. | |
| EXPERIMENTAL WORK..... | 14 |
| Apparatus and Technique; | |
| Procedure; | |
| Results of Experimental Work | |
| DISCUSSION AND INTERPRETATION OF EXPERIMENTAL WORK AND RESULTS..... | 34 |
| CONCLUSIONS..... | 42 |
| BIBLIOGRAPHY..... | 43 |
| ACKNOWLEDGMENTS..... | 45 |

INTRODUCTION

This work, as it was originally planned, was the arranging of an apparatus whereby electrical resistivity measurements could be made on powder compacts. It was also to include measurements on a series of copper-nickel compacts both before and after sintering. In this manner might be determined both the sintering temperature and the sintering time necessary to produce compacts having a resistivity approaching that of cast metal alloys.

The apparatus of measurement was developed and was found successful, but time was lacking for extensive work on actual measurements.

The purpose of this paper is to present as nearly as possible a complete history of the work done, including the obstacles which were met and which were in some measure overcome. It is hoped that by this presentation further research will be stimulated along the lines of investigation of powder compacts by electrical resistivity measurements.

POWDER METALLURGY

General

"Powder metallurgy is the art of making objects by the heat treatment of compressed metallic powders with or without the addition of a non-metallic substance."^{(1)*} Exception might well be taken to this definition since a compact metallic mass may be obtained by compression alone. In commercial practice, today, a simultaneous or subsequent heat treatment below the melting point of the components is standard practice.

Powder metallurgy found its beginning in the production of platinum from powders, as was done by Wollaston in 1828. This process gave rise to the production of articles of tungsten, molybdenum, and tantalum all of which are refractory metals either difficult or impossible to melt and cast.

Today, powder metallurgy is used not only for metals which are difficult or inconvenient to cast but also for metals of low melting temperatures. In these cases, special properties is the goal of powder technique.⁽²⁾

In the electrical industry for example, low melting, high conductivity metals are compressed with refractory metallic powders, such as tungsten and molybdenum to

* Refers to numbers in bibliography.

form a good conductor which will not fuse or volatilize at extremely high temperatures. An application of this is the manufacture of welding electrodes which must withstand high arcing temperatures. The combining of metals and non-metals is another great use of powders. The particular applications are in the manufacturing of cemented carbides, which are used for abrasives. Controlled porosity is an attribute of powdered articles and has led to the production of the so-called self-oiling bronze bearings. Very small metallic parts may be made directly by methods of powder metallurgy, thus eliminating costly machining.

Theory of Compacting

Adhesion accounts for the rigidity of the compact when pressed without heating. The greater the pressure and the greater the number of contact points, the larger are the forces of adhesion. It is evident, therefore, that finer powders will yield a sturdier compact than will coarse powders. The soft metallic powders are more plastic and therefore find a greater number of contact points. The consolidation of the compacted powders is said to be caused by the atomic mobility of the metal

at elevated temperatures. The cohesive and adhesive forces are replaced by an atomic interlocking and recrystallization takes place within the particles and at their boundaries.⁽²⁾ With further rise of temperature, grain growth caused by coalescence, results in shifting of the new grain boundaries and in the gradual disappearance of the old powder particle interstices. This process may go on until the compact attains the regular metallic state of an annealed casting of the same material.

Inter-diffusion⁽³⁾ is another type of bonding action which is believed to take place. It accompanies recrystallization and grain growth. Its effect is similar to that of having one component molten at the sintering temperature and results in a cementing action.

Properties of Powder Compacts

From research on powder compacts which has been done mainly on the refractory metals and on the cemented carbides, the following generalizations can be made.

Powders which have been compacted but not subsequently heat treated are, in general, weak and brittle. Their rigidity is an exponential function of the pressure undergone during compression. Their tendency to form a

rigid mass is also a function of the plasticity of the powders. The density of the compact depends upon the pressure, within the limits of compression.

The greater the amount of sintering, both as to time and temperature, the more nearly the compact approaches in its physical properties that of an annealed metallic casting of the same material.

In the field of powder metallurgy it has been found that simultaneous compression and heat treatment make for a speedy approach toward cast metal properties. In some cases where ductile metal powders were used it was found that by this method a compact could be made much harder than ordinary material of the same composition which had been severely cold worked.

Shortcomings and Limitations

Economically, in industry, powder methods are restricted. Other than articles made from tungsten, molybdenum, tantalum or the carbides, it is as yet cheaper to produce cast parts. The expense of dies, suitable for withstanding pressures as high as one hundred tons per square inch which will not deform during the molding operation is also a problem of economy which has not yet

been solved. The capacity of presses is limited so that very large compacts cannot be made.

In a great many cases it is difficult to obtain metal powders of a purity suitable for production of parts. Oxide films are the most vicious offenders in this respect. The oxides of the metals tin, bismuth, cadmium, zinc, and lead cannot be reduced at temperatures below the melting points of these respective metals. It is therefore impossible to get good bonding action at temperatures of sintering, which must necessarily be below the melting point.

The only method whereby the porosity of compacts can be eliminated is by simultaneous pressure and heat treatment. Porosity, however, is sometimes the main property sought after in the production of compacts.

Trends and Possibilities

The future of powder metallurgy is bright. An unlimited amount of work can be done in perfecting methods of compression, sintering, and the combination of these two, hot-pressing.⁽⁴⁾ Methods will undoubtedly be forthcoming for the production of impurity free powders.

Pre-alloyed powders⁽⁵⁾ are at present being developed

for the purpose of getting greater diffusion and cutting down sintering time.

Direct production of iron powders from ore is another line of research which bears watching.⁽⁶⁾

Swaging of compacted metal, as is done in the production of tungsten rod and wire is another comparatively new process which corresponds to a forging operation. Much work is possible along this line also.⁽⁷⁾

ELECTRICAL RESISTANCE

Problem

The object of this work was to investigate the property of electrical resistivity for a series of copper-nickel alloys prepared by the methods of powder metallurgy, and to correlate the results obtained with resistivity data on cast metals of the same composition.

It is known that if two metals form a heterogeneous mixture over a range of composition, the resistivity relation to composition is linear. If, however, the two metals form solid solutions, the resistivity relation to composition is not linear. If two metals, as copper and nickel, the ones used for this experimental work, are cast from the molten condition over the entire range of composition, and electrical resistance measurements made it would be found that with increasing amounts of one metal in the other, the resistivity would increase, reaching a maximum at about fifty atomic percent. The accepted explanation for this phenomenon is based on the fact that with two component alloys which form solid solutions over their entire range of composition, the lattice distortion is maximum at fifty atomic percent mixtures. Lattice distortion is in turn the argument

for increasing electrical resistance.

This study was made, keeping in mind the theoretical aspect of the problem as outlined above, and attempting to justify the results obtained in the light of that argument.

Previous Work on Electrical Resistivity

The first work of importance done on electrical resistivity measurements of powder compacts was by Streintz. (8) (9) His apparatus consisted of an ebonite cylinder, 2.2 centimeters in diameter. The cylinder was divided into three sections, the center one being one centimeter in length. A sheet of platinum foil was placed on each face of the center section and the three sections were screwed together. A hole was then drilled, 0.16 centimeters in diameter through the entire cylinder. The powder was compressed into the hole by means of a screw. No information is given concerning the pressure used. Electrical resistance measurements were conducted while the powder was under pressure, current being passed momentarily through the whole length of the powder compact and the resistance measured between the two platinum inserts. Measurements were made at 10°C . and at -78°C .

The work was conducted on platinum powder, which had been compressed to a density of 11.6 grams/cc. The mean of the measurements at both temperatures is given by the expression: $R_T = 0.92(1 + 0.00145T)$ ohms mm.² meter.

Skaupy and Kantorowicz⁽¹⁰⁾ have given careful attention to design of apparatus. The type used by these investigators was a horizontal mold, 15 mm. x 8 mm. The faces of the mold and ram were lined with bakelite and cambric. The pressure was applied at right angles to the direction of the current. This was done in order that no pressure gradients existed through the section involved. Provision was made for heating and cooling the mold. Pressure was applied by a direct loading hydraulic press designed to give constant pressures over long periods. The thickness of the pressings could be measured at any time by making microscopic observations of the position of the ram.

The results of these investigators is summarized as follows:

1. When under pressure the resistance of a metal powder in general is always greater than that of a cast metal. For soft metals; zinc, lead, tin, and silver the difference is smaller than for the hard metals; iron, nickel, tungsten.

2. The resistance of the compacts varies with the time of application of the pressure.
3. During the pressing, and with rising pressures, the resistance R may be expressed for a large range of values by an expression of the form:

$$1/R = c\sqrt{P} + C$$

where c and C are constants depending on the material and its previous treatment.

4. The resistance versus pressure curves are not reversible. With diminishing pressure, the resistance remains close to the minimum value obtained for the highest pressures and rises gradually at first, often increasing to an appreciably higher value only at low pressures.
5. After such treatment the initial values for the resistances of tungsten, nickel, silver, zinc, bismuth, iron, copper, antimony, platinum, and graphite, but not of gold, tin, and some varieties of lead, are lower than those obtained on repressing, each resistance being higher than that obtained on the preceding pressing. This is more particularly marked with the harder metals. With repeated pressings, in general,

the resistance versus pressure curves lie higher with each pressing up to about the seventieth, by which time, in the case of tungsten, the resistance under pressure will be ten times that at the initial pressure. These remarks apply both to the resistance and the specific resistance.

6. The increase in electrical resistance is dependant upon the duration of pressure. With short, high pressure and a soft metal powder, the resistance versus pressure curves obtained after succeeding applications of pressure lie below each other.
7. If a powder remains for a long time under constant pressure, its resistance increases rapidly at first, then falls slowly.
8. The resistance of a powder compact depends on the particle size, being in general higher, the finer the particles.
9. The temperature coefficient of a powder before pressing is strongly negative, but at high pressures depends on the direction of alternation of pressure, being negative for increasing and

positive for decreasing pressures.

10. Increasing the temperature of sintering reduces the resistance measured at room temperatures.
11. These results are reproducible, although certain departures from these generalizations have been noticed.

EXPERIMENTAL WORK

Apparatus and Technique

Mixer

In order to obtain a uniformly mixed charge of powders, a mixing mill was employed. This apparatus consisted of a small cubical tin box, $1 \frac{3}{4}$ inches on each edge. The axis upon which this mixing box rotated passed through the mid-points of two parallel edges. The shaft axis was driven by a small electric motor which was operated on 110 volt A. C. Two reduction pulleys were employed to obtain the desired speed of rotation which was about 30 r. p. m. Rubber bands were used as driving belts. Charges of the desired proportions were weighed on an analytical balance and then placed in the mixing mill. All charges made up in carrying on this work were mixed for 30 minutes.

Mold

The mold in which the compacts were pressed was a steel cylinder, $2 \frac{1}{2}$ inches in diameter and 4 inches deep. Axially through the center was a hole, $\frac{1}{2}$ inch in diameter through which was passed the pressing ram. The mold

was equipped with a heating element and openings for the admission of gases for atmosphere control during compression. All of the compacts produced in this work were made in an air atmosphere and at room temperature. After the pressure had come to a constant value, it was maintained for five minutes. The source of pressure was a Blackhawk, number EA 11, 20 ton hydraulic jack. The mold and jack were arranged in a frame as is shown in Plate I, page 19.

After mixing the powders in the mixing mill, 15 gram quantities were charged into the mold and compressed at 25 tons per square inch. The weight of powders and pressures applied were constant for all compacts.

Sintering Furnace

The sintering of compacts was done in an electric tube furnace. (Plate II, page 20) The temperature was controlled by a thermocouple unit which was encased in a fused quartz pipe and inserted through a cork into the end of the tube of the furnace. Through this cork also passed a tube for the passing of gases into the furnace for atmosphere control.

All sintering was done at 700^o C. in an hydrogen atmosphere.

Electrical Resistance Apparatus

The apparatus for measuring the resistances of the compacts consisted of a galvanometer, two small, known resistances on the order of one ohm each, a large variable resistance, on the order of 60 ohms, a 6 volt storage battery, a double pole, double throw switch, and a bench vise to which was mounted silver plates to make the contacts. The principle upon which the setup works is based upon comparing the IR drop across the compact in terms of a deflection on the galvanometer with the IR drop across one of the small known resistances, which is measured by a second deflection on the galvanometer scale. Referring to Plate III, page 21:

X is the resistance of the compact.

S is a small resistance and equals for all measurements, the value 1.2721 ohms.

T is a second small resistance, whose value is 1.2690 ohms.

R is the large, variable resistance of the order of 60 ohms.

I is the current in the main circuit.

When the double pole switch is thrown to the left, across the resistance of the compact, there is registered

on the galvanometer a deflection, D_1 . When the switch is reversed, across the resistance, S , a deflection D_2 is registered. The relation is then as follows:

$$\begin{aligned} IX &= KD_1 \\ \text{and } I_S S &= KD_2 \end{aligned} \quad \text{-----(1)}$$

K is the deflection constant of the galvanometer.

I_S is the current flowing through S and R which are in series, and both of which are in parallel with T .

From the above relationship the following equation may be written:

$$\frac{IX}{I_S S} = \frac{D_1}{D_2} \quad \text{-----(2)}$$

Since the current divides, in a parallel circuit, inversely as the resistances it follows:

$$\text{Since } \frac{1}{S + R} + \frac{1}{T} = \frac{1}{R'} = \frac{T + S + R}{(S + R)T}$$

R' being the effective resistance of the parallel part of the circuit,

$$\frac{T}{S + R + T} I = I_S \quad \text{-----(3)}$$

With this relation established between I and I_S , by substituting in equation (2),

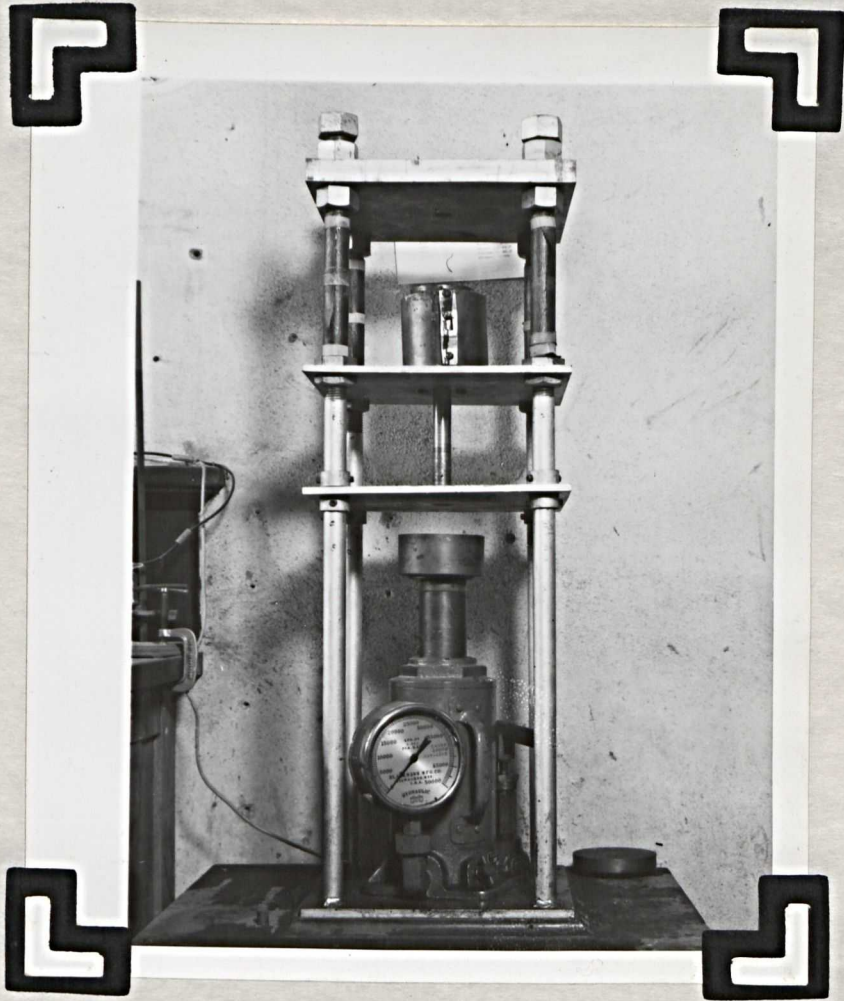
$$\frac{\frac{IX}{I_S S} \cdot T}{S + R + T} = \frac{D_1}{D_2}$$

$$\text{and } X = \frac{D_1}{D_2} \frac{ST}{S + R + T} \quad \text{----(4)}$$

Equation (4) was the one used to determine the resistance, X , of the compact. S and T were fixed, D_1 and D_2 were measured, and R was fixed, making the calculation possible.

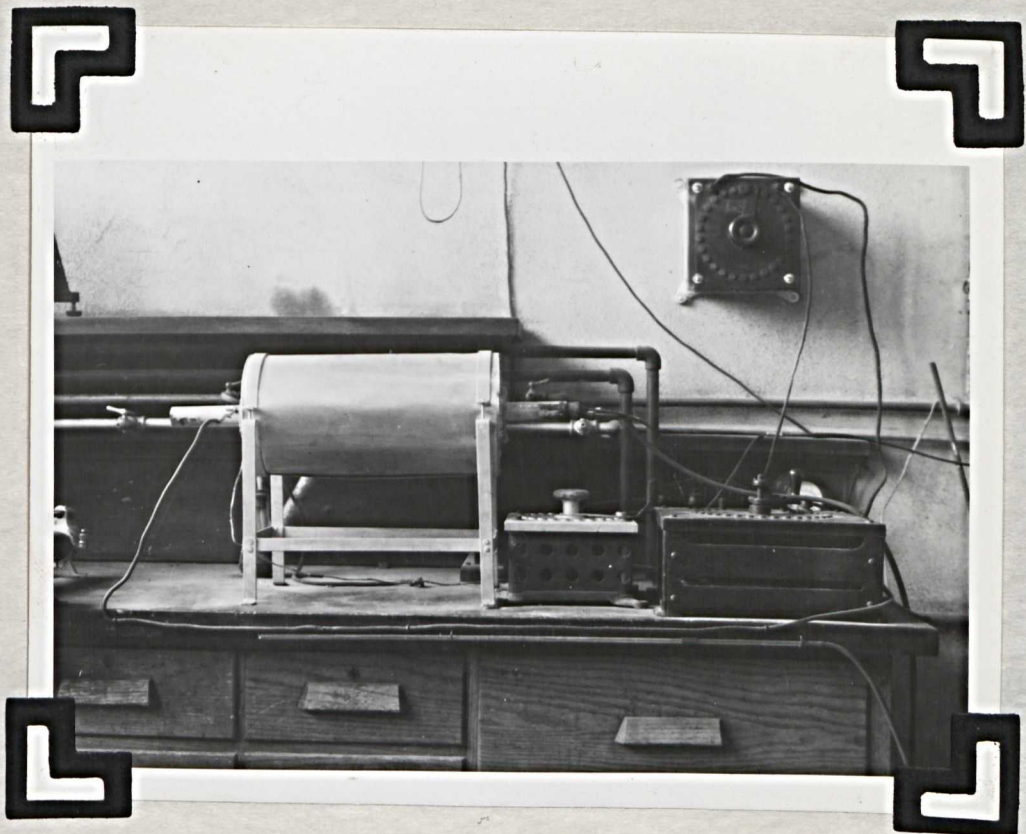
When the resistance of a compact was measured, the compact was placed between the silver plates on the jaws of the vise and the jaws tightened against the ends of the compact. The switch was then thrown and D_1 read on the galvanometer. This reading was then recorded and the switch thrown to the opposite set of poles and D_2 was read. To check the readings, the compact was removed from the vise, turned end for end and replaced. Readings were again taken in the same manner.

See Plate IV, page 22, for picture of this equipment.



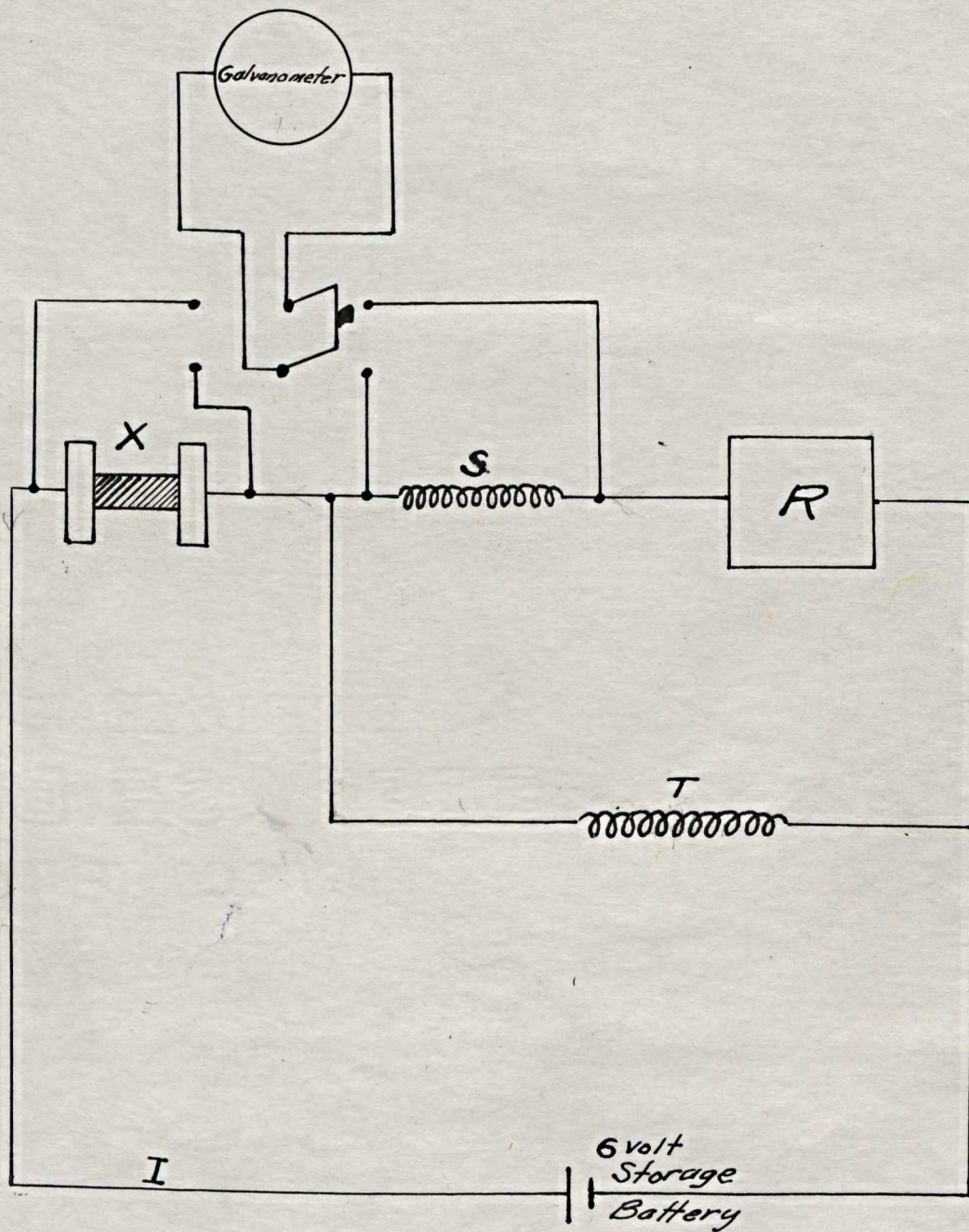
MOLD AND PRESS

Plate I



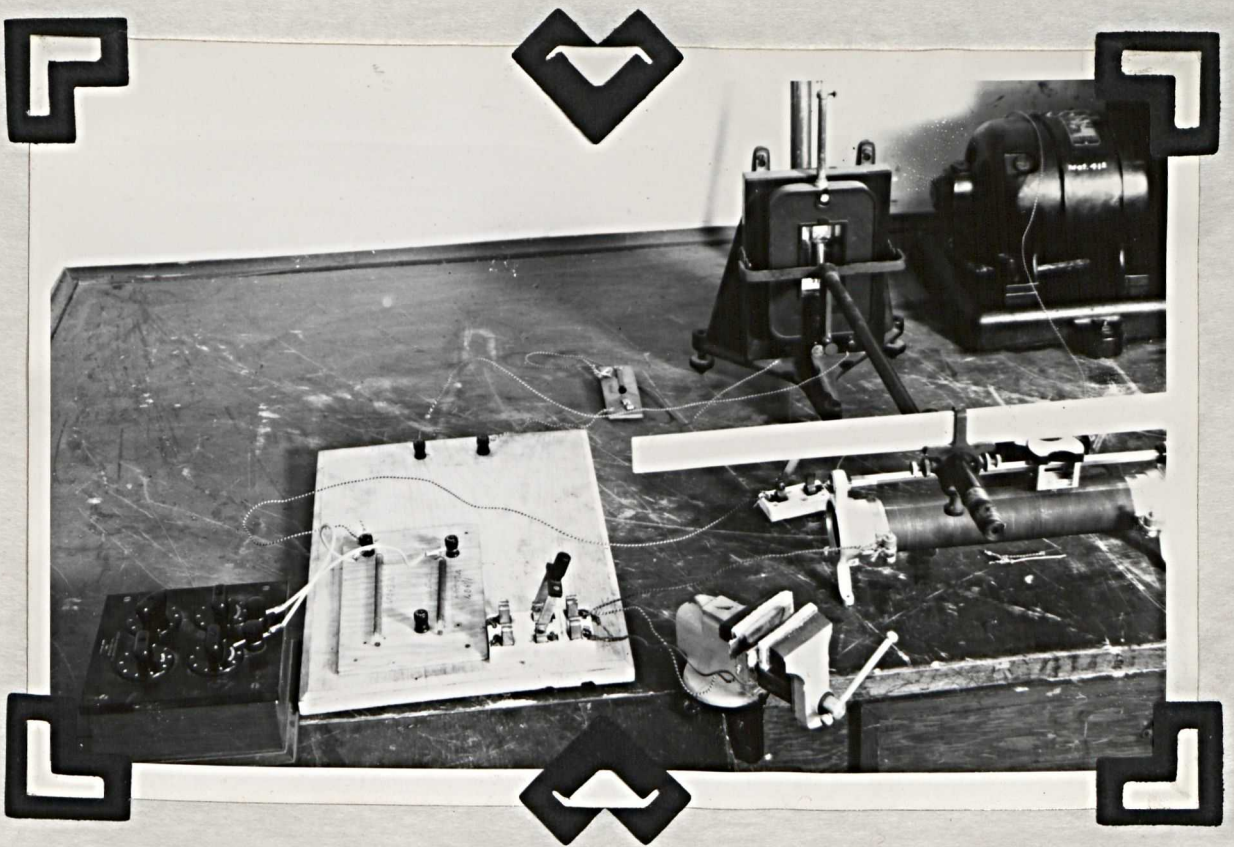
SINTERING. FURNACE

Plate II



ELECTRICAL RESISTANCE MEASUREMENT APPARATUS

Plate III



ELECTRICAL RESISTANCE APPARATUS

Plate IV

MONTANA SCHOOL OF MINES LIBRARY.

Procedure

Seven compacts were made with compositions varying from 100 percent copper to 100 percent nickel. The compositions of the various compacts were as follows:

1. 100 percent copper
2. 85 percent copper, 15 percent nickel
3. 65 " " 35 " "
4. 45 " " 55 " "
5. 25 " " 75 " "
6. 5 " " 95 " "
7. 100 percent nickel

These compacts were pressed at 25 tons per square inch for five minutes in an air atmosphere. After polishing the compacts with 000. polishing paper, resistance measurements were made. The length and diameter of each compact was measured with a micrometer, and the resistivity, ρ was calculated. The compacts were then put in a nickel boat and placed in the sintering furnace for a period of two hours. The temperature was maintained over this period at 700° C. and the atmosphere was made reducing by maintaining a stream of hydrogen through the furnace from a Kipp generator.

Upon removal from the furnace the compacts required

a slight repolishing on 000. paper. When this was done, resistance measurements were again made and the resistivity calculated. The compacts were again placed in the sintering furnace at 700° C. in an hydrogen atmosphere and kept there for a period of two hours. The total sintering time was then four hours, since the compacts had undergone two sintering periods of two hours each. This procedure was followed, using the same temperature and atmosphere, until resistance measurements had been made on the compacts for the following conditions:

1. Unsintered
2. Sintered for 2 hours.
3. Sintered for 4 hours.
4. Sintered for 8 hours.
5. Sintered for 16 hours.

Incidental to this work, there were made three photomicrographs, one of the unsintered material, one after the 4 hour sintering and one after the 8 hour sintering. (See Plate V, page 32, and Plate VI, page 33.)

Results of Experimental Work

Data

Unsintered

| <u>Composition percent</u> | <u>Length Cm.</u> | <u>* Area cm.²</u> | <u>X ohms</u> | <u>Resistivity ohm-cm.</u> |
|--------------------------------|-----------------------|-----------------------------------|-------------------|--------------------------------|
| Cu 100 | 1.700 | 1.261 | .0230 | .01710 |
| Cu 85,Ni 15 | 1.696 | 1.261 | .0254 | .01920 |
| Cu 65,Ni 35 | 1.767 | 1.261 | .0210 | .01500 |
| Cu 45,Ni 55 | 1.789 | 1.261 | .0140 | .00990 |
| Cu 25,Ni 75 | 1.882 | 1.261 | .0068 | .00455 |
| Cu 05,Ni 95 | 1.846 | 1.261 | .0050 | .00342 |
| Ni 100 | 1.904 | 1.261 | .0040 | .00265 |

Sintered 2 hours at 700^o C.

| <u>Composition percent</u> | <u>Length cm.</u> | <u>Area cm.²</u> | <u>X ohms</u> | <u>Resistivity ohm-cm.</u> |
|--------------------------------|-----------------------|---------------------------------|-------------------|--------------------------------|
| Cu 100 | 1.643 | 1.205 | .0007 | .00051 |
| Cu 85,Ni 15 | 1.727 | 1.338 | .0011 | .00085 |
| Cu 65,Ni 35 | 1.848 | 1.398 | .0013 | .000985 |
| Cu 45,Ni 55 | 1.832 | 1.350 | .0016 | .00118 |
| Cu 25,Ni 75 | 2.001 | 1.310 | .0016 | .00105 |
| Cu 05,Ni 95 | 1.826 | 1.260 | .0018 | .00124 |
| Ni 100 | 1.873 | 1.250 | .0018 | .00120 |

Sintered 4 hours at 700° C.

| <u>Composition percent</u> | <u>Length cm.</u> | <u>Area cm.²</u> | <u>X ohms</u> | <u>Resistivity ohm-cm.</u> |
|--------------------------------|-----------------------|---------------------------------|-------------------|--------------------------------|
| Cu 100 | 1.614 | 1.175 | .0006 | .00044 |
| Cu 85,Ni 15 | 1.665 | 1.281 | .0006 | .00046 |
| Cu 65,Ni 35 | 1.798 | 1.360 | .0016 | .00123 |
| Cu 45,Ni 55 | 1.807 | 1.340 | .0016 | .00119 |
| Cu 25,Ni 75 | 1.881 | 1.280 | .0012 | .00082 |
| Cu 05,Ni 95 | 1.801 | 1.240 | .0009 | .00062 |
| Ni 100 | 1.852 | 1.235 | .0014 | .00093 |

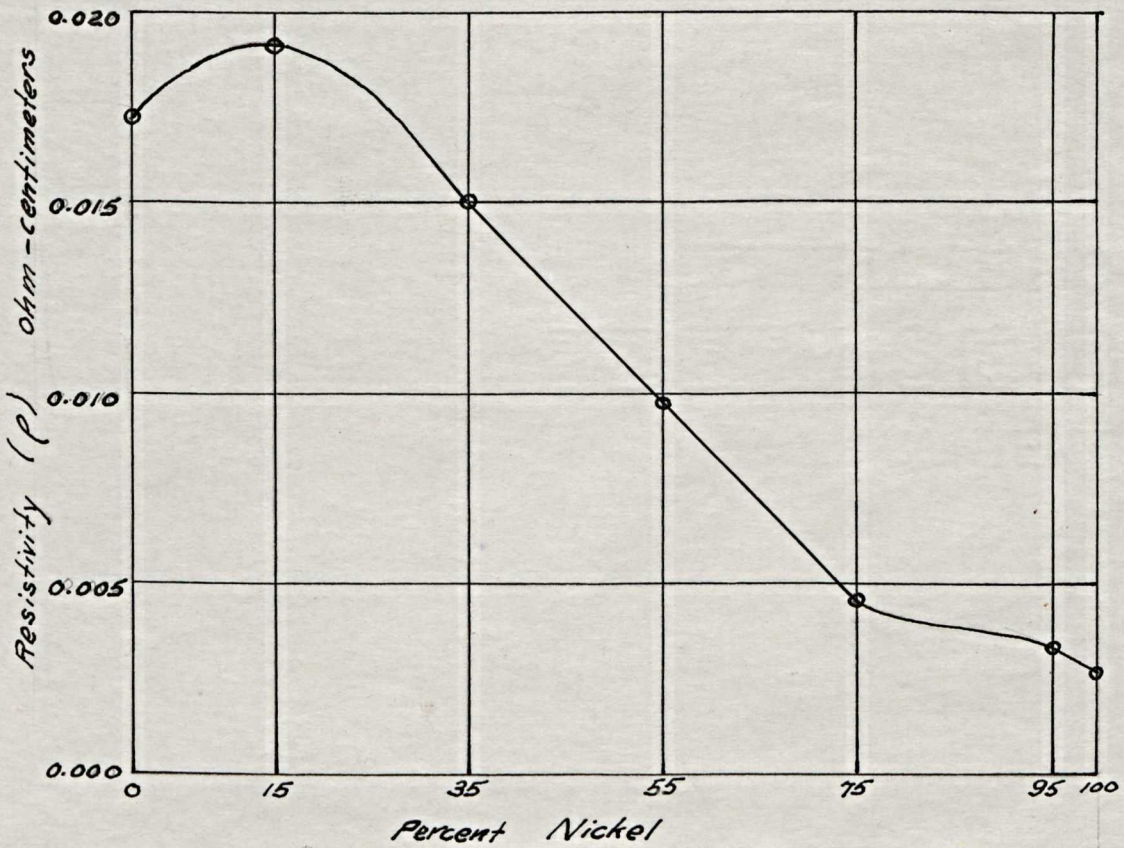
Sintered 8 hours at 700° C.

| <u>Composition percent</u> | <u>Length cm.</u> | <u>Area cm.²</u> | <u>X ohms</u> | <u>Resistivity ohm-cm.</u> |
|--------------------------------|-----------------------|---------------------------------|-------------------|--------------------------------|
| Cu 100 | 1.617 | 1.175 | .0006 | .00044 |
| Cu 85,Ni 15 | 1.614 | 1.228 | .0008 | .00061 |
| Cu 65,Ni 35 | 1.761 | 1.340 | .0011 | .00084 |
| Cu 45,Ni 55 | 1.790 | 1.320 | .0013 | .00096 |
| Cu 25,Ni 75 | 1.870 | 1.276 | .0011 | .00075 |
| Cu 05,Ni 95 | 1.774 | 1.200 | .0008 | .00054 |
| Ni 100 | 1.810 | 1.175 | .0008 | .00052 |

Sintered 16 hours at 700^o C.

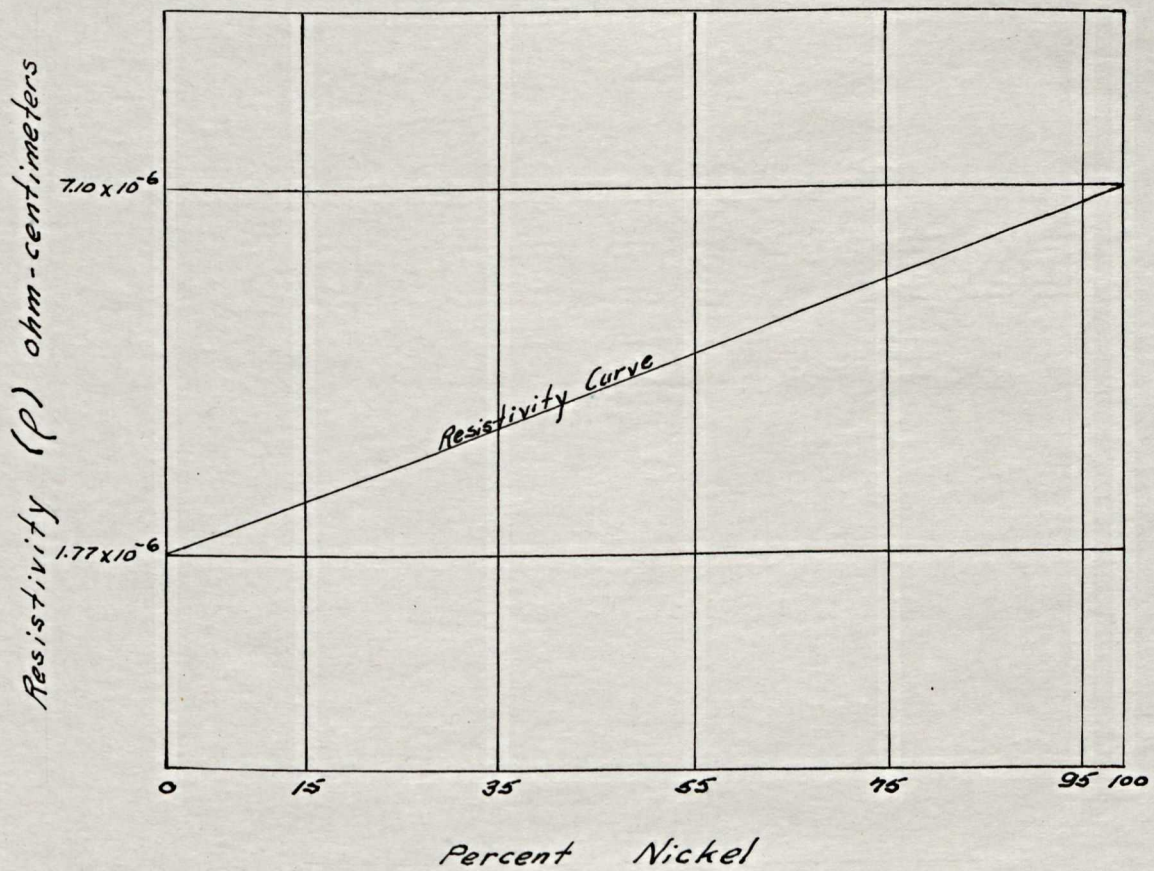
| Composition percent | Length cm. | Area cm. ² | X ohms | Resistivity ohm-cm. |
|------------------------|---------------|--------------------------|-----------|------------------------|
| Cu 100 | 1.641 | 1.20 | .0006 | .00044 |
| Cu 85,Ni 15 | 1.603 | 1.21 | .0008 | .00060 |
| Cu 65,Ni 35 | 1.751 | 1.31 | .0010 | .00075 |
| Cu 45,Ni 55 | 1.780 | 1.31 | .0011 | .00081 |
| Cu 25,Ni 75 | 1.819 | 1.27 | .0011 | .00078 |
| Cu 05,Ni 95 | 1.767 | 1.19 | .0008 | .00054 |
| Ni 100 | 1.808 | 1.16 | .0007 | .00045 |

* Area in cm.² refers to cross-section area of compact;
that area exposed to contact when measurements were
taken.



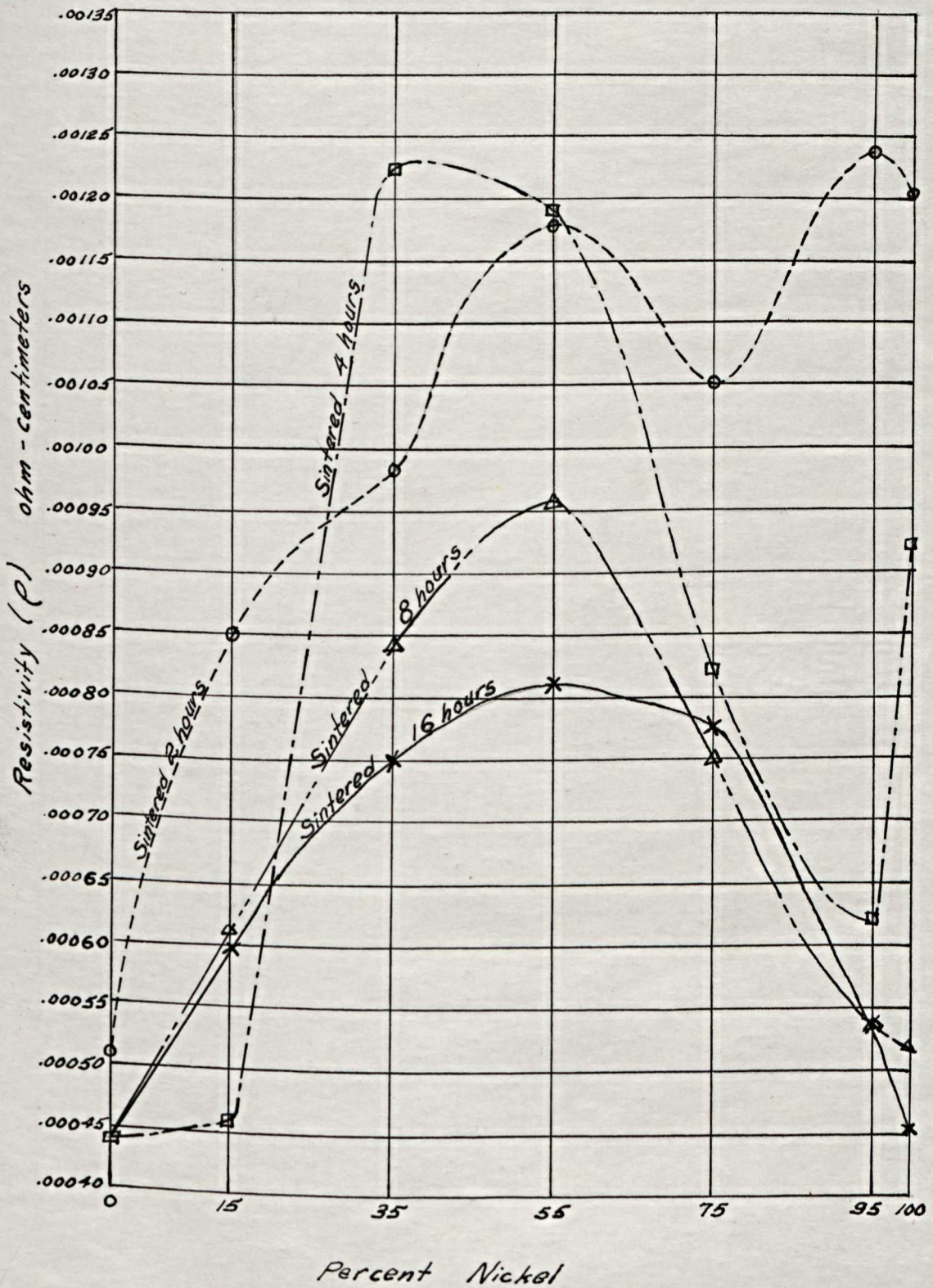
RESISTIVITY OF UNSINTERED COMPACTS

Figure 1.



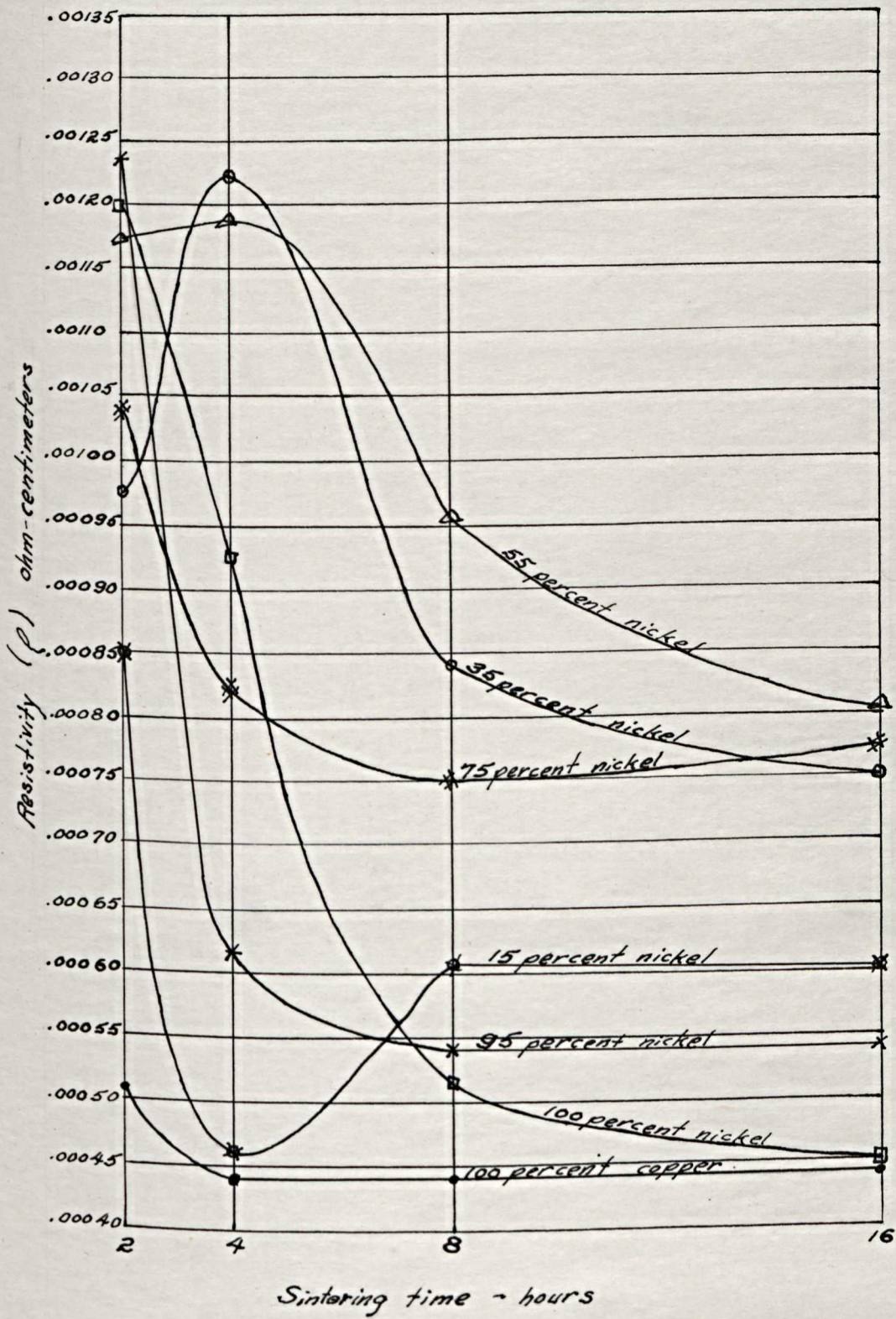
EXPECTED RESISTIVITY CURVE FOR UNSINTERED COMPACTS

Figure 2.



RESISTIVITY OF SINTERED COMPACTS

Figure 3.



CHANGE IN RESISTIVITY WITH SINTERING TIME

Figure 4.



UNSINTERED COMPACT

Copper 45 percent
Nickel 55 percent

250 diameters

Plate V

SINTERED
4 hours.
Copper 45
Nickel 55

250 X



SINTERED
8 hours.
Copper 45
Nickel 55

250 X

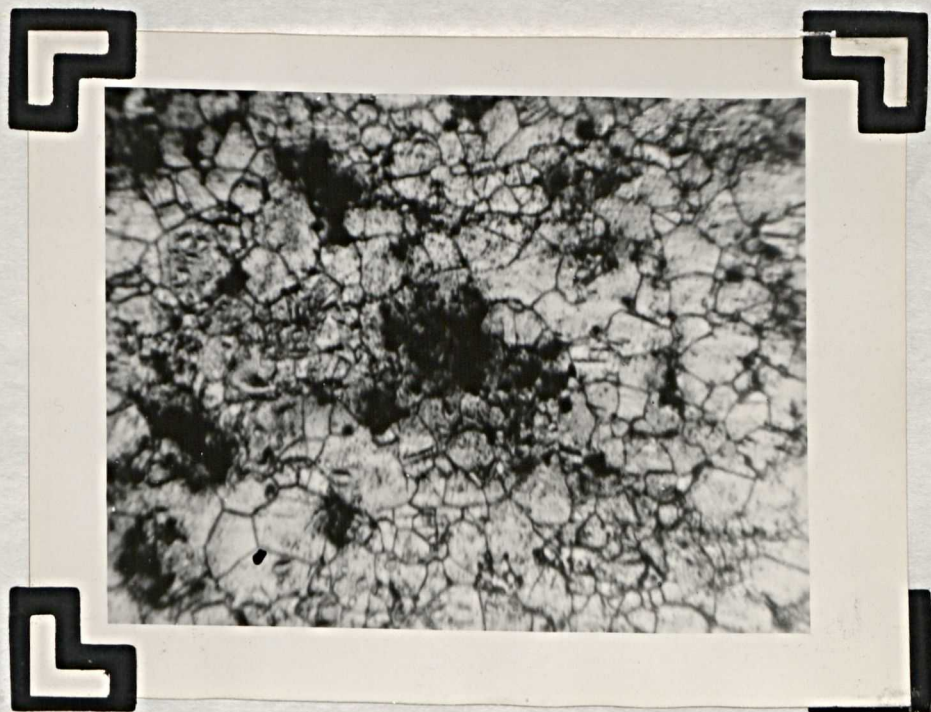


Plate VI

DISCUSSION AND INTERPRETATION OF EXPERIMENTAL
WORK AND RESULTS

The making of the electrical resistance apparatus which, in principle, is comparatively simple, consumed the greater part of the time spent on this work.

The first problem to be overcome was that of obtaining good electrical contacts on the ends of the powder compacts. By good electrical contacts is meant contacts whose resistances are so small in comparison to the resistances of the compacts that the inclusion of them in the resistance readings will not cause an appreciable error. Another attribute of good contacts is that they are constant, so that even though their magnitude may be relatively great, they will not interfere with comparative results except as to make the interval of variation smaller.

The solution to this problem of contacts was found in the use of highly polished silver plates. The material for these plates was scrap silver which was melted and cast in the reducing atmosphere of a carbon arc furnace. After casting, the silver was rolled and cut to the desired size and shape. After rolling, the surface of the silver was very rough and required intensive grinding

and polishing before the desired smoothness was attained.

The second problem to arise was that of obtaining a continuous contact over the entire end surfaces of the compacts. Since the compacts were not perfectly cylindrical, one face being slightly oblique, caused by an imperfection in the molding apparatus, this promised to be a difficult problem. This problem was answered by mounting the silver plates on sponge rubber, thus allowing the plates to fit firmly against the compact ends even though the ends were not parallel planes.

A small bench vise was used to apply pressure to the contact points. To each face of the vise jaws was glued, first the sheet of sponge rubber and then the sheet of polished silver.

Before reproducible resistance measurements could be made on any compact, the contact faces were polished with 000. polishing paper.

The small, known resistances, S and T, were made by winding Manganin wire on glass rods. The reason for using Manganin instead of Constantan was because Manganin wire may be soldered and Constantan cannot be soldered. Connections were made by soldering or by the use of small binding posts.

The double pole, double throw switch, resistances, S and T, and galvanometer binding posts were mounted on a board into which holes were drilled for the wires. As much of the assembly was soldered and connected together on this board as possible, thus eliminating variations in current due to changing contact resistances throughout the circuit. The leads to the silver plates were soldered in place.

In order to obtain actual resistance of a compact it was necessary to subtract from the reading, obtained by equation (4), the leads to the silver plates. To ascertain the value of these leads, and also those on S and T, a Wheatstone Bridge was used. Once these values were established it was not necessary to determine them again.

An unusual obstacle arose during the assembling of the resistance apparatus which although having no direct bearing on the specific problem involved, might be worth relating. In making preliminary determinations of resistance, to check the apparatus, it was found impossible to get reproducible results. The fault was finally brought to light after an aggravating search of every point in the circuit. The resistance box, R,

was found to be yielding resistances which were not in accordance with those read on the plug out openings. Since the error was not constant, the box could not be used. Another resistance box was secured and before placing it in the circuit was tested on a Wheatstone Bridge. This box had a constant error, of 0.5 ohm. By substituting this box and knowing its constant error, reproducible results could be obtained. It is advised, that when working with measurements of this kind, a complete check up of equipment be made before actually making determinations.

Referring to Figure 1., page 28, it is noted that the resistivity of the compacts decreases almost linearly with increasing amounts of nickel. The irregularity at the extremities of the curve cannot be accounted for except that they may be due to experimental error. The linear relationship shown was anticipated because the unsintered compacts represented a two phase alloy, a mixture of copper and nickel. The underlying reason for this relationship is stated in the problem on page 8.

The direction of the curve in Figure 1. is exactly opposite to that which was expected. It would be reasonable to suppose that resistivity should increase with

higher nickel content, since nickel has a greater resistivity than copper. (See expected curve, Figure 2, page 29) The fact that the curve slopes the other way may be partially explained by the assumption that the copper powders used in the experiment were highly oxidized, thus very greatly increasing the resistivity. If a coating of CuO surrounded each particle it would be an effective insulator and would indeed account for the high resistivity of the high copper compacts. It must also be assumed, if this explanation be accepted, that the copper powders were much more highly oxidized than the nickel powders. A second explanation for the direction of this curve may be found in the experimental work of Skaupy and Kantorowicz. (See generalization number 8, page 12.) The copper powders were much finer than those of nickel and therefore more surface was exposed to oxidizing action. This would bear out the oxide coating explanation.

Considering Figure 3., page 30, it is noted that the resistivity of the compacts, after sintering dropped about ten fold or more. If an oxide coating accounted for the high resistance of the unsintered material, it would be expected that by the treatment at 700°C . in an hydrogen atmosphere that resistivity should drop

due to the metal particle surfaces being reduced by the hydrogen. The results obtained after the two hour sintering show only a tendency for an increase in resistivity with increasing amounts of nickel. This is thought to be due primarily to the reducing action of the hydrogen. Assuming that very little diffusion took place during this operation, the general tendency of the curve is reasonable. It is supposed that some diffusion has taken place, but not to an extent where enough material is in solid solution to upset the roughly linear relationship of a two phase system. The curves of resistivity for the subsequent sintering times of 4, 8, and 16 hours all show the general shape of curves of solid solution alloys, rising to a maximum at about fifty atomic percent. The shapes of the 8 hour, and 16 hour curves are strikingly similar to those of cast copper-nickel alloys, the absolute values, however, being about ten times as great as those of cast alloys. The general tendency of the values is to decrease with increased sintering time, the intermediate compositions decreasing faster and more uniformly than the terminal compositions. This is borne out by a study of Figure 4., page 31, which illustrates the change in resistivity of each compact

with increasing sintering time.

The 8 and 16 hour sintering curves of Figure 3. are explained by the fact that with increased sintering, more diffusion takes place and thus the compacts become more solid solution and less heterogeneous mixture. An examination of Plate V and VI, pages 32 and 33, shows that recrystallization has taken place after sintering and therefore it is reasonable to suppose that inter-diffusion has also taken place. Another factor which bears out the assumption that the metal particles diffused, one in the other is their colors, before and after sintering. Before sintering, the compacts could be mixed and returned to their order of composition by noting the various shades of pink imparted by the presence of copper. After sintering, however, the only compact which had a different color from the rest was the one of 100 percent copper. The others appeared alike, all being the same as the 100 percent nickel compact. The connection between this phenomenon and diffusion lies in the fact that with cast alloys of copper and nickel, all above 15 percent nickel have the characteristic white color of pure nickel. This surely would indicate that sintering caused the compacts to have properties approaching those of

cast alloys.

Porosity may account for the much greater resistivity of the compacts after their final sintering, than the resistivity of cast alloys. The specific gravity of the compacts lies between 6.5 and 7.5, whereas the cast alloys of copper-nickel are close to 8.5.

CONCLUSIONS

Electrical resistivity measurements can be made in the manner outlined in this paper. Extraordinary care must be taken in the preparation of the contact points in order to get reproducible results. Highly polished silver plates make good contacts.

Alloys made by the methods of powder metallurgy show similar relationships between resistivity and composition as do normally cast alloys. It is believed that other properties of a compact may be determined by correlating resistance measurements with those properties. The nearness to which the properties of a compact approach those of cast alloys of the same composition is determinable by comparing resistance measurements.

It may be possible by the methods of powder metallurgy, to produce wires for use in resistance furnaces which have greater resistivity than the ones now being used, since with prolonged sintering, the compacts approach the resistivity of cast alloys very slowly.

BIBLIOGRAPHY

- (1) Hardy, Charles; Balke, Clarence W.
Metals Handbook, 1939 Ed., page 104.
- (2) Schwarzkopf, Paul; Goetzel, Claus G.
Iron Age, September 19, 1940, page 39.
- (3) Balke, Claire C.
Iron Age, April 17, 1941, page 23.
- (4) Schwarzkopf, Paul; Goetzel, Claus G.
Iron Age, September 4, 1941, page 37.
- (5) Comstock, G. J.
Metals Progress, Vol. 35, 1939, page 343.
- (6) Wyman, L. L.
Metals Progress, Vol. 35, 1939, page 585.
- (7) Sykes.
Journal of Chemical Education, April, 1940,
page 190.
- (8) Streintz.
Ann. d. Phys. Vol. 308, 1900, pages 1-19
Vol. 314, 1902, pages 854- 55

(9) Jones, W. D.

Powder Metallurgy.

(10) Skaupy, F. and Kantorowicz, O.

Z. f. Elek. Chem., Vol. 37, 1931, page 482.

ACKNOWLEDGMENTS

I wish to express my sincere thanks to Professor John P. Spielman, and Professor B. D. Cullity, under whose guidance this work was done.

I wish also to express my appreciation to Dr. George L. Shue for his assistance in making the resistance apparatus.

John M. Fitzpatrick