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Measurements of the Magnetic Permeabilities of the Iron-Manganese Alloy System Prepared by the Methods of Powder Metallurgy

O. Cihad Onbulak

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MEASUREMENTS OF THE MAGNETIC PERMEABILITIES OF THE
IRON-MANGANESE ALLOY SYSTEM PREPARED BY THE
METHODS OF POWDER METALLURGY

by

O. Cihad Onbulak

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 15, 1941

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INTRODUCTION

This work is concerned only with the measurements of the permeabilities of the iron-manganese alloy system prepared by methods of powder metallurgy. The investigations are made for the alloys containing manganese between zero and fifty percent, and it is not applied to the alloys of higher manganese contents because of the brittleness of Mn.

It was necessary to prepare the apparatus for the magnetic measurements and the heat treatments.

To investigate the effects of some variables of the method applied, preliminary work is done on an alloy containing Fe, Cr, Co, Mo, and Mn, which gave a good deal of practical and theoretical knowledge of obtaining a satisfactory compact from the powders of the metals. For that reason, the effects of the variables such as temperature, time, and the amount of pressing as well as the effect of the heat treatment on the permeabilities of the products were measured carefully. According to this knowledge, the compacts of the series of the Mn-Fe alloys are pressed at a temperature of 400^oc under a pressure of 50 tons per square inch and with a 2 minute time for pressing.

The permeabilities of these pellets which were

$\frac{1}{2}$ " in diameter and $\frac{1}{2}$ " high, are measured by marking off the magnetic intensity of the materials and using the formula of

$$B = H + 4\pi I; \quad \mu = 1 + 4\pi k$$

where μ = the permeability
 I = the intensity.

After this was done, the pellets were annealed at 600--700--800--900^oc, at each temperature about two hours; and after each annealing the permeabilities were measured; then they were carried on the tables by taking the permeability of the soft iron in the magnetic field strength used as 100.

The reasons for the changes in the permeabilities were carefully studied and theoretically explained.

Chapter 1

POWDER METALLURGY

Definition:¹⁾

Powder metallurgy is a branch of metallurgy which produces metallic compacts in their final forms by means of pressure and heat-treatment from the powders.

The products of powder metallurgy are being used in our daily lives quite often. From the tungsten wires in the electric bulbs to the silver-tin fillings of our teeth, some small parts of automobile motors, and oilless bearings are being produced by the methods of powder metallurgy.

Historical:

At the beginning of the 19th century the technique and the material of the furnace construction did not allow the acquiring of metals with high melting points, such as platinum and tungsten, by the methods of pyrometallurgy, in a pure state.²⁾ It was necessary, for example, to add some As into the platinum furnace in order to obtain an As-Ag alloy with a lower melting point than the pure platinum. This alloy, which had the composition of

1) Charles Hardy and Clarence W. Balke, Metals Handbook, 1939 edition, 104.
Richard Kieffer, Metall und Erz, Februar 1940, Band 37, No. 4, 67.

As-Ag eutectic, was poured out of the furnace and solidified. This bar was then heated to a red hot temperature in an oxidizing atmosphere, usually in air, until all the arsenic was volatilized. The platinum obtained was fairly ductile. But it still did not have the correct working properties of pure platinum.

In 1826³⁾ another method of obtaining platinum compacts was applied when the Czar issued the Russian platinum coins. With a chemical method of precipitation of the pure platinum metal from its solution, a platinum powder was pressed to a metallic compact under a high pressure which was heat-treated afterwards. In order to increase the density and tightness of the product, it was pressed again under a higher pressure and at a higher temperature than before. The pellets obtained were cut and minted as old Russian coins. These were used until 1865.

In 1829, Wallaston of England, explained the theory of the sintering of platinum. He also obtained some practical methods of pressing and sintering of the metal powders which are even in use in the powdered metal production of the Ferrous metals of today. Wallaston first produced a finely divided powder of platinum by the precipitation from its solution. Then he pressed this powder wet in a steel mold. This product had a specific

gravity of 10. The same pellet was then annealed in a furnace fired with charcoal, where its specific gravity increased from 10 to 17.0 or 17.7. After forging it he obtained a compact having a specific gravity of 21.44 which is the same as the theoretical.

The commercial application of powder metallurgy was started in the field of electrical industry.

It was necessary to have wires for many electrical purposes which should have high electrical resistivity, a high melting temperature, good working properties for drawing wires, and a high corrosion resistance at high temperatures. Tungsten, tantalum, and molybdenum were the only metals with these properties mentioned. Since the ovens and crucibles could not resist the high melting temperatures of these metals, it was necessary to melt first an alloy; for example, a W-Cu-Ni alloy, by the addition of amalgams which were drawn into wires. These wires were refined of the impurities by heating these in an oxidizing atmosphere.

In 1908, Coolidge⁴⁾ discovered a process of ob-

- 2) Richard Kieffer, Metall und Erz, Februar 1940, Band 37, No. 4, 67-68.
- 3) Richard Kieffer, Metall und Erz, Februar 1940, Band 37, No. 4, 67-68.
- 4) C. Coolidge, Journ. Am. Inst. Eng., 29, 1910, 971.

taining thin wires from the tungsten powders. The powder of tungsten which was obtained by the reduction of powdered oxide of the metal, was pressed at a relatively high pressure and the compact obtained was forged at about 1500°C. This product then could be drawn into the wires of 3mm diameter.

The next products of powder metallurgy were the contact alloys of non-alloying⁵⁾ elements. It was possible to produce aggregates⁶⁾ of non-alloying elements, which have the physical properties of their components. The contact alloys of W-Cu are being used for electrical switches, while the aggregate of Cu-graphite is the most common material of dynamo brushes.

The production of materials of the desired porosity was the next and one of the largest applications of powder metallurgy of the 20th century. Oilless bearings and filters belong to this group.

In the last ten or fifteen years the methods of powder metallurgy has been proved on such a large scale that its application in the metallurgical processes of obtaining small particles of machinery is almost

5) Richard Kiffer, Metall und Erz, Band 37, No. 4, Februar 1940.

6) Charles Hardy and Clarence W. Balke, Metals Handbook, 1939 edition, 104.

equal to the application of diecasting.⁷⁾

Advantages and Disadvantages of Powder Metallurgy⁸⁾

The advantages are:

1. Obtaining of refractory metal in useful forms.
2. Form obtained has a definite accuracy.
3. No waste of scrap.
4. None or a relatively low cost of machining.
5. Exact composition of alloys obtained.
6. No impurities in the products which is very common for the conventional methods of pyrometallurgy.
7. Aggregates of non-alloying elements can be obtained.

Parallel to these advantages are some disadvantages which limitate the application of powder metallurgy:

1. High pressure is required which increases the cost of equipment and tools.
2. Many metal powders have a tendency to cling to the walls of the die which gives an uneven distribution of pressure throughout the mass. This fact limitates the size of the product.
3. Another limitation might be the production of metal powders for some particular cases.⁹⁾

Theory of Sintering:

The products of powder metallurgy depending upon the theory of sintering may be classified into two major groups;¹⁰⁾

1. The products which are cemented by means of a formed liquid phase.
2. Those products which are cemented by means of cohesive forces, available between the surface crystals of the particles without the formation of any liquid phase.

The examples for the first group are the hard carbide tools and sintered contact alloys and dental alloys. The examples for the second class of production are the wires of tungsten, molybdenum, titanium, tantalum and the compacts of the ferrous metals.

The most satisfactory theory of cohesion of today can be summerized as follows:

It has been discovered that the particles having a grease film on their surfaces cannot be pressed

- 7) Earl S. Patch, Iron Age Magazine, December 19, 1940.
- 8) Charles Hardy and Clarence W. Balke, Metals Handbook. 1939 edition, 104-105.
- 9) Earl S. Patch, Iron Age Magazine, December 19, 1940.
- 10) Richard Kieffer, Metall und Erz, Februar 1940, Band 37, No. 4, 69.

into compacts as well as clean particles can be. It is claimed that the pressure applied brings the clean surfaces of the particles to such a small distance from each other that the molecular forces would be effective. The thermal agitation of atoms increase with the temperature and probably the atoms on the surfaces of the particles enter the crystal space lattice of neighboring atoms.¹¹⁾ This fact can be proved by the occurrence of grain growth in the pressed powders.

The effects of different variables may be summarized as follows:

1. The metals with clean surfaces adhere strongly by means of a relatively low pressure.
2. By the increase of the plasticity of the metals by means of higher temperatures, the force of adhesion increases strongly.
3. A slight film of grease prevents cohesion entirely.
4. The oxide film mostly decreases the force of adhesion; therefore, it is undesirable; but in some cases, such as by the pressing of molybdenum powders, an oxide film is highly favorable.

In practice some products would be obtained at a temperature at which about 5--20% of the entire mass liquifies. A typical example for this class of sintering is the product of the tungsten carbide and cobalt, which results in a very hard material. For this purpose, the mixture of cobalt and tungsten carbide is pressed at a temperature of about 1400°C, which forms a liquid of the ternary eutectic of tungsten, cobalt, and carbon. This liquid fills each capillary and results in a shrinkage of 20% of the entire length.

Another example for this class of sintering is the production of heavy metals¹²⁾ which contain 90% tungsten, 6% nickel, and 4% copper. In this case, a ternary eutectic of tungsten, nickel, and copper forms which melts at 1400°C, at which the mixture is pressed.

- 11) Richard Kieffer, Metall und Erz, Februar 1940, Band 37, No. 4, 70.
- 12) Richard Kieffer, Metall und Erz, Band 37, No. 4, Februar 1940, 70.

The Production of Metal Powders:13)

The production of metal powders with an economical method of a sufficient purity has been a very important factor of powder metallurgy. One of the oldest known methods of metal powder production was the chemical process of precipitation of metals from their solutions which was applied to the precious metals. Also, the method of the reduction of metals from their powdered salts by means of an alkaly or earth alkaly, is being used at the present time only for the production of rare metal powders.

The reduction of powdered metallic oxides, especially the oxides of tungsten, molybdenum, and cobalt with hydrogen, is also a common process.

The mass production of Iron and Nickel powders will be carried on by the carbonyl process. This process depends upon the fact that nickel forms a carbonyl, $\text{Ni}(\text{CO})_4$, and iron $\text{Fe}(\text{CO})_5$ or $\text{Fe}(\text{CO})_7$ when CO gases are sent through. The carbonyls formed are unstable above a definite temperature; for example, $\text{Ni}(\text{CO})_4$ is unstable above 180°C at which they are in a gaseous state. The deformation of this gas in the pure metal and the CO gas

13) A summary of Metalls Handboock and Metall und Erz.

makes it possible to obtain as fine particles of nickel or of iron as is desired. Today this process is being applied for the production of metallic powders of iron, nickel, cobalt, tungsten, molybdenum, and chromium.

The electrolytic process of metal powder production by the addition of a reagent to the electrolyte is a very commonly applied method. The powders of copper¹⁴⁾ and iron are being prepared by this method. These powders are especially good because of their porous surfaces.

A mechanical method of metal powder production is the grinding of pure metals or their alloys. This method today is the most widely used process of the powder production of bronze, brass, aluminum, and magnetic alloys.¹⁵⁾

Another way of obtaining powders is the granulation of the melted metals. This is one of the most economical processes of producing the powders of the metals with low melting temperatures. This method makes it possible to obtain the powders of very ductile metals, which cannot be ground easily.

The method of volatilization or evaporation and the condensing of metals is being applied to those which have a higher vapor pressure at relatively lower temperatures. The zinc powders are being produced by this process.

The powders of very hard metals or metallic compounds, which have high melting points such as the carbides of molybdenum, tungsten, titanium, and tantalum are being produced by heating the powders of these metals or their oxides with finely ground carbon.¹⁶⁾

- 14) Claus G. Goetzl, Metals and Alloys, July and August 1940.
Richard P. Seeling, Metal and Alloys, December 1940, 744.
- 15) V.E. Legg and F.J. Given, The Bell System Technical Journal, July 1940.
- 16) Richard Kieffer, Metall und Erz, Band 37, No. 4, Februar 1940.

The Products of Powder Metallurgy and their Uses:

The materials with very high melting points were the first groups produced by the methods of powder metallurgy. The tungsten wires, for example, are being used for many electrical purposes because of their high electrical resistance, high fusing temperature, and relatively good mechanical properties at very high temperatures. Today the wires in electric light bulbs, in radio tubes, anti-cathodes in X-ray tubes, and contact interrupters in the benzine motors are made of tungsten. Furthermore, tungsten is utilized as electrodes for arc-atom-arc-welding¹⁷⁾ purposes where by means of nascent hydrogen, a temperature of $4,000^{\circ}\text{C}$ can be obtained. Arc-atom-arc-welding is used in the light metal industries, especially in the manufacturing of airplanes.

The products made of molybdenum are also very important. They are used for particular purposes, preferably the tungsten products because of their higher flexibility. The heating bodies for high temperature furnaces are made of molybdenum.

The products of powder metallurgy from tantalum are used in the chemical industry because of their high resistivities against the acids and against high temperatures.

One of the most important products of powder

metallurgy are the oilless-bearings, which are in use in the U.S.A. of more than one-billion at the present time.¹⁸⁾ In order to produce these bearings, 20% of graphite and some organic substances are added to the mixture of copper and tin powders. The whole mixture is then pressed into the forms of bearings at a temperature at which a liquid phase of tin-copper alloy forms, containing mostly tin. The evaporation of the organic substance added, produces pores in the body of the bearing which can be controlled exactly. This porous product which is in the shape of bearings is dipped into the oil in order to fill the pores, and then it is placed in the machinery. The increase in temperature, because of the friction of the shaft, brings the oil out of the pores which forms a grease film between the shaft and the surfaces of the bearing.

The production of the hard materials of tungsten carbide have been discussed previously. In the U.S.A., however, the compacts of the following compositions are obtained by the methods of powder metallurgy:

- 1) W 90%, Fe 10%
- 2) W 80%, Co 15%, Cr 5%
- 3) W 80%, Cr 19.5%, C. 0.5%

Another group in the production of powder metallurgy are the compacts of non-alloying elements¹⁹⁾

which are called contact-alloys.²⁰⁾ It is, for example, impossible to obtain an alloy of copper containing 5-20% graphite since carbon is insoluble in liquid copper. But the fact that such a mixture would have easy sliding properties of graphite and the high electrical conductivity of copper, it is an ideal material for dynamo brushes. In order to increase the hardness of the copper, from 5 to 10% of Sn, Zn or lead powder is added to the powdered copper-graphite mixture and then it is pressed into the forms of dynamo brushes at a temperature, at which a liquid phase of Cu-Sn-Zn-Pb is formed. The contact alloys of W-Cu, W-Ag, W-Mo are produced with the same methods; they are used for electrical switches and circuit breakers.

-
- 17) Richard Kieffer, Reutte(Tirol), Metall und Erz, Band 37, No. 5, Marz 1940, 88.
 - 18) Earl S. Patch, Iron Age Magazine, December 19, 1940.
 - 19) Charles Hardy and Clarence W. Balke, Metals Handbook, 1939 edition, 104.
 - 20) Richard Kieffer, Metall und Erz, Band 37, No. 5, Marz 1940, 89.

Chapter 2

MAGNETISM

Historical:23)

The ancient Greeks and Romans discovered an iron ore, magnetite, in the Magnesia (Magnisa) district in Asia Minor, which had the properties of attracting small pieces of iron. This property of attraction was called magnetism, while the substances which had the magnetic properties were named magnets.

As early as 1100 B.C. the Chinese were familiar with the fact that a piece of magnetite suspended on a needle which could swing freely on a horizontal plane would set itself in a definite direction, which is now known as the magnetic south and the magnetic north of the earth. It is even known that the Chinese led their ships using a piece of magnetite suspended on a needle. This was known by the Europeans 2000 years later, when they named the magnetite the lodestone (leading stone). Later, a thin iron rod was used for the same purpose which was magnetized with a lodestone. When the properties of electricity were discovered, the name of lodestone was entirely forgotten; while it was possible to magnetize a piece of iron by means of an electrical current.

The Properties of Magnets

Magnetic Poles:²⁴⁾

When we take a magnetized thin iron rod suspended on a needle and mark one of the ends showing the north with "N" and the other end with "S" and bring it near to another iron rod which is prepared the same way, we then discover that the ends of the rods marked with the same letters repel each other while the different named ends attract each other. The force of repulsion and attraction seem to be concentrated near the ends of the rods, which are called magnetic poles.

Magnetization:

When an iron rod shows the properties of a magnet it is magnetized. This rod does not show any change in its weight before and after the magnetization. It has been discovered that in a piece of iron an increase in length occurs parallel to the magnetization, which indicates some rearrangement of the particles in the body of the iron bar; when the same bar is cut into small pieces, each particle would still have magnetic properties. These facts might give us an idea about the architecture of the iron bar which is formed by small permanent magnets.

The Strength of Poles:

Coulomb²⁵⁾ discovered in 1785 that the forces of repulsion and attraction between the two poles of two different magnets are in reverse proportion with the square of distance between the poles. He also discovered that different magnets have different attraction or repulsion forces against one another from the same distance. These facts were expressed in a formula

$$F = \frac{m_1 \times m_2}{d^2}$$

where d = the distance between the poles concerned;

m_1 and m_2 = the strength of the magnetic poles.

But each medium between these two poles does not have the same characteristics of behavior to the magnetic forces which is expressed with the Greek letter μ and called the magnetic permeability. Thus the formula would be

$$F = \frac{m_1 \times m_2}{\mu d^2}$$

where μ is practically equal to 1, for gases.

23) Poor, Electricity and Magnetism, 1931 Chapter 2.

24) Duff, Physics, Eighth Edition, 346.

25) Hausmann-Slack, Physics, Fourth printing, Chap. 2.

From this formula we can define the unit pole; a unit pole is one, which repels another similar pole of the same strength with a force of one dyne when placed one centimeter apart from it.

A magnet forms a magnetic field whose unit is named one Oersted which is the intensity of a field that would exert unit force on a unit pole. The strength of the field expelled by a magnetic pole of strength (m) is given by the formula of

$$H = \frac{m}{r^2} \text{ Oersted's per sq. in.}$$

and H is called fluxdensity or magnetic induction which is measured in Gausses and is shown:

$$B = H$$

When a coil of a substance having a permeability of μ is put into a selenoid which has a magnetic field strength of H Oersteds, the field strength of the selenoid increases. The excess magnetic force produced by the iron coil collectively with the magnetic forces of selenoid is called magnetic induction or magnetic flux; and the strength of it is called fluxdensity which is also measured in Gausses and shown with B.

When an area of A sq. cm. has an equally divided magnetic strength of H Gausses, the flux of this field

is equal to $\phi = B \times A$ Maxwells.

The ratio between the flux density B and the field strength H which produces this fluxdensity is called the permeability of the substance concerned.

$$\mu = \frac{B}{H}$$

The intensity for magnetization, I, which is the pole strength of a unit area produced in a magnetized bar by a field of H is equal to

$$I = \frac{m}{a}$$

Since the forces produced by a pole of m strength is equal to $4\pi \frac{m^2}{a^2}$, we would find for a unit area,

$$F = \frac{4\pi m}{a} = 4\pi I.$$

While the magnetic induction B is equal to the field strength H collectively with the field strength by the magnetized metal then

$$B = H + 4\pi I.$$

The ratio of $\frac{I}{H} = K$ is called magnetic susceptibility.

The above formula then can be written

$$\mu = 1 + 4\pi K.$$

This formula gives the relation between the permeability

and susceptibility which may be used to indicate the permeability of the metals.

The permeability and susceptibility of the of the magnetic substances depends upon their temperatures and the field strength applied. For example, the permeability of iron increases with increasing H up to a certain point, then decreases. When we examine the relation between B and H, first the magnetic induction B increases very rapidly with the increasing strength of the magnetic field applied; then it remains constant; thus the iron is at this point saturated. According to their permeabilities, the substances are classified into three groups:

1. Ferromagnetic substances; these have a permeability much greater than unity at ordinary conditions. Iron, cobalt, and nickel belong to this group.
2. Paramagnetic substances; these have a permeability slightly greater than unity; they are feebly attracted by a magnet. Aluminum, chromium, manganese, sodium, and platinum are some of the 20 paramagnetic elements.
3. Diamagnetic substances; mostly gases have a permeability slightly smaller than unity, which are called diamagnetic substances. Bismuth is also diamagnetic, having a permeability of 0.9998. These elements are repelled by a magnet.

The Physical Basis of Magnetism

There has been many theories advanced in order to explain the physical basis of magnetism. The latest theory which has been the most accurate explanation of magnetism depends upon the connection between magnetism and the atomic structure of the magnetic metals. To make our explanations understandable, it is necessary to give a short review about the atomic structure of the elements.

Structure of an Atom:²⁶⁾

The static electricity of an atom which is neutralized by positive and negative electricity, was known in the 18th century, but the distribution of this electricity in an atom was not yet discovered. In the 18th century physicists accepted the theory of Thomson who described an atom as an area of positive electricity in which negative electricity was moving. In 1903 Lenard bombarded the atom with cathodic rays and found that positive electricity did not cover all the body of an atom. More exact experiments were carried on by Rutherford in 1911 which had already been made by Geiger and Marsten by using alpha-rays.

It was disclosed that the positive electricity of an atom was concentrated in the center of the atom

forming a nucleus around which the negative electricity or electrons were moving. The nucleus of an atom had a size from 10^{-13} to 10^{-11} cm, depending upon the kind of the atom. For example, hydrogen has a nucleus of a 2×10^{-13} cm diameter. The nucleus never occurs free of the matter, while the electrons which are only as big as a $\frac{1}{1847}$ th part of a hydrogen atom can be free of the matter.

Bohr's Theory:²⁷⁾

In 1913 Niels Bohr, a Danish physicist, developed the latest theory of atom-structure connected with the atomic numbers of the elements in the periodic system. According to this theory, an atom consists of a nucleus which has a net positive charge and a number of electrons which are in orbital motions around the nucleus. The distance between the nucleus and electrons are very large compared to their sizes.

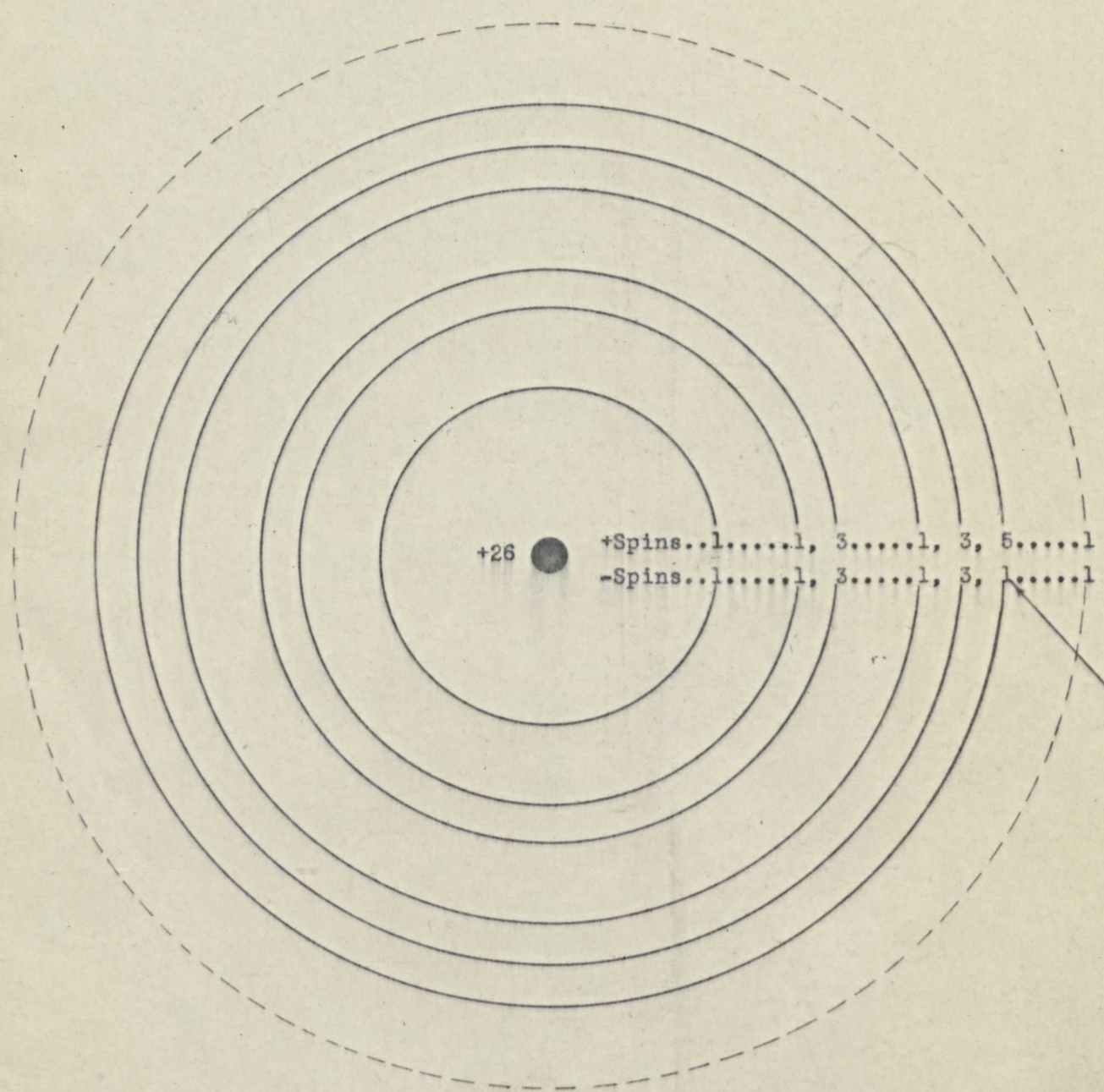
The nucleus is formed of positive charges or protons and some negative charges containing positive electricity in excess. It is connected to the matter and never occurs freely.

- 26) H. Remy, Lehrbuch der Anorganischen Chemie 1937, Band 1, 70-80.
Duff, Physics, eighth edition, 394-396.
- 27) Duff, Physics, eighth edition, 394-396
H. Remy, Lehrbuch der Anorganischen Chemie, 1937
Band 1.

The electrons are negative charges which have an orbital motion around the nucleus and spin around their own axis. The hydrogen atom is the lightest atom whose nucleus consists only of one unit positive charge around which an electron is moving on an orbit. This orbit is called the "K" orbit. The next element with the atomic number 2 is helium, which has two electrons on the "K" orbit while its nucleus contains 2 negative and 4 positive charges. The next atom contains three free electrons; two of them on the "K" orbit and the third one on another larger orbit, which is called the "L" orbit. This orbit is completed with 8 electrons in neon; then the "M" orbit starts to form. So from the hydrogen with an atomic number "1" to uranium with the atomic number "92" new shells are formed and completed periodically. These shells are named with the letters alphabetized from "K" to "P" and are completed by 2, 8, 18, and 32 electrons.

In some cases a new shell may be formed before the outer shell of the atom has been completed. This occurs in the ferromagnetic elements which interests as being one of the causes of ferromagnetism. The Iron Atom and Ferromagnetism:²⁸⁾

In the periodic system iron has an atomic number 26. The figure on page 26 shows that the electrons



ELECTRON SHELLS IN AN ATOM OF IRON*

(The arrow indicates the incomplete sub-shell that is responsible for ferremagnetism. The numbers specify how many electrons with each spin are in the corresponding sub-shells.)

*Bogorth, op. cit., p.2.

are moving in four groups of shells which are divided into sub-shells. The "K" shell has been completed from two electrons as the "L" shell is with 8 electrons. But on the "M" shell, which is sub-divided into three orbits, there are only 14 electrons; and although it has not yet been completed the other two electrons of the iron atom has started another "N" shell.

It has been stated that the electrons have two kinds of motion, one of them a spinning motion, and the other an orbital motion. Both of these motions could give magnetic properties to the atom. The spinings of the electrons on a completed shell neutralize each other in such a manner that half of them spin in one direction while the others spin in the opposite direction. But this is not the case for iron. As it is shown on the figure of page 26, the third shell of the atom has only 14 electrons which are moving on three sub-shells. The outer sub-shell contains 6 electrons, five of which are spinning in one direction, while only one of them spin in the opposite direction. This incomplete sub-shell is considered as a cause of magnetism. Because of the unbalanced spinning on this orbit, the atom acts as a permanent magnet.

The orbital motion of the electrons also contribute to the magnetic moments, but some gyro-magnetic

experiments²⁸⁾ show that there is no change in orbital moments when the intensity of magnetism changes. It is supposed that the orbital moments of the electrons in various atoms neutralize one another.²⁹⁾

The phenomena that the atom itself might be the cause of magnetism was invented first by Weber. Fifty years ago, Ewing attempted to explain the ferromagnetic phenomena in terms of the forces between the atoms. But his model and calculations did not clearly define the forces which maintain parallel the dipole moments of neighboring atoms. Heisenberg³⁰⁾ suggested that these forces were powerful enough to align elementary magnets below a certain temperature which is called Curie temperature. Above this temperature the forces which maintain the dipoles of the atoms parallel to each other are over-forced by the thermal agitation of the atoms. This means that the energy of thermal agitation of the atoms at Curie temperature is equal to the force which keeps them parallel to each other.

It has been discovered that there is an electrostatic and a much weaker electro-magnetic force existing in an atom. In addition to these two forces, a force of interaction exists between the atoms. But this force is negligible when two atoms are two or three times as

far apart as they are in crystals. But it is supposed that when two atoms are brought nearer to each other gradually, these forces cause the electron spins in the two atoms to become parallel (positive interaction). But this fact occurs only to a certain distance; it then diminishes gradually and becomes zero. When the distance is decreased further, the spins set themselves anti-parallel(negative interaction). It has been discovered that different relations exist between the radius of unbalanced orbits and the radius of the atoms for different elements.³¹⁾

When $R =$ the atomic radius of the element
concerned

$r =$ the radius of the shell on which
the spinings are not balanced

then it is shown that:

$\frac{R}{r}$ is larger than $1\frac{1}{2}$ (positive interaction)
for the magnetic substances of Fe, Ni,
Co and gadolinium;

and $\frac{R}{r}$ is smaller than $1\frac{1}{2}$ (negative interaction)
for paramagnetic substances.

This theory has been proved that when a ferromagnetic nickel alloy was held under a pressure of 10,000 atmospheres it loses its ferromagnetic properties.

According to this theory the assumption of the molecular field explains the formation of the domains

readily. To give an idea about the size of the volumes in which this force seems to be effective, we take 1cc iron which contains about 10,000 single crystals; each crystal containing about 100,000 domains, then each domain is formed from 10^{14} to 10^{15} atoms.

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- 28), 29) R.M. Bosorth, The Bell System Technical Journal, January 1940, 1-40.
30) W. Heisenberg, Z.J. Physik, 1938, 619-636.
31) R.M. Bosorth, The Bell System Technical Journal January 1940, 24-25.

Chapter 3

APPARATUS

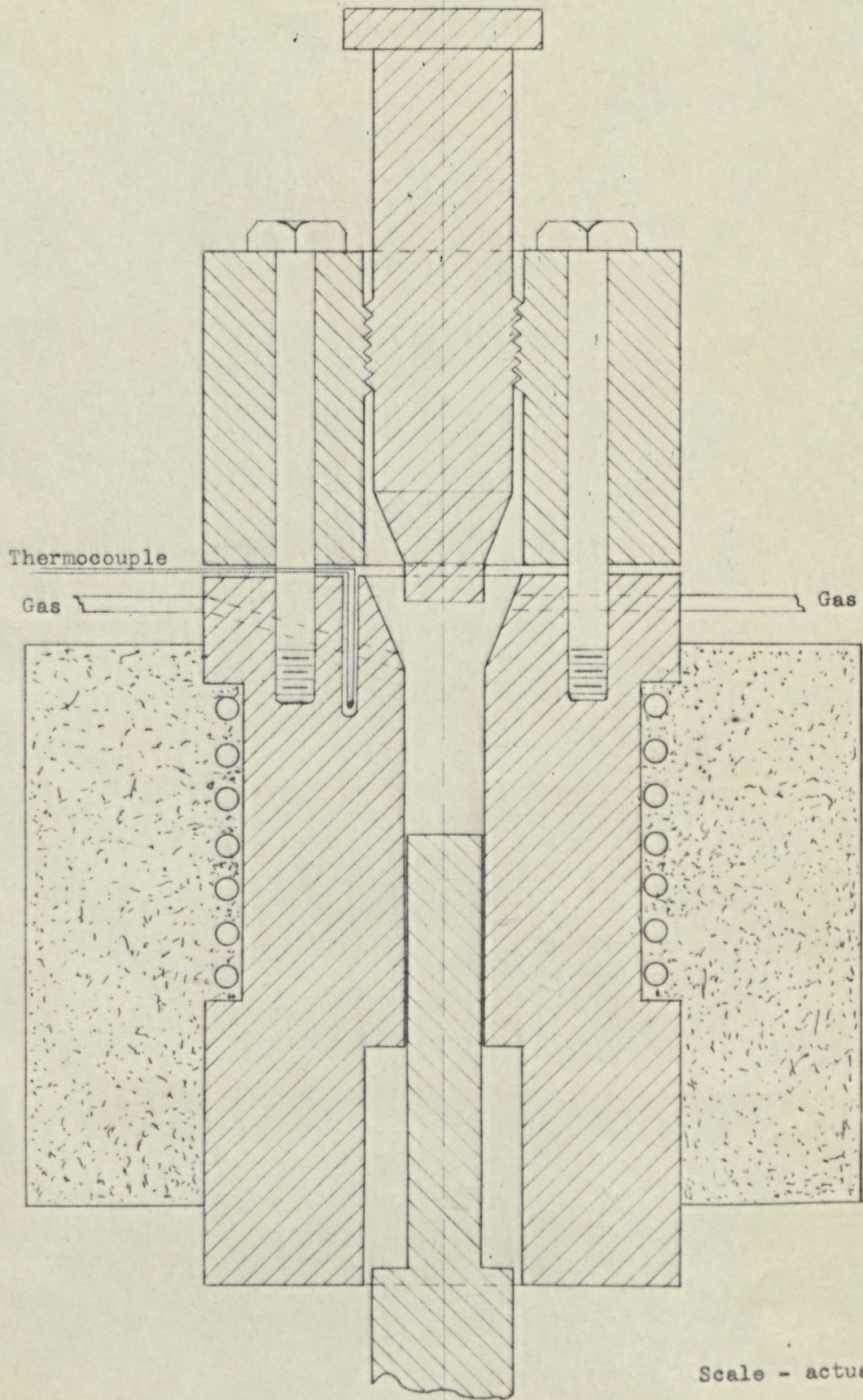
In order to obtain a pellet from the powders of the metals and to measure its permeability, the material was brought into different apparatuses in the following order.

Mixer:

The powders of different metals were weighed in the desired ratio and put into a mixer in order to obtain an aggregate of uniform composition. The mixer itself consisted of a simple tin box, cubic in shape, which had an axis that goes through the box from the center of one edge and continued through the center of the opposite parallel edge. This axis was connected to a small electric motor by means of a narrow rubber belt, as it is shown in picture 1, and was driven with about 40-60- returns per minute.

Mold:

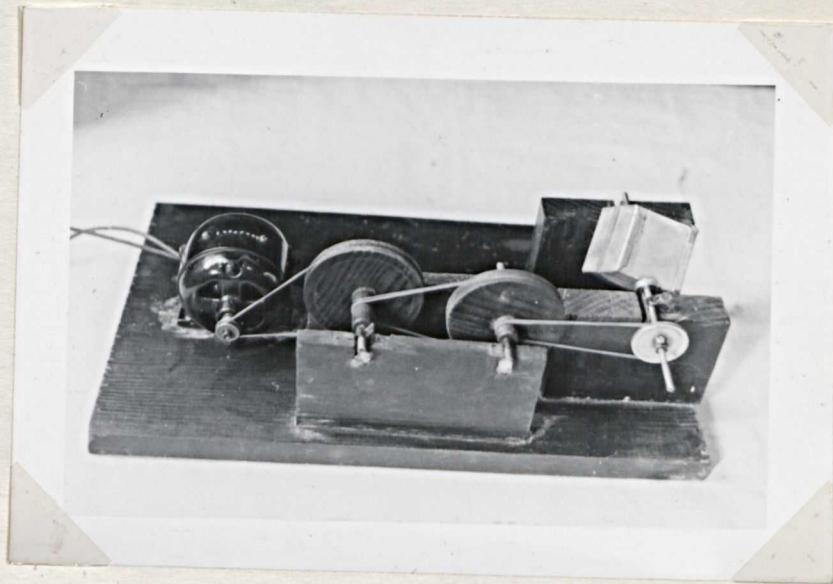
After the powder was mixed, a definite weight of it was put into the mold, picture 2. This mold which is shown on page 32, in its actual size was cylindrical in shape and was connected to various apparatuses which gave the opportunity of controlling different variables of powder metallurgy.



Scale - actual size

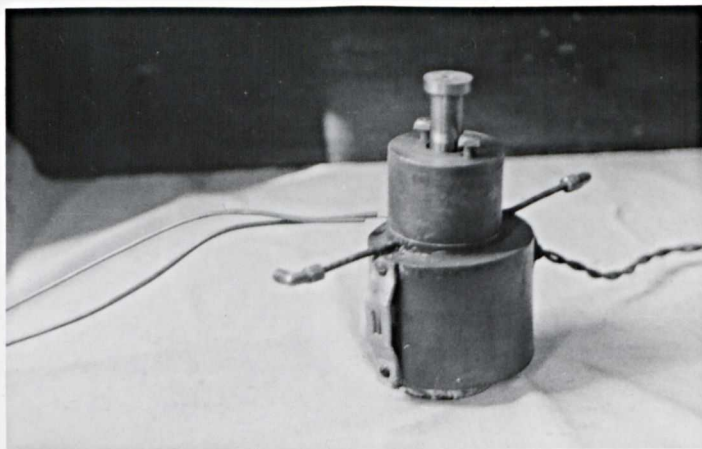
PRESSING MOLD SHOWING REDUCING CHAMBER AND HEATING UNIT

Picture of Mixer

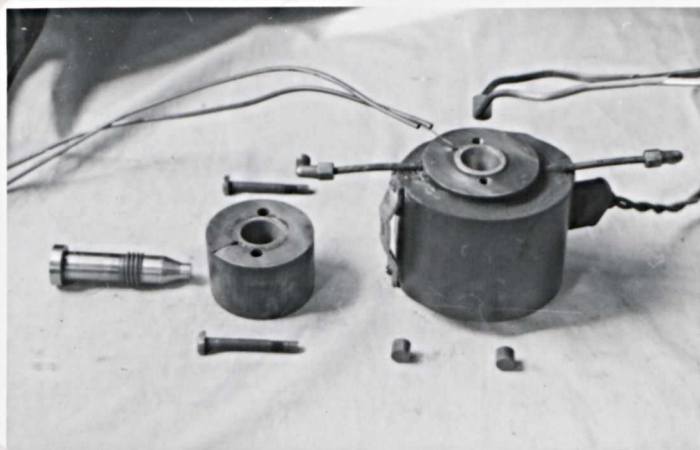


Picture 1

Picture of the Mold



Assembled



Apart

Picture 2

1. Atmosphere Control Apparatus:

In order to remove the oxide film from the particles, it was necessary to reduce the powder mixture in the reducing chamber of the mold which was connected to the natural gas source and to a hydrogen generator. As it is shown on the flow-sheet of page 37 and picture 3, between the reducing gas sources and the mold there were some flasks containing concentrated H_2SO_4 , $CaCl_2$ and $CaSO_4$ for the drying and purification of the gases. The hydrogen and natural gas could be sent into the reducing chamber separately or mixed which were burned after leaving the mold. The natural gas was taken from the city gas lines, while hydrogen was produced by using zinc and H_2SO_4 .

2. Heating Unit:

The mold was heated electrically by means of a heating unit, as it is shown on page 32, around the mold. The temperature was measured with a thermocouple which was regulated before the pressings had been started. The temperature of the mold was increased or decreased by changing the resistance of the circuit.

3. Pressing Apparatus:

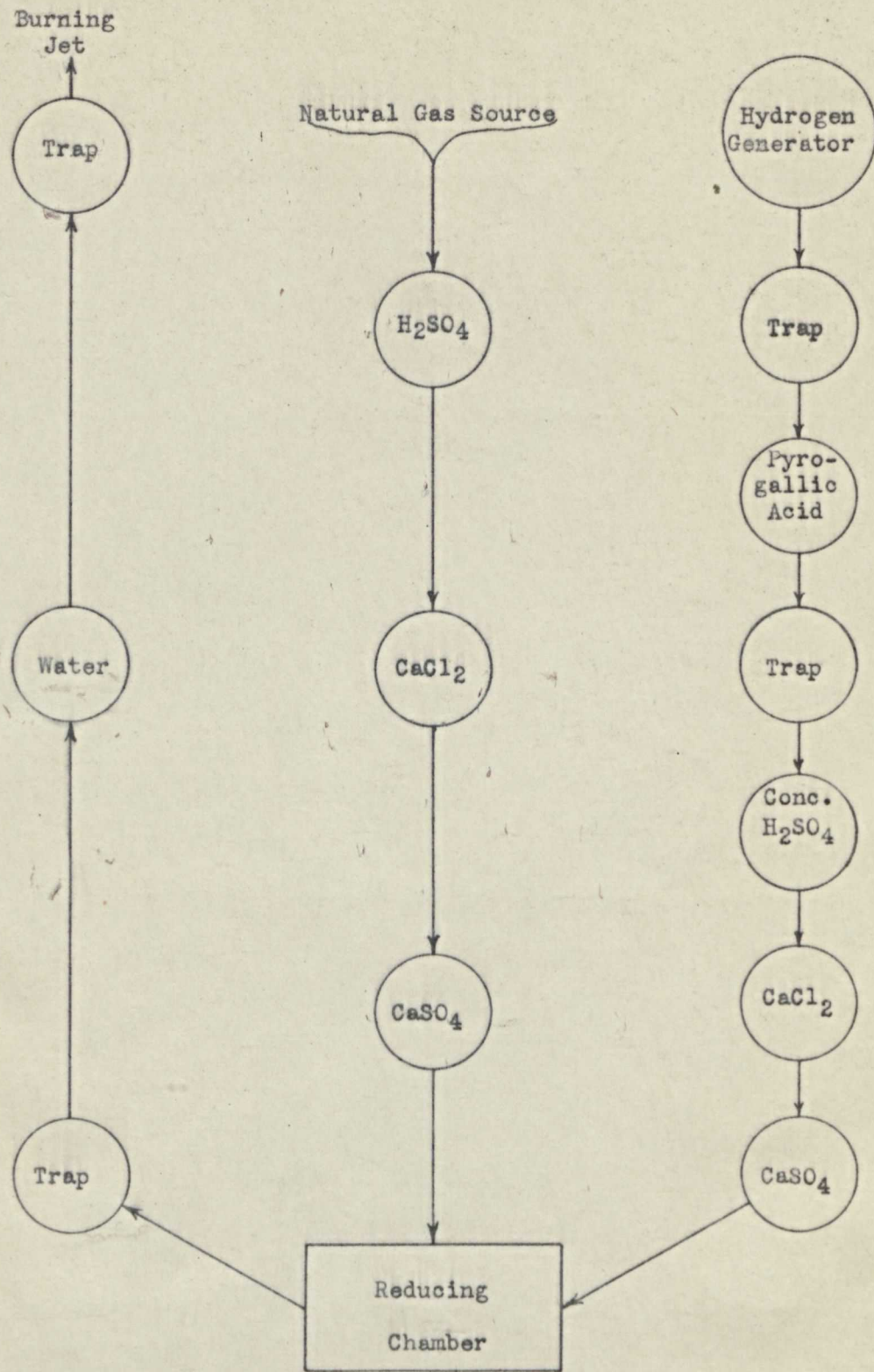
For pressing the powders in the mold a hydraulic jack was placed below the mold as it is shown in picture 4. The presser was made by Blackhawk MFG. Co., Milwaukee, Wisconsin, and had a capacity of 20 tons per a

circular area having a diameter of 2.562 inches. Since the diameter of the ram which transferred this pressure to the powder was only 0.5 inches, its area was calculated 0.1965 sq. in. or practically 0.2 sq. in., then the pressure recorded on the scale of the hydraulic jack was divided by 0.2 in order to find the actual pressure per sq. inch.

Heat-treatment Furnace:

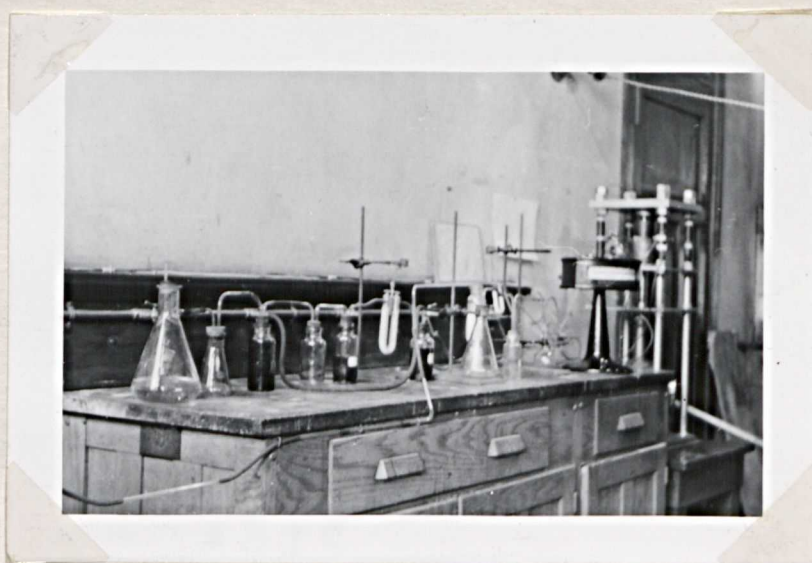
There were two multiple unit electric combustion furnaces for the heat-treatment of the pellets as shown in picture 5. The information about these furnaces and their prices is given in the catalog c-227, of the Central Scientific Co., page 349.

Two of the different variables of heat-treating, such as the temperature and the atmosphere was controlled by means of a thermocouple and by using natural gas. Both of the furnaces were similar in their construction, having a heating unit in which a quartz pipe was placed. The end of the thermocouple was inserted into this pipe through a cork which had another hole for the entering of natural gas. The other end of the pipe was also closed with a rubber cork which had a glass pipe in order to let out the gases coming through the furnace. The gases, leaving the furnace were burned immediately.



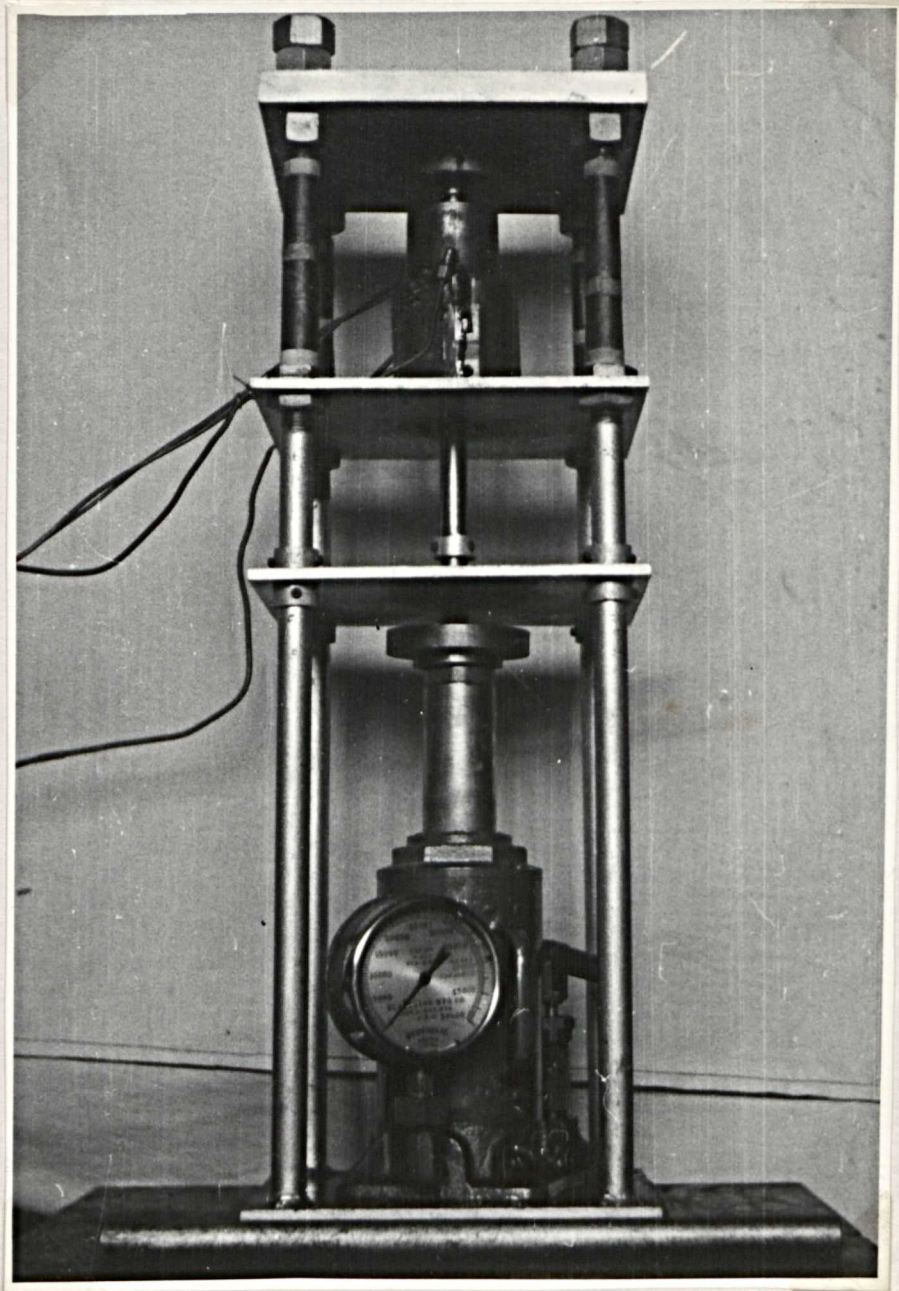
ATMOSPHERE CONTROL APPARATUS FOR PRESSING MOLD

Picture of Atmosphere Control Apparatus



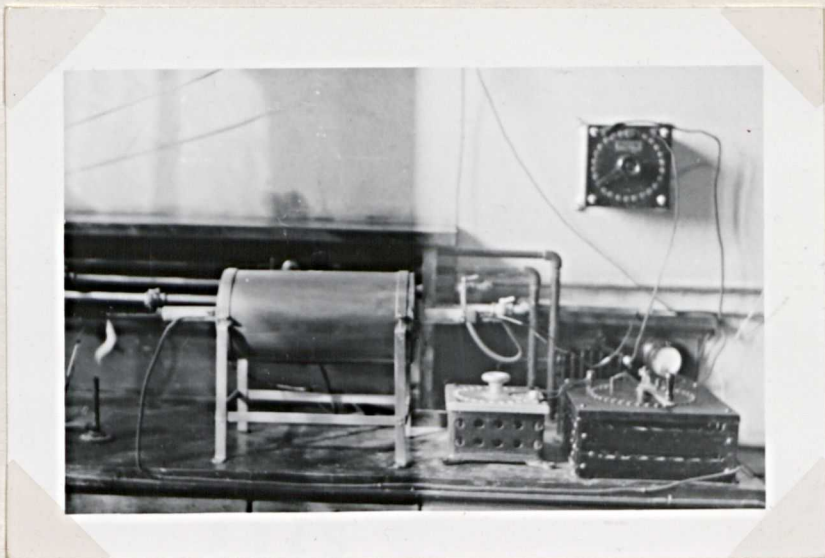
Picture 3

Picture of the Pressing Apparatus



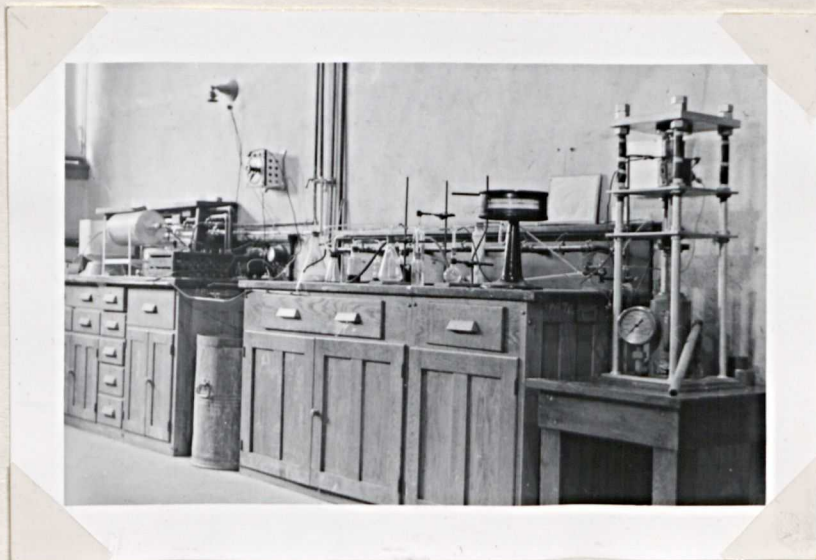
Picture 4

Picture of the Annealing Furnace



Picture 5

Picture of Whole Set-up



Circuit for Measuring Magnetic Susceptibility:

In order to find the magnetic permeability of the pellets, the circuit on page 42 is used. This method of measuring was similar to Faraday's experiment.³²⁾ This apparatus was used by Faraday in order to investigate the relation of B and H; but it also could be applied for the measuring of magnetic susceptibility k, and the relation between k and μ is given by the formula

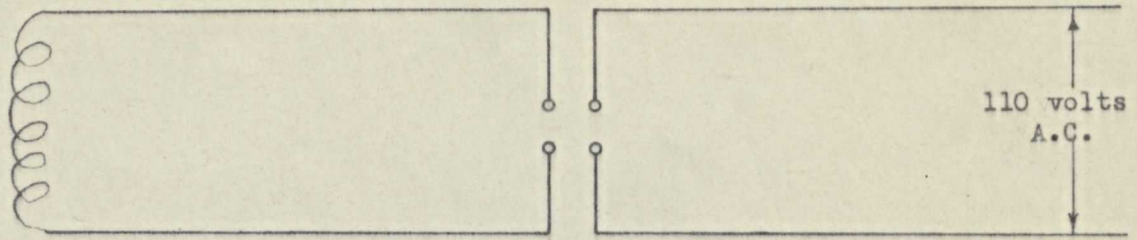
$$\mu = 1 + 4\pi k$$

which is demonstrated in chapter 2.

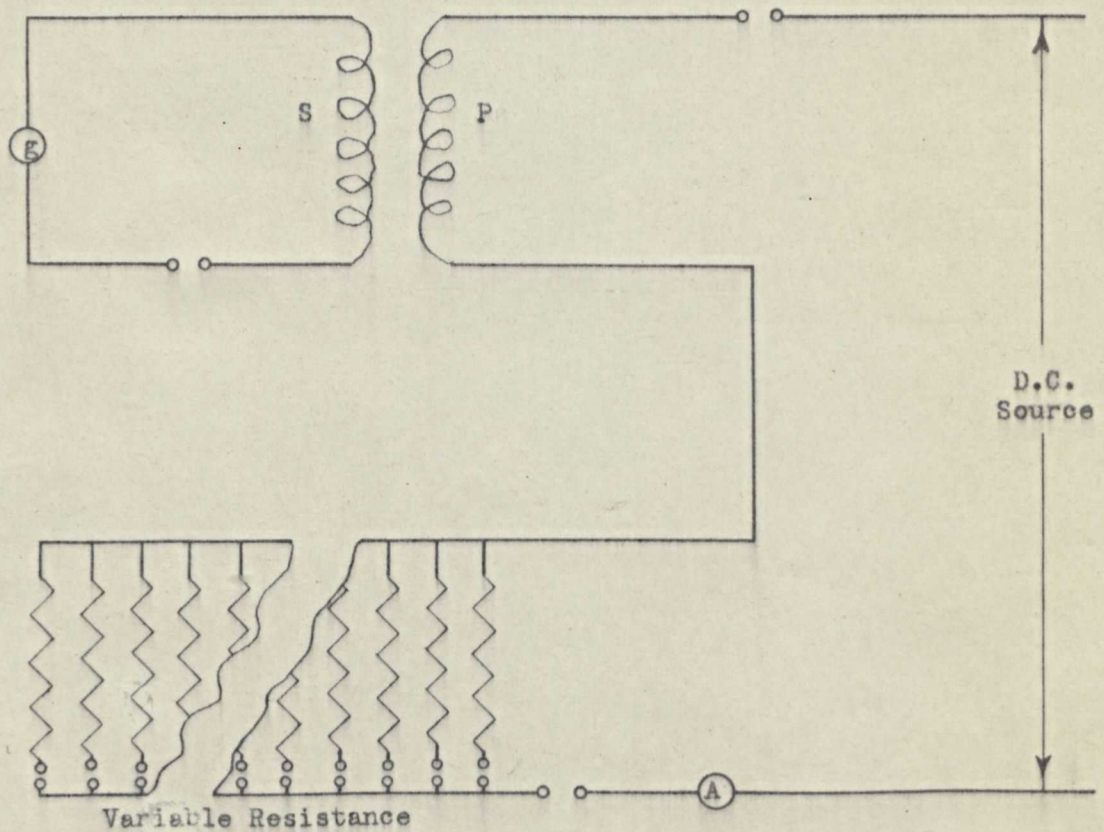
The circuit on page 42 consisted of two coils which are marked on the figure with P (primary coil) and S (secondary coil). The primary coil was connected to a direct current source. In order to regulate the current, an ammeter and a variable resistance was connected to this circuit.

The secondary coil, which was in the same selenoid of the primary coil, was connected to a galvanometer in order to record the changes of the magnetic field in the selenoid.

For the demagnetizing of the pellets before dropping them into the selenoid, a demagnetizer was used which was connected to the alternating current line of Butte.



Demagnetizing Circuit



CIRCUIT FOR MEASURING MAGNETIC SUSCEPTIBILITY (K)

The whole set-up for the magnetic measurements
is shown in picture 6.



Picture 6

- 32) E. Heyn--A. Grossman, Physical Metallurgy, 1935,
Chapter 8.
Duff, Physics, Eighth Edition, 439.

Chapter 4

PROCESS OF PRODUCTION AND MEASUREMENTS

Mixing:

In order to obtain an evenly divided powder mixture of the desired composition, the powders of the metals were weighed in calculated proportions and put into the mixer. After a half an hour of mixing, the powder was put into a beaker and then it was taken out in portions of about 9 grams to obtain a pellet of 0.5" in diameter and 0.5" in height.

Pressing:

The ram of the pressing machine was lifted up to the lower gas hole of the reduction chamber of the mold and the powder previously weighed was put into it. After the lid of the mold was made gas-proof by means of heavy grease and was securely tightened, by connecting the switches the temperature was increased to a desired point; and in the meantime, either natural gas or hydrogen or both was sent into the mold which passed through the powder and was burned after leaving the reducing chamber. After the powder particles had been reduced for a desired length of time and the temperature reached the desired point, the ram was dropped so that all the powders from the reducing chamber could fill the pressing chamber of the mold. The lid of the mold was then twisted lower

to prevent the reentering of the powder back into the reducing chamber when it was being pressed. The next step was the increasing of pressure up to a certain point by means of the jack mentioned in chapter 3. The pressure was then removed 2 minutes later, and the pellet was taken out of the mold. After this, the mold was cleaned and greased in order to be prepared for the next pressing.

Measuring the Magnetic Permeability:

After a number of pellets had been produced, their magnetic susceptibilities were measured by dropping the pellets into the selenoid and by comparing the readings for a soft iron pellet and for the pellets produced.

The primary coil which contained about 100 returns, produced a magnetic field in the selenoid by using a direct current of 1.5 amps. which was much higher than the magnetic saturation point of iron. The galvanometer for iron measured about 19.5. From the formula of

$$\mu = 1 + 4\pi k \text{ for soft iron and}$$

$$\mu_1 = 1 + 4\pi k_1 \text{ for the pellet was compared as}$$

$$\frac{\mu - 1}{\mu_1 - 1} = \frac{k}{k_1}$$

from the equations

$$\frac{k}{k_1} = \frac{\text{readings for soft iron}}{\text{readings for pellet}} = \frac{\text{permeability of soft iron} - 1}{\text{permeability of pellet} - 1}$$

The permeability for soft iron was taken 100 and for the pellets which were pressed from the powders were calculated, and the records were carried on the tables as they are shown on the following pages.

Annealing:

When the magnetic permeabilities of the pellets were recorded, they were put into the annealing furnaces under a reducing atmosphere and were heated to a certain temperature and annealed for two hours. After the allotted time the furnace was disconnected of the electrical current and was cooled slowly until next day. The pellets were removed after cooling the furnace and their permeabilities were measured as before. The same pellets were then annealed at higher temperatures.

Chapter 5

EXPERIMENTAL RESULTS

The experiments made can be classified into two different groups:

1. Those which are practiced in order to investigate the effects of some variables of powder metallurgy on the permeability of the pellet obtained. This group work was done to become acquainted with powder metallurgy and with the apparatus used.

2. The investigations of the permeabilities of the Fe-Mn alloy system produced by the methods of powder metallurgy.

The investigations for group 1 is made for an alloy containing

Fe	73.4 %
Cr	9.0
Co	16.0
Mn	0.3
Mo	1.3

and the effects of some variables recorded as follows:

Time of Pressing:

Only a few pressings of a various length of time have shown that the pressing longer than two and a half minutes does not effect the permeability of the material to a large extent.

Temperature of Pressing:

For this group of investigations the powder of

the above composition was pressed. It was first reduced for a half an hour while the temperature of the mold was increased up to a certain point desired; then the powder was pressed for two minutes at a pressure of 30 tons per sq. inch. After the pellets were produced, their magnetic permeabilities were measured as it is shown in chapter 4. Table 1 shows the variation in permeabilities parallel to the different temperatures of pressing.

Table 1

Temp. of Pressing in °c	Permeability Iron = 100	Permeability after Annealing Iron=100
Room temp.	76	78
70	78	78
100	78	80
150	83	90
200	88	90
250	93	93
300	98	100
350	99	100
400	100	100
450	100	100

Annealing:

When the permeabilities had been recorded, the pellets were put into the annealing furnace and annealed

for about two hours at 700°C under a reducing atmosphere of natural gas. After cooling slowly, the permeabilities were measured again. Table 1 shows that the annealing at 700°C did not affect the permeabilities to a large degree.

As it was mentioned before, these experiments were made only to become acquainted with the equipment and with the methods of powder metallurgy.

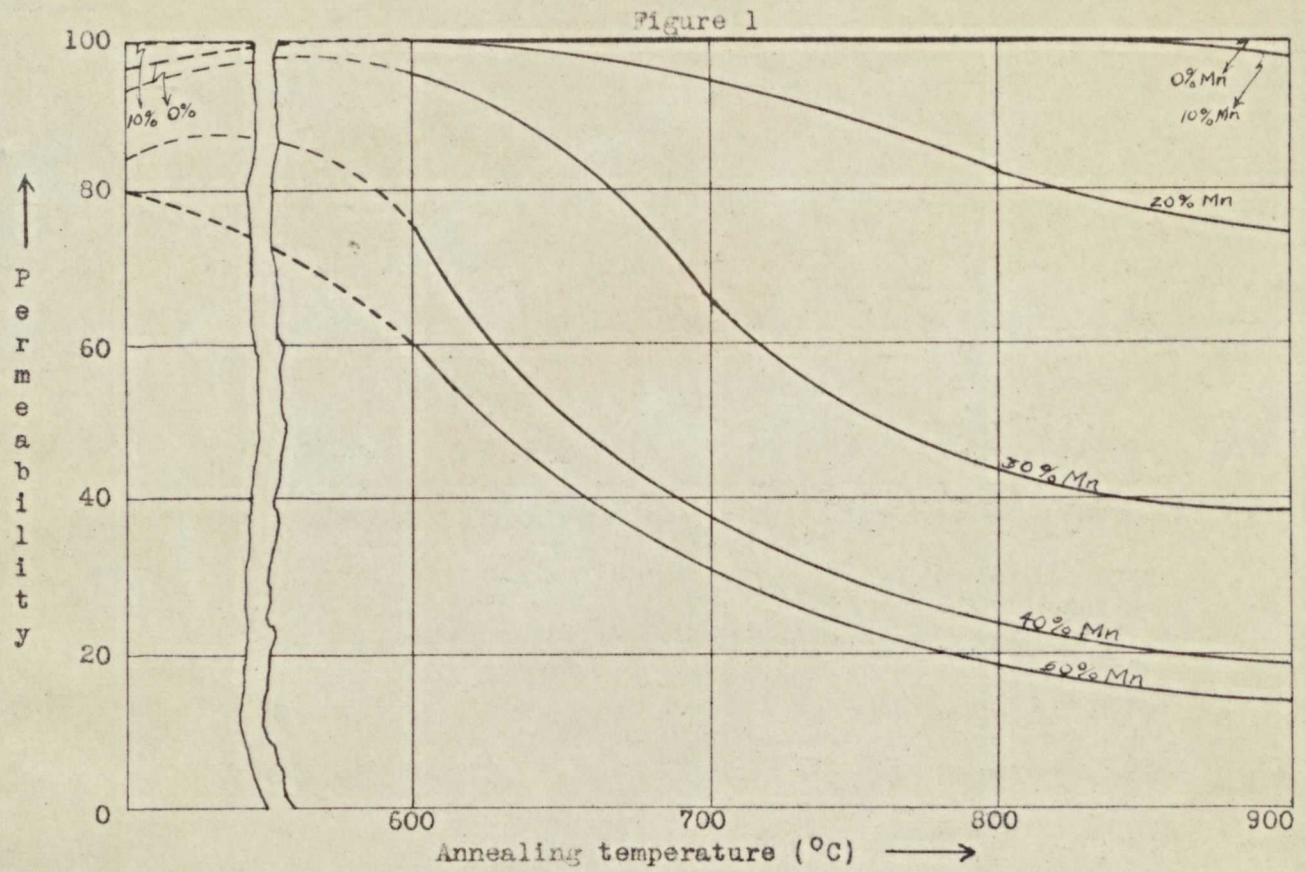
The investigations for group 2 were made in order to find the magnetic permeabilities of the Fe-Mn alloy system of powder metallurgy. The powders of iron and manganese were mixed in different compositions containing from zero to fifty per cent manganese and balance iron. From each composition two pellets were pressed at 400°C and 40 tons per sq. inch pressure for two minutes. After their permeabilities were recorded, one pellet of each composition was annealed at 600°C in a reducing atmosphere for approximately two hours. After cooling slowly, their permeabilities were measured and the same pellets were again annealed at 700°C for two more hours. In this order the permeabilities were carried on the tables as it is shown in table 2.

Figure 1, which is taken from table 2, shows the effect of the annealing temperature when the composition is held constant. The decrease in the permeability by in-

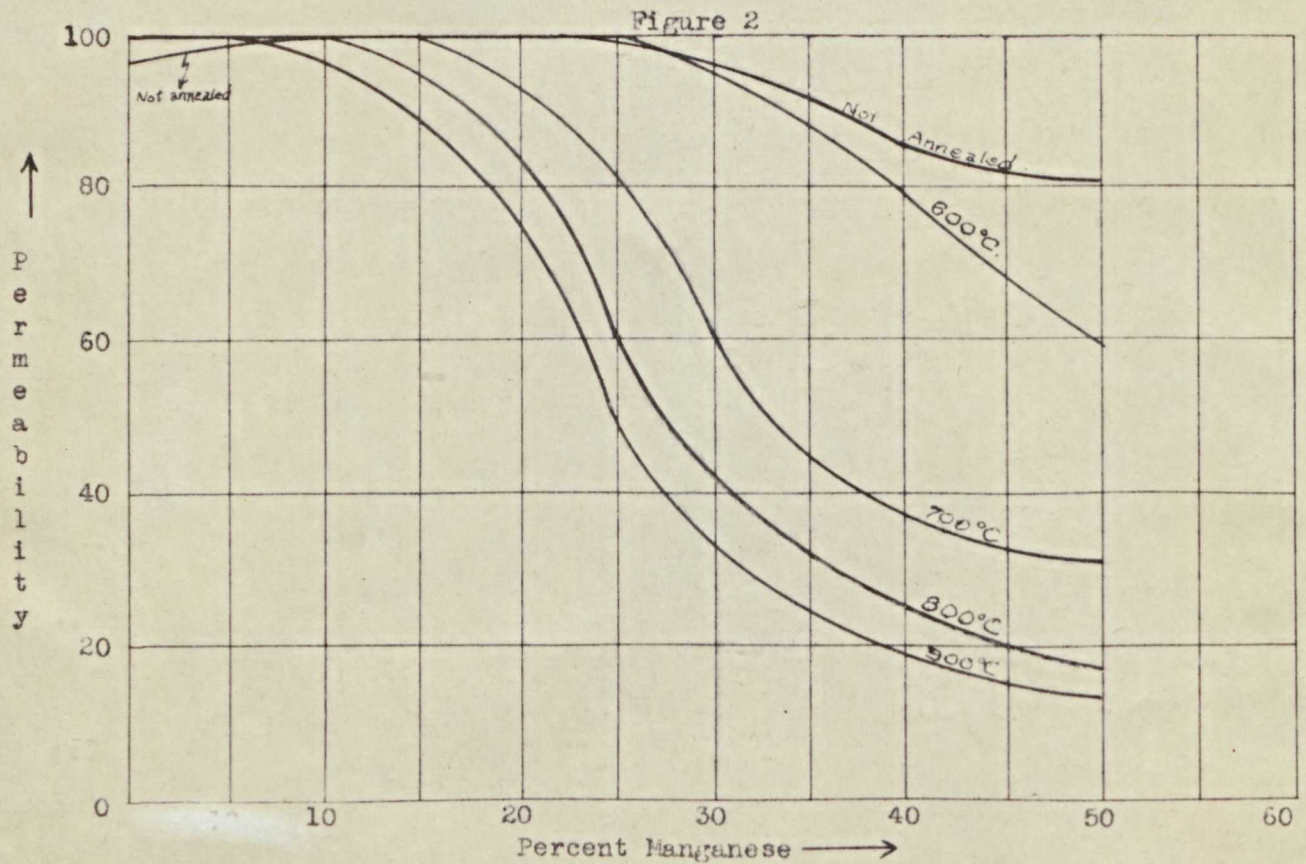
creasing the temperature of annealing can be explained by the fact that the diffusion of manganese is increased with the temperature which decreases the permeability of the product. Figure 2 is also taken from table 1; here the temperature of the heat-treatment is constant while the percentage of manganese is varied. As we can see from this figure, the increasing content of manganese decreases the permeability. The explanation can be only the paramagnetism of manganese. Another particular fact in this curve is the steep slope which results when the alloy contains more than 15% Mn. The reason for this can be explained as follows: Manganese depresses the critical temperatures of iron; and iron is Austenitic at ordinary conditions when an iron manganese steel contains more than 12% manganese. Since Austenite is paramagnetic, the formation of Austenite decreases the permeability of the product.

Table 2

Per cent Mn	Before anneal.	<u>after annealing at:</u>			
		600°c	700°c	800°c	900°c
0	97	100	100	100	100
5	98	100	100	100	99
10	100	100	100	100	97
15	100	100	100	97	92
20	100	100	93	82	73
25	100	100	76	63	53
30	99	97	63	48	39
35	93	88	50	33	27
40	85	78	37	22	19
45	82	68	34	18	14
50	80	61	32	17	13



THE EFFECT OF ANNEALING ON THE MAGNETIC PERMEABILITY OF THE Fe-Mn SERIES



THE EFFECT OF MANGANESE ON THE MAGNETIC PERMEABILITY OF THE Fe-Mn SERIES

CONCLUSION

These experiments have shown that it is possible to produce a manganese-iron aggregate by the methods of powder metallurgy with a magnetic permeability of a desired degree. The experiments made could not be applied to the Mn-Fe alloy system containing more than 50% Mn because of the necessity of a higher pressure and temperature for increasing the plasticity of manganese which could not be practiced in the apparatus used.

The metallic compacts obtained could not be annealed at higher temperatures because of the decomposition of natural gas which decomposes to carbon and hydrogen. The danger of carburizing the metal with this carbon at higher temperatures did not allow the use of a higher annealing heat. It was possible to have a reducing atmosphere with hydrogen for higher temperatures; but it was not practiced because of the danger of explosion. Thus these factors limited the extension of the experiment; the results obtained were satisfactory, and changes in the permeabilities could be explained depending upon the theory of diffusion or the depressing effect of manganese upon the critical temperatures of iron.

Acknowledgment

I wish to express my sincere thanks to Dr. C.L. Wilson, Head of the Department of Metallurgy, under whose direction this work was done.

O. Cihad Onbulak