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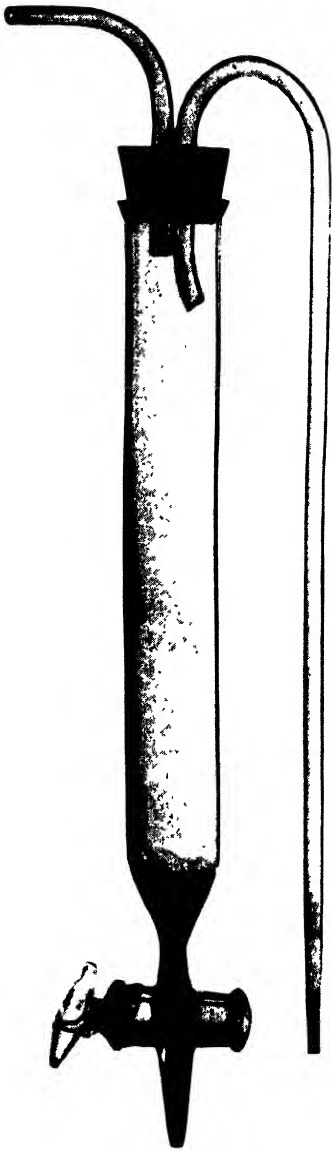
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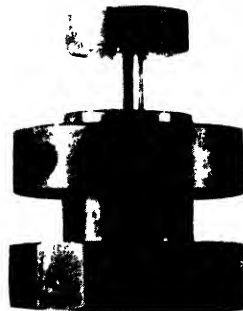




Sedimentation Pipette



Break Tester



Die

Apparatus for the Powder Metallurgy Laboratory

A COURSE IN  
**POWDER  
METALLURGY**

By

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Engineering, Science, and Management War Training  
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## Chapter 1

### Introduction

In attempting to write a book on powder metallurgy, it is necessary to limit the interpretation of the term very carefully, and here an immediate difficulty arises. We are faced with a subject in a state of flux, and a definition that might well cover powder metallurgy today, by the time of publication might exclude a form of operation which quite obviously belongs within the field. We cannot hope to cover all aspects of the subject. For many generations most of the metals produced in powdered form were employed in the paint industry, and any discussion of powder metallurgy would necessarily have included a large section on pigments; now, however, most powder metallurgists consider pigments as outside of their field. In short, powder metallurgy has become a specialty in its own right.

On the other hand, certain processes now fall into the general classification of border-line cases, and the question arises whether or not they properly belong in a book on powder metallurgy. For example, powder metallurgists have recently made intimate mixtures of powdered oxides with reducing agents and have processed these to yield a unique metallic product. It may well be that in a few years such processes will occupy a large proportion of the time of specialists in the powder metallurgy field. In view of this ever-changing picture, the difficulty of writing a satisfactory definition of the term becomes apparent.

#### Definition

Powder metallurgy is the technique which produces metals or alloys in a finely divided state; or which produces metals and alloys in ingot or crude form from powdered metal, alloy, purified oxides or salts, employing temperatures well below the fusion point of the

product. The present significance of powder metallurgy is its ability to produce useful metal-like products to exact dimensions without fusion or machine operation.

Naturally the definition includes the production of the raw material which is the basis of all powder metallurgy processes, namely, powdered metals. It includes the production of ingots of rough shape from powder, for there are a large number of products which cannot be made commercially into workable form without going through the powdered metal stage. And finally it includes the production of useful shapes to exact dimensions from powdered metals and combinations, including metals, alloys or non-metals.

The importance of powder metallurgy lies in the ability of this technique to produce complicated metal shapes within a close limit of tolerance by a process requiring much less time and skill than machining, and much lower temperatures and fewer finishing operations than casting. Moreover, the products of powder metallurgy may possess all the physical characteristics of those made by the older methods, as well as many which cannot be obtained thereby, for instance, controlled porosity.

It is impossible under war conditions to do more than hint at the most recent industrial developments in this field, since many of these are taking place under Government supervision and at the present time cannot be broadcast. However, the need for a book on this subject is especially great under war conditions, since more and more men with a knowledge of the subject are required to promote the war effort.

## Chapter 2

### History and Modern Applications

Classically, powder metallurgy dates from the early experiments of Wollaston, who as far back as 1829 disclosed the method he had employed for many years to produce malleable platinum. He compressed a finely divided platinum powder in a toggle press and heated the compact so formed in a crude blast furnace. Subsequently this was hot-forged. Essentially, this is the basic process employed today by powder metallurgy. Wollaston's was the forerunner of many sporadic experiments which employed the same principle, but these experiments produced only laboratory curiosities. It was not until five years before World War I (1909), when the Coolidge patent for the production of tungsten wire for lamp filaments was granted—which became of commercial importance a few years later—that powder metallurgy reached its adolescence. Shortly after the war, the first porous bearings were marketed.

#### Early Products

In a broad sense powder metallurgy may be said to extend beyond historic record. Gold, copper and bronze powders and many oxides, particularly iron oxide and what are generally classified as the pigments, were used for decorative purposes in ceramics and painting as well as for beautifying the person as far back as our knowledge goes. The production of powders for pigments continues in inks, paints and ceramics today. Some of the earliest manuscripts known are illuminated with gold. Egyptian implements made of iron are known to date from 3,000 B.C. and the Delhi Pillar, a famous monument of India which weighs  $6\frac{1}{2}$  tons, dates from about 300 A.D. Until 1800 there was no way of obtaining temperatures high enough to melt pure iron so that it could be molded or poured to form such a monument. It is doubtful that any ingot iron of any

kind was formed before then, as it was impossible to get sufficient heat to the ores by known methods to reduce and melt them. Examination of the Delhi monument shows that it was made from a sponge iron, beaten and worked into solid form, and sponge iron always originates in powder or a very friable, highly fretted powder form. The early Egyptian implements, too, were formed by hammering iron sponge.

### **Wollaston's Experiment**

About 1829, Wollaston told how he produced a solid ingot from platinum sponge. He used ammonium chloro-platinate and reduced this with a carbonaceous material, probably sugar. The black powder which he secured at this stage was washed and compressed and the compact he drew from his press was fired to redness and hammered while hot. This gave a product which could be forged and worked to form.

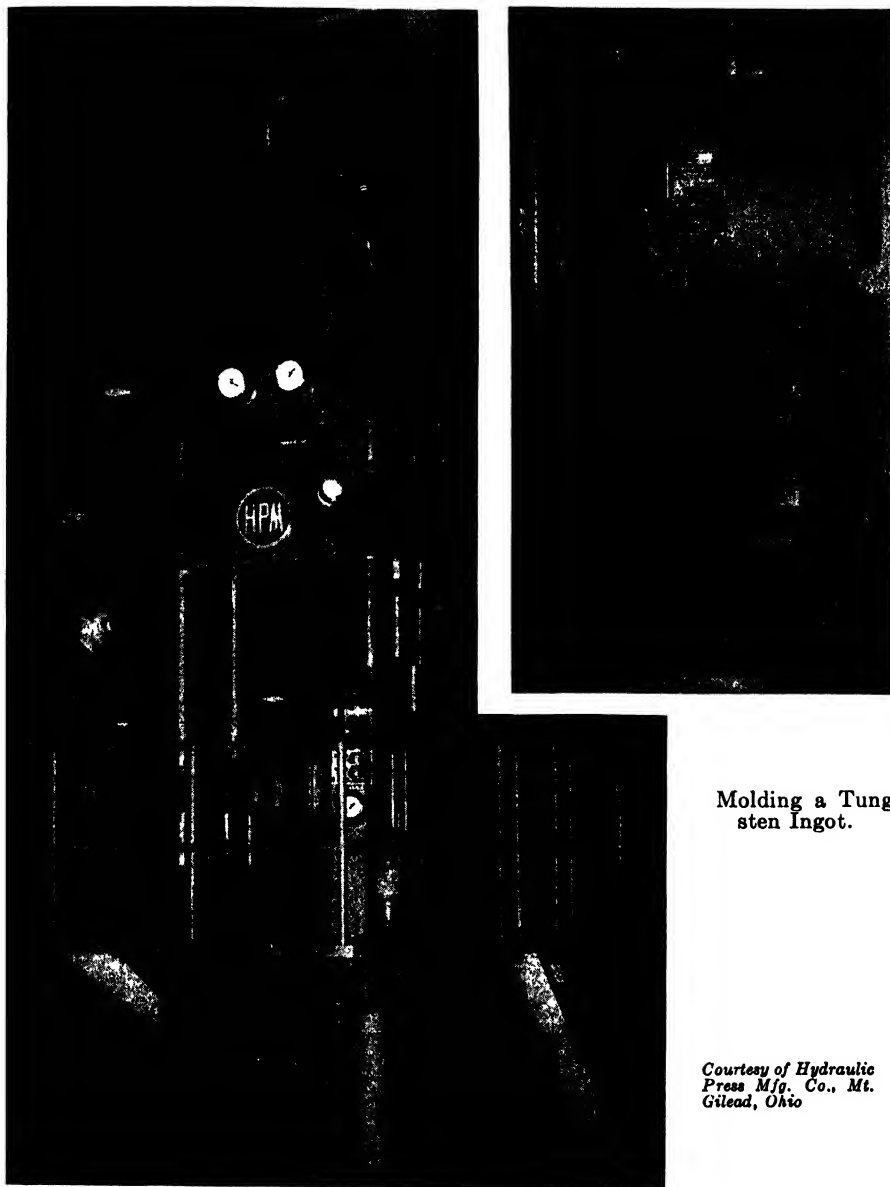
### **Catalysts**

Around 1900 great interest was shown in catalysts, the effectiveness of which in theory depends in large part upon the surface area that can be presented for reaction; thus powders, which afford the greatest amount of surface, became of great interest. From 1900 to 1915 nickel powder was the focus of much experimental work; it is now produced in great commercial quantities to be used as catalysts for the hydrogenation of oils.

Coolidge made the next great contribution to this field.

### **Tungsten**

It had long been evident that tungsten would make an ideal filament for the electric lamp, but to work tungsten into the necessary fine wire was beyond conventional metallurgy. When finely divided tungsten was compressed and heat-treated at temperatures well below the melting point, ingots were formed which could be drawn into wire finer than one thousandth of an inch in diameter and having tensile strength close to 600,000 pounds per square inch. This stimulated the scientific imagination: perfect welding had been obtained without fusion. Interphase cohesion was greater than might have been anticipated through fusion and interlocking crystallization.



Molding a Tungsten Ingot.

*Courtesy of Hydraulic Press Mfg. Co., Mt. Gilead, Ohio*

### **High Tensile Strength Without Fusion**

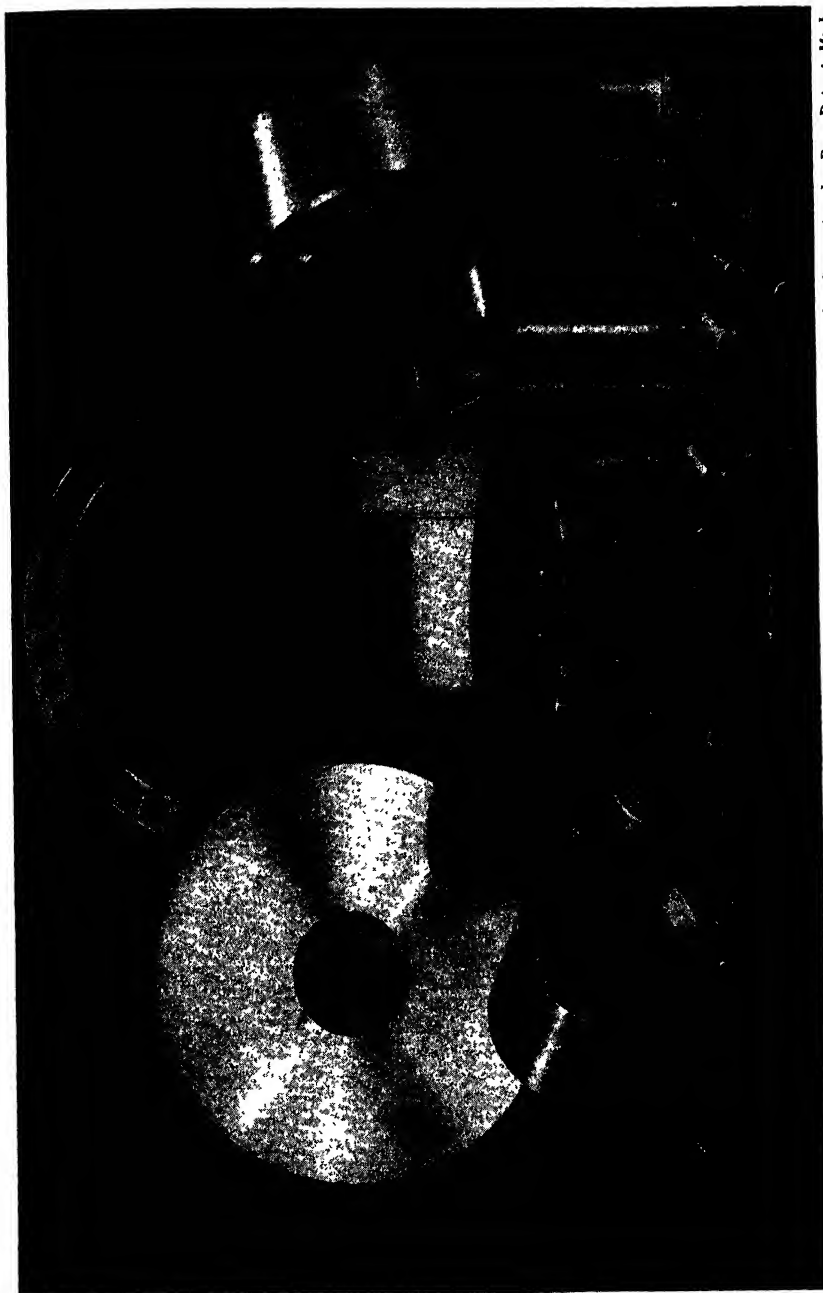
Since then, a similar phenomenon has been observed innumerable times. Indeed, one whole class of powder metallurgy work has resulted from the knowledge that it is possible to obtain products of very high tensile strength from materials melting at such high temperatures that no commercial furnaces can be built with sufficient heat insulation to be practical for fusion. The fact that excellent products can be had from powdered metals well below the temperature of fusion has been an invaluable aid in the production of high-melting-point metals.

Coolidge's earliest patent dates from about 1909; in this he claims a process of compacting tungsten powder, heat-treating the compact and subsequently work-hardening the red-hot briquette. It was not until about 1916 that tungsten wire made by this process became a common commercial product employed universally as a lamp filament in the pressure-filled electric bulb.

The dramatic effect of Coolidge's work lies in the fact that he had obtained a product of very high tensile strength (590,000 lbs. per sq. in.) from a metal which had not been fused. Metallurgists had taken it for granted that what gave tensile strength to a product was the interphase and interlocking of the crystal growth as a fused mass cooled to solid form. Yet although the tungsten product had never been heated to a point where it could fuse and recrystallize, it possessed greater tensile strength than that of metal products made in the usual way.

### **Porous Bearings**

At about this time the oilless bearing obtained commercial significance, and here was another product which, made by the new technique, exhibited physical characteristics not obtainable by usual methods. The bearings produced at that time did not have the strength of those that required oiling, but they could be made sufficiently strong to serve many useful purposes and could not be burned out, as they were self-lubricating. They were made by producing a metal mass which was porous, the pores being subsequently filled with oil under pressure. The openings were capillary in character, and with the increased heat set up by the moving part within the bearing, the oil flowed freely to the bearing surface. The weight on the bearing also aided the lubrication.



*Courtesy of Chrysler Corp., Amplex Div., Detroit, Mich.*

**Oilite, Super-Oilite and Iron Oilite Bearings and Machine Parts**



One variation of this porous bearing is a product which is part metal and part graphite; another, for a heavy bearing, is a porous form designed with an ordinary oil cup led into the outer surface of the bearing to permit a constant feed of oil so that the bearing, while not oilless, will remain lubricated long after the last oil in the cup has been fed to the porous metal part.

### Cutting Tools

Cemented carbides from which high-speed tool bits are made take advantage of the powder metallurgy technique. In drawing tungsten wire the wear on the dies was so excessive that many efforts were made to find a material hard enough to increase their useful life. Diamond dies are very expensive. Tungsten carbide was known to have the necessary physical strength, but it could not be machined because there was no material sufficiently hard to cut it. It was found, however, that if tungsten carbide was ground to a powder and a small quantity (from 3 to 13 per cent) of cobalt added, the product made roughly to the dimensions of the die could be molded, and that when heat-treated at fairly low temperatures, it would reach dimensions which subsequently would retain their shape. Allowance could be made for shrinkage. The carbide was intimately mixed with the cobalt, the rough shape formed and heat-treated, and at this stage it was sufficiently soft so that it could be machined into final shape. Upon further heat treatment the resulting product was strong enough to resist wear many times longer than any previously known metallic product.

There followed the production of tool bits to cut hard alloys more rapidly than had been possible previously. Such bits today are made of tungsten carbide, tantalum carbide or molybdenum carbide, or combinations of these with 3 to 13 per cent of cobalt or nickel, or both, as the cementing medium. The significance of this development may be appreciated when we consider that before the advent of high-speed steel tools ordinary tool steel could cut metals at the rate of 14 to 20 feet a minute before they became so hot that they lost their cutting edge. With the advent of high-speed tools the rate could be increased to 50 feet per minute, but Carboly and the other cemented carbides can be run at a speed of over 600 feet per minute. It has been said that Hitler's war program reached its stage of preparedness two years sooner than would have been

possible had the factories of Germany not been tooled almost exclusively with carbide products. It is easy to believe this when one recognizes that, using the same turret lathe slightly modified and replacing the tool bit in common use with a cemented carbide bit, the same turret will produce 12 times as many products in a given time.

The life of dies made from carbides is some 60 times as long as those made from ordinary steel. It was noticed by those familiar with the production of cemented carbides that during the sintering period the original compact shrank some 30 per cent from its original size. Since no material was lost, this could only result in a great increase in density.

### **Heavy Metal**

For a number of reasons it is useful to have a heavy metal. Heavy metals may be used for balancing moving parts. It is possible to add to a rapidly revolving wheel a very small piece of heavy metal close to one part of the circumference in order to correct any minor unbalance.

It is possible to store radium in a relatively small heavy-metal safe, whereas storing it in lead requires a very thick-walled safe. There are a number of other uses for this product. With the evidence of the shrinkage in the production of carbides, metallurgists used this as a key to find the solution to the problem of producing heavy metal. Such metals have been made, the best known of which is roughly of the composition tungsten 90 per cent, cobalt 5 per cent, nickel 5 per cent. This has a specific gravity of 17, exceeded only by a few expensive or rare metals such as gold, with a specific gravity of 19; osmium, with a specific gravity of 22; and platinum, with a specific gravity of 21. Lead, which had previously been used, has a specific gravity of only 11.3.

### **Current Collector Brush**

At about this time there came a new development in current-collector brushes. The common collector-brush was made by graphitizing at high temperature powdered carbon bound with pitch. The pitch volatilized at the temperature of graphitizing. Increased conductivity could be obtained by using copper powder to replace the binder. The copper sintered and both bound the graphite and added to the conductivity of the brush.

With the development of this product an entirely new principle is introduced to metallurgy. This is the discovery that by powder metallurgy techniques two materials of divergent characteristics may be combined to give a single product which retains the desired characteristics of both. The same phenomenon occurs in the graphite bronze bearing, where tin, copper and graphite powders are mixed, formed to shape, and sintered, to produce a graphite bearing bronze. The bronze takes the weight of the load and holds a pinion in place, and the graphite lubricates. But the significance of combining divergent materials is emphasized in the collector-brush.

A laminated brush made up of sections of copper bound to sections of graphite is made by employing flake in place of regular copper powder. If the green compact is formed by pressing at right angles to the direction in which the current is to flow when the brush is in use, the brush will have much lower electrical resistance in the direction of the current flow. Advantage is taken of this to produce current collector-brushes with very low resistance.

### **The First Small Part**

About 1937 a man with no training as a chemist, metallurgist or engineer, but with deep annoyance at the trouble he was having with the door snubbers on his automobile, made a contribution to this field which has given it its modern significance. At that time, if a door snubber was not properly oiled it squeaked, and if it was oiled, the oil subsequently smeared the clothing or the hands of those entering and leaving the car. It occurred to him that if the metal in the snubber could be combined with graphite as a lubricant, the snubber would remain permanently free of squeaks and the lubricant would not damage clothing.

Iron and graphite were mixed and a product similar to the oilless bearing was formed in a single molding operation. Something like a year after these parts had become standard equipment on cars, one of Ford's engineers discovered that the molding operation had taken the place of 17 machine operations. Simple as they may seem, these door snubbers are of a rather complicated shape. If 17 operations could be eliminated from a door snubber, were there not other parts which could be made by the powder metallurgy technique, which would eliminate the work of many skilled mechanics?

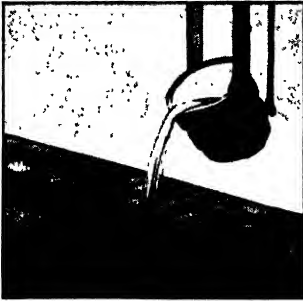
### **The Molded Gear**

An outstanding product based on this line of reasoning is perhaps the gear of the oil pump, which has become standard equipment on practically all automobiles and was first introduced on one of the General Motors cars. In its simplest form this oil pump consists of two involute gears in a closed housing; the teeth mesh and push the oil under positive pressure to the parts where lubrication is required. It is of extreme importance that the teeth be accurately formed or the pump will be very inefficient. Only very skilled machinists can be trusted to make these gears from die-castings, but any high-school boy can fill a hopper and operate a press which can turn out hundreds of these gears with greater accuracy than the best machinists can produce a few. Gears made by this technique require only a minor finishing operation which removes but a few thousandths of an inch of excess metal, and the teeth more nearly fulfill the theoretical requirements of a perfect involute gear than those produced in the ordinary way. In addition to this, but a fraction of 1 per cent of metal is waste, whereas in cutting a machined gear from a cast blank, 64 per cent of the metal is lost in chips.

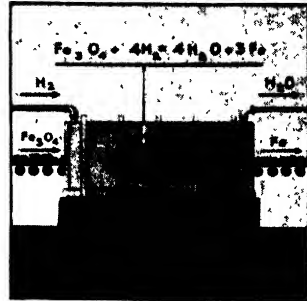
It must not be thought that a great saving has been made in the cost of production, for although 64 per cent of the cheap raw material is wasted in cutting gears, and although many skilled man-power hours are saved per unit of product, the cost of the raw material used in the powder metallurgy technique is very much greater than that of a die-casting. In actual practice, with iron powders costing around 60¢ a pound, the cost per unit is almost identical. On the other hand, as we shall see subsequently, there is every reason to believe that satisfactory iron powders can be made for less than 10¢ a pound, and it is probable that since the curtain of war has hidden recent developments products which require such close tolerance as the gear are already being made from iron powder in this cost range. It is certain that very many somewhat cruder parts are being produced from low-priced iron powder. The developments, beginning at about the time of the snubber, came so rapidly that it is difficult if not impossible to list them chronologically.

### **Double Character Products**

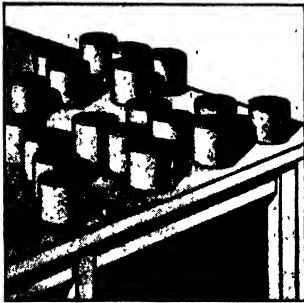
Contact points were made, taking advantage of the fact that metals with two very dissimilar characteristics can be combined to



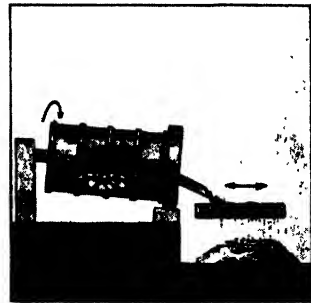
Molten metal is cast into pigs. But the iron still contains impurities and requires further refining.



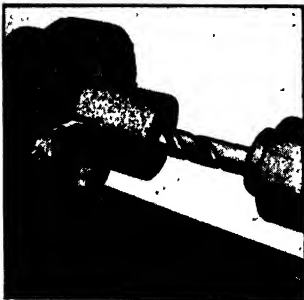
As in blast-furnace operation, iron oxide is reduced. Furnace here contains hydrogen atmosphere.



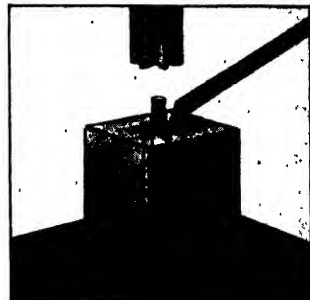
With remelting of the pigs, the iron is refined and cast into cylindrical blanks that weigh fourteen ounces each.



Pure iron powder is milled. After this dustlike particles may be smaller than diameter of human hair.



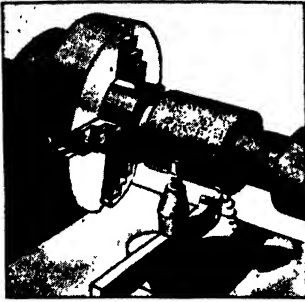
First of several machining operations to make small gear is boring hole through center of the iron cylinder.



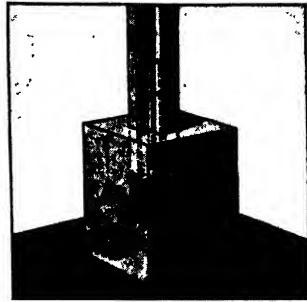
Powder is fed into steel die set in huge hydraulic press. Cost of dies is major problem in powder metallurgy.

*Courtesy of Fortune*

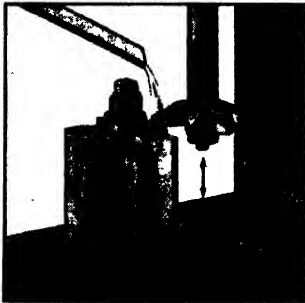
## Schematic View of Production of a Processed Part



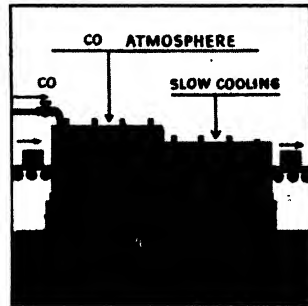
Next production step is to reduce the outside diameter of the cylinder and make it concentric with center hole.



Ram compresses powder into compact solid with one-third original bulk at 50,000 pounds per square inch.



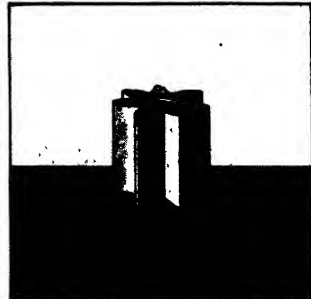
Longest operation is cutting of the gear teeth. Machining never forms gear as perfectly as pressing method.



Fragile pressed pieces are heated into sintering furnace, heated to 1,100° C., emerge stronger than cast iron.



To hold 0.002-inch tolerance, even pressed gear needs slight final trimming. Tooth surfaces are untouched.



Old way: over thirty man-hours of highly skilled labor per 1,000 gears. New way: fifteen semiskilled man-hours.

*Drawings by Lowenstein*

Standard vs. Powder Metallurgy Method

give a product retaining the characteristics of both. Many such combinations cannot possibly be made by fusing the metals, since this would result in an alloy with unique properties; moreover, the metals might be so dissimilar in melting point or other characteristics that they could not be fused at all. A combination of tungsten and copper, tungsten and silver, or zinc, nickel, silver or copper with chromium or ferro alloys are all possible with the powder metallurgy technique.

### **Contact Points**

Contact points are usually combinations of tungsten and copper or tungsten and silver. Here the high resistance of the tungsten to arc temperatures is combined with the high conductivity of the copper or silver to form a product of low resistance which will not pit or burn out rapidly under the temperature of the arcing contact points. The value of these products to industry is suggested when we consider that a contact point on an ordinary telegraph relay operates some six million times a day; such points burn out in a very short period. The new points made with powder metallurgy technique operate for more than two years. The life of a tungsten-copper contact in an oil circuit-breaker is 150,000 operations. An ordinary copper contact fails after 25,000.<sup>1</sup>

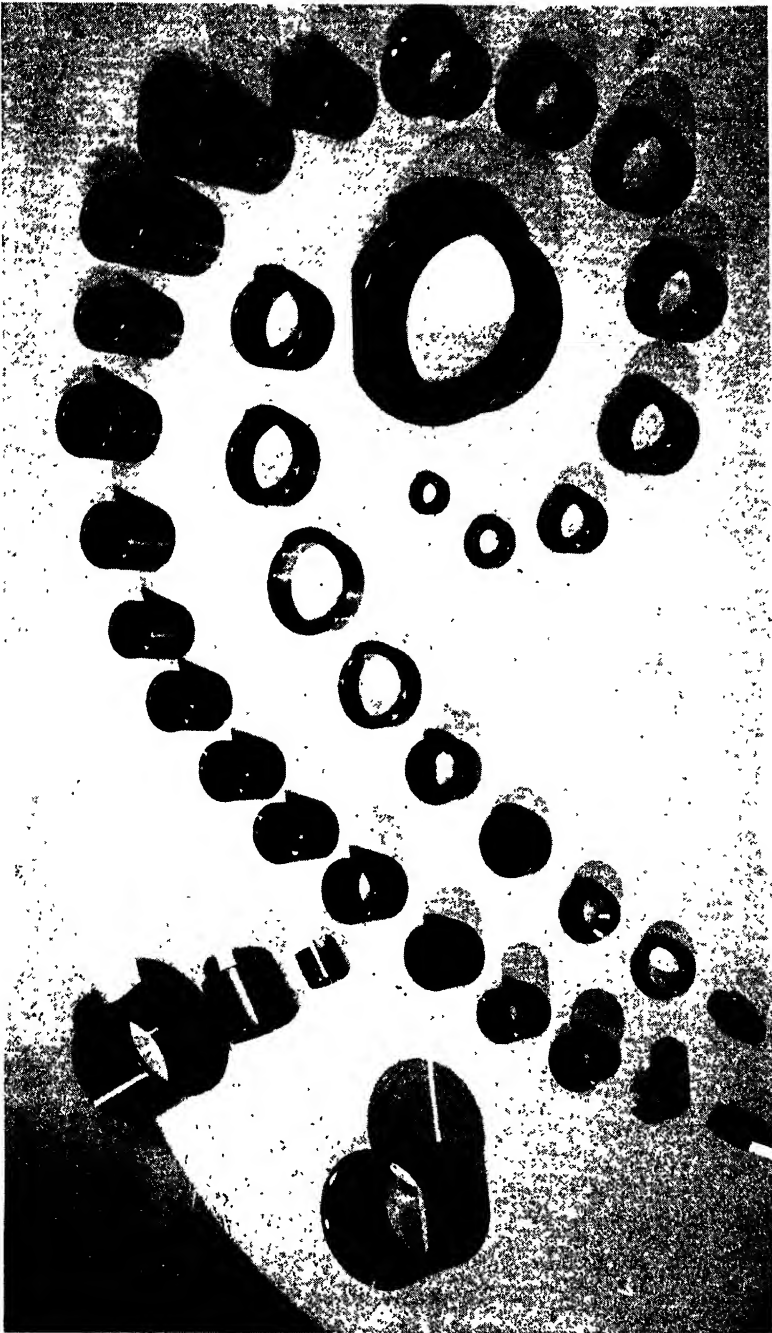
### **Laminated Parts**

Another significant development is the combination of laminated parts. A powder of one composition may be loaded in a die already partly filled with the powder of another metal and the two layers compressed so as to produce a single unit with characteristics quite different in the two sections. The bond is intimate and as strong as the internal structure of any part of the product. Or a powder mixture may be welded during the sintering process or riveted after formation to a metal part formed in the conventional way. This basic method has been utilized to produce clutches and brakes.

### **Clutch Faces**

Although larger parts are being made now for war purposes, some time ago such parts made for clutch bearings had already exceeded units 18.5 in. in diameter, and very much larger units are built up

<sup>1</sup> G. Windred, "Switchgear Contacts," *Engineer*, 558, (1940).



*Courtesy Chrysler Corp., Amplex Div., Detroit, Mich.*

**Super-Oilite, Oil Cushion, Self-Lubricating Bearings and Machine Parts**



in segments. The powdered metal surface may vary from 0.010 to 0.25 inch in thickness. The brake bands on practically all airplanes are made by the powder metallurgy technique, as are also clutches, including those which control the revolving turrets on armored tanks. An airplane landing at one hundred miles per hour creates a temperature on the surface of the brake of 1200° F. in eight seconds. Powder metallurgy products alone can endure under such intense treatment.


### **Electronic Parts**

There have been a great many developments in the production of induction and condenser cores used for radio receiving equipment, and the Alnico magnet which actuates the loud speaker of the late model radios is made by a powder metallurgy technique. The induction cores made by this process are very much smaller and more efficient than those made in the conventional way.

The equipment on war planes for radio transmission employs many parts made by this process. It is said that if these were made from solid metals, no efficient radio could be designed that would not occupy many times the space of these powder metallurgy equipped radios and that they would weigh twelve times as much. The advantages of smaller units, which make space and weight for a useful load and increase carrying capacity, are self-evident. The use of such parts for "walkie-talkie" equipment are compulsory. The Signal Corps now equips soldiers who go directly into front-line action with radio apparatus which can be carried on their backs, providing a means by which they can receive orders from and transmit information to the rear at a distance of fifty miles. The weight of such equipment is nearly sixty pounds. It is obvious that no man could carry into action equipment weighing twelve times as much as this.

### **Another Application of Porous Parts**

The fact that metals can be combined to give products of varying porosity, which can be controlled from 0.25 to 40 per cent voids with a close degree of accuracy, has been utilized to produce filter plates for the separation of solids from liquids and gases, or for the intimate diffusion of gases.



### **Metal and Non-Metal**

Powdered metals may be combined with non-metallic products to produce units having special characteristics. Thus we have a resistance element made of copper powder mixed with porcelain powder which is said to have a resistance 40 per cent greater than Nichrome.

Grinding wheels may be made by combining diamond dust or carbide dust with softer metals or with plastics either at the periphery of the wheel or throughout the structure. Diamond-impregnated drill heads are used for oil-well drills and for other equipment.

The above description of the products made by the powder metallurgy technique and in commercial use is by no means exhaustive, and it would probably be impossible to list all the applications in present use. Moraine Products, a division of General Motors, has a 76-page catalog of standard parts in stock which are all made in this way.

### **Metal Spray Gun**

The Schoop and Schori guns spray a fine stream of powdered metal upon a freshly cleaned surface to build up a new surface. The Schoop gun feeds a wire through a hot flame and the melted metal meets a strong air stream which atomizes it and impinges it upon the prepared surface. The Schori gun uses metal powders instead of wire. The size of the particle can be closely controlled, and modification of the flame produces a powder having a controlled oxide surface. The method has been used to set diamonds in a diamond cutter, and is widely used to build up faulty parts and damaged surfaces. The gun may also be used to make powders for the usual powder metallurgy operations, but it does not appear to be an economical method for the production of powder at present. It is interesting to note that this is an excellent example of "cold welding." The impinging powder is quite cool; the operator tests it by placing his hand in the stream. It coheres perfectly, if the surface has been properly prepared.

### **Recent Catalysts**

A number of molded products which may properly be called powder metallurgy products are now prepared as catalysts, since they are formed from metal powders, specially prepared with high

catalytic properties, though in some cases they are not sintered. No great tensile strength is required of these, but they must have sufficient mechanical strength to resist light impact and side stresses; for example, a copper compact molded at forty thousand pounds per square inch has all the mechanical strength required. Such a compact about one-quarter inch deep with a diameter of one inch presents an enormous surface area for the reaction, infinitely greater than could be obtained by using a solid disc, and is much more convenient to use and replace than loose powder.

### Newer Electronic Parts

No catalog of powder metallurgy products is complete without further mention of developments in induction coils. The eddy losses which reach considerable magnitude in the high-frequency cycles used in communication prohibit the use of solid magnetic cores; but it is known that if instead of a solid magnet, the core is composed of discrete particles, each well insulated from the other, these eddy currents are reduced to a minimum. Radio cores made of a high-grade, reduced iron powder or, better still, an iron reduced from carbonyl, have already been mentioned. In some applications, Permaloy, an alloy of iron with nickel, is employed. Schumacher<sup>2</sup> recently reported a modification of Permaloy by the addition of molybdenum to the original alloy, which further suppresses eddy current loss, and makes it possible to use very much smaller cores to do the work of the large cores made by the older Permaloy.

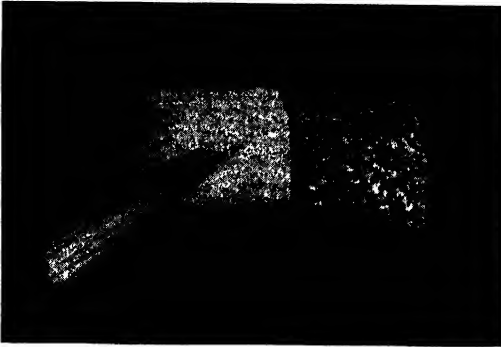
### The Alnico Magnet

Another well known magnetic material is Alnico. Alnico magnets present no difficulty to casting operations, if the final product is to be of large, regular size. But the product is mechanically weak, because of large grain, and is difficult to machine into small parts or those of intricate shape. These small magnets are now made by the powder metallurgy technique. Howe<sup>3</sup> claims many advantages for this method of production, among which are greater strength; freedom from poles, due to lack of homogeneity, and from physical imperfection common to casting; and the fact that products can be made with small dimensional deviation, which can be easily cor-

<sup>2</sup> Earle E. Schumacher, "Powder Metallurgy," p. 166, Edited by John Wulff, 1942.

<sup>3</sup> G. H. Howe, "Development of Sintered Alnico," p. 183, 1940 Powder Metallurgy Conference at M.I.T.

rected by grinding without the danger of breaking, a danger always present with cast Alnico.



Alnico pieces broken to show smooth grain of sintered product in contrast with cast product.

*Courtesy General Electric Co.  
Schenectady, N. Y.*

## Welding Rods

Techniques in welding have changed considerably in the last few years, and new developments continue to occupy much space in the journals. The reason for this is that the welding electrode is no longer the simple piece of apparatus of other times. Powder metallurgy has made it possible to produce a number of combinations of metals and metals with non-metals which were previously impossible. Fluxes are incorporated, or are bound as a surface coating to the principal material; and other materials which serve to control atmosphere, sputtering, current density, and other important factors, have become an integral part of the welding electrode. The extrusion press rather than the die press dominates the field in forming these products from powdered metals, with or without the addition of non-metallic powders.

## General Uses

Before leaving this subject, it might be well to classify the general applications of powder metallurgy.

(1) Powder metallurgy may be used to produce solid ingots from highly refractory metals which cannot be commercially fused in available furnace equipment.

(2) It may be used to combine in a single unit metals of greatly disparate melting points or highly distinctive characteristics with the object of producing a unit with the characteristics of the several combined metals unmodified.



*Courtesy Hydraulic Press Mfg. Co., Mt. Giload, O.*

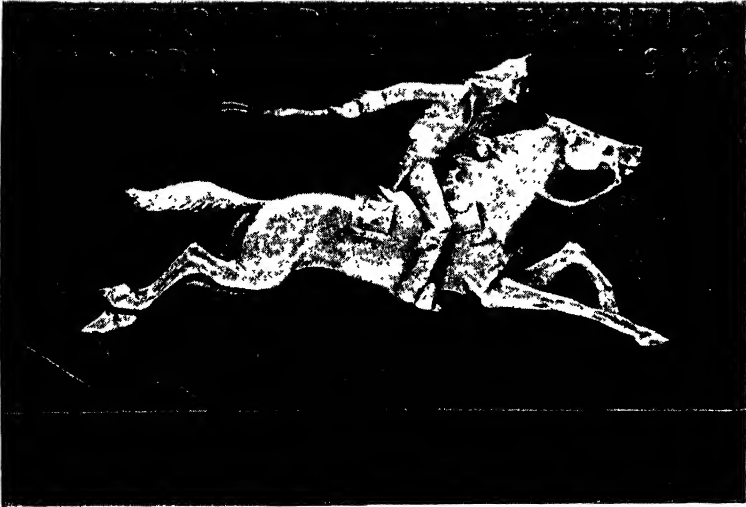
**Extruding Welding Rods from a Hydraulic Press**

(3) It may be used to make precise-dimension products which are so hard or brittle that conventional shaping operations are difficult and sometimes impossible.

(4) It may be used for the production of units having characteristics unobtainable by conventional methods, such as heavy metal, porous metals, and electronic products.

(5) It may be used for laminated parts with a more intimate binding than would be possible by conventional methods of attaching one metal to another.

(6) It may supplant machined precision units, when cheaper or more accurate than machining, or when there is a shortage of lathes or skilled machinists.



*Courtesy Hardy Metallurgical Co., New York, N. Y.*

**A Gold and Silver Medal Compressed from Metal Powder**

## Chapter 3

### Production of Metal Powders

Metal powders have been made in a great number of ways, but these fall into three general classes—mechanical, physical and chemical.

#### MECHANICAL METHODS

Under the heading of mechanical methods is machining. The ordinary chips which come from the machining of any metal or alloy and may be made very fine may be either used directly or ground with a ball mill to produce still finer particles.

#### Milling

Hard metals may be milled satisfactorily by stamping or by rod- or ball-milling. The products may be made over a large range of sizes. The milling of soft metals by the stamping process is extremely flexible; it usually produces particles that are shaped like a flat disc rather than granular; this is the method commonly employed in making aluminum powder for pigments. It has also been used for the production of a flake-like copper powder which is utilized primarily for carbon collector-brushes. The shape of the balled material is usually characteristic and may be identified by microscopic examination.

Stamped powders present a flat disc-like shape, as would be expected.

In the production of magnesium powder, great care must be taken during machining since there is considerable explosion hazard. The ball-milling which subsequently takes place is carried out in a neutral atmosphere. Ball mills, stamping, jaw crushers, rolls and discs are employed on such brittle metals as antimony, silicon and bismuth. It is usual to shot the hard metals, such as chromium, manganese, molybdenum and tellurium before they are balled. It is possible to ball-mill successfully some of the softer metals by

changing the temperature at which the ball mill operates. Thus tin, for example, becomes brittle just below the melting point, and lead at low temperatures.

Brittle alloys may be made by introducing small proportions of foreign materials into soft metals. Thus the addition of iron to aluminum produces an alloy that may be readily milled.

The Eddy mill has been employed to make powders which are extremely fine and very pure. This involves the principle of impacting one powder particle upon another, so that all the attrition is derived from the powder itself, and not from other materials, as is the case when ball or rod mills or disc grinding is used to reduce the size. Under a microscope, Eddy mill powders exhibit various dish-like shapes showing where a pellet has partly flattened out another pellet and indented it slightly.

Aluminum, when either stamped or balled, is often made with a lubricant; and such powders, while satisfactory for pigment manufacture, usually require special treatment when used to form a product from a die.

### **Shotting**

Shotting of the low-melting-point metals produces a very good powder. Aluminum, lead and zinc powders are frequently made by this method, and although they run high in oxide content, they are employed for certain processes. Thus chemical zinc is usually made in this manner, as is small gunshot.

The process of shotting involves melting the metal and allowing it to fall, usually through a screen, the length of a shot tower. If oxidation must be prevented, the tower may be filled with an inert or reducing gas. The metal may be collected under water, or the tower may be so high that the tiny pellets have cooled enough by the time they reach the bottom so that they will not sinter. Zn, Al, Pb, and Ni powders have been made by this process. Powders made by shotting approach the teardrop in shape and vary from spherical to pear-shaped.

### **Granulation**

The process of granulation employed for the production of coarse powders from aluminum, cadmium, zinc and brass results in a rather high oxide powder, which is usually coarse. The aluminum pro-



duced by this method is frequently used in the manufacture of thermit; other metals are often employed for abrasives. Brass powder made by this process is used in some brazing operations.

The process of granulation begins with melting the metal and stirring it as it cools. The surface is somewhat cooler than the body of the melt, and it crystallizes as it is stirred. It is these crystals which become the powder.

Products of granulation run extremely high in oxide. They are not easily distinguished from other powders under a microscope, but the presence of two or three per cent of oxide is a good indication that they have been made by granulating.

### **Atomizing**

Atomizing is employed for the production of zinc, aluminum, cadmium, lead and a number of other low-melting-point metals. It gives a powder over a large range of sizes, and the size can be closely controlled. Surprisingly enough, when we consider the method of production, the oxide content is not high, and the powders are used for making many molded and sintered products. This process consists of melting the metal. The molten metal falls in front of a stream of air or steam which blows it through a tunnel in fine particles. The product can be closely controlled by varying temperature, orifice, fall and pressure of the atomizing stream. Atomized powders are always very irregular in shape. They have a good density and are fairly uniform in size.

### **Condensation**

Metals which may be vaporized at low temperature are condensed on glass or other surfaces to which they do not adhere too closely. The first metal powder made in this way was zinc. By means of an electric arc higher-melting-point metals are vaporized and condensed to form very pure powders. There are a number of patents on condensation processes including several which cover producing magnesium powder by this method. Another means of producing vapor is to drop the metal on a very hot plate; fine droplets of metal are thus formed, which vaporize rapidly just like water dropped on a hot plate. The vapor is condensed to produce a very fine powder of high purity.

## PHYSICAL METHODS

**Electrodeposition**

Turning now to what we may arbitrarily call the physical methods of powder production, the most significant is the electrodeposition of metal from solution. Tantalum, tin, silver, copper, zinc, iron, cadmium and antimony powders are all commercially made by this process. They are invariably dendritic and their chief weakness lies in their low specific gravity. Unless subsequently heat-treated, they are not as easy to mold as products made by chemical processes.

The electrodeposition method is precisely the same as the plating processes that are so well known, except that in the latter every effort is made to form a continuous and tenacious sheet of metal on the electrode, whereas in the case of powders the conditions which lead to successful electroplating are exactly reversed. The object is to produce a fine, loose powder, not a continuous, tenacious sheet.

The electrolytic powders are easily identified, as they exhibit a distinctly dendritic structure. They are made by causing a brittle or a spongy deposit to form on the anode, and this deposit is either rubbed or ground to a powder. The proper characteristic is obtained in the deposit either by increasing the current density, by a high acidity, or by the addition of some organic substances to the electrolyte; glucose is most commonly employed. The low specific gravity of these powders is considerably improved by a subsequent heat treatment and grinding. If an electrolytic powder has received such treatment, the essentially dendritic structure is sometimes difficult to detect by microscopic examination.

**Carbonyls**

The carbonyl process is a bridge between purely physical and chemical methods of producing powder; in a sense, this is true also of the electrolytic process. A number of metals, such as zinc, magnesium, iron and nickel, form organic compounds which under proper temperature and pressure decompose to yield a gas and a metal. The best known of these are the iron and nickel carbonyls, which give some of the finest and purest of all commercial metal powders. They are used largely for molded parts and in great quantity for electronic products. The carbon monoxide combines with these

metals at a sufficiently elevated temperature and pressure to produce the carbonyl liquid. At a reduced pressure and at an elevated temperature these carbonyls decompose and precipitate the metal, releasing the carbon monoxide, which recycles and is again employed to produce more carbonyl liquid. The products of the carbonyl process are perhaps the easiest to identify under a microscope. They are absolutely spherical and rarely reach 10 microns in diameter. A cross-section shows them to be distinctly lamellae spheres, and the layers are characteristic.

### CHEMICAL PROCESSES

#### Reduction

The chemical methods produce the greatest quantity of metal powder employed in the powder metallurgy industry. Tungsten, molybdenum, iron, copper, nickel and cobalt powders are all made by reducing their oxides with either hydrogen or one of the common reducing gases. It is an extremely flexible process and the product may be closely controlled by the temperature, pressure and gas employed. It is, however, nearly always slightly porous; and if any oxide remains, it is more significant than the oxide which is so frequently present in mechanically produced powders. The latter is primarily a surface oxide which does not materially reduce cohesion when the powder is used in a die. The oxide which may remain in a powder obtained by reduction is primarily that part of the original oxide which has not been reduced and therefore lies in the center of the particle. Obviously, mechanical handling or the frictional attrition which may arise in the die during molding cannot reach this oxide and its action is more objectionable.

The oxides reduced under hydrogen are somewhat fluffy in appearance, but it is difficult to distinguish them from those made by chemical precipitation.

#### Precipitation

If aluminum is added to copper, iron, or nickel sulfate, the aluminum replaces the other metal, which precipitates as a fine powder. Iron scrap is conventionally used to precipitate copper from copper solutions. The copper precipitates as a pure powder. Zinc, added to solutions of tin salts, replaces the tin; the production of tin powder by this method is common practice.

### Chemical Reactions

Some metals are made into powders from their salts by chemical reaction. Tin, silver, copper, selenium and tellurium powders are frequently made this way. The product is extremely fine and very pure. It is commonly used for coating paper. Tellurium is sometimes mixed with lead, as a small proportion of tellurium adds considerable hardness to this soft metal. All these products may be employed in the molding process, but they are usually expensive.

### Alloy Disintegration

A new process, which at the beginning of the war was in its pilot stage, is the chemical treatment of alloys. This is based on the fact that stainless steel consists of a series of alloy crystals bound together by a network of material more chemically active than the stainless steel crystals. The process involves washing out this network with chemicals to leave the crystals as separate entities.

During 1941 alone, nearly a dozen new powder metallurgy products came on the market. A number of these were high-purity iron powders and some were steel powders with a controlled carbon content. There were also a new aluminum alloy equivalent to cast aluminum, a low-porosity bronze with excellent wearing qualities, and a new bronze porous bearing material capable of sustaining much greater loads than had previously been possible for powder metallurgy bearings.

There are practically no metals that cannot be made in powder form suitable for powder metallurgy, and certainly most of the metals employed commercially in conventional products are now obtainable as powders in commercial quantities.

### RECENT DEVELOPMENTS

Powders whose outer surface has properties different from those of the interior of the particle are of special interest to powder metallurgists. A highly plastic surface adds to the ease with which powders may be molded, but a powder which is soft throughout may be objectionable for other reasons. An iron powder which becomes converted to a carbon steel is an example of such a powder. A high-carbon powder is subjected to a treatment which reduces the carbon content of the surface, thus making it more plastic than high-carbon iron; but the reducing process does not penetrate to the cen-

ter, which remains rather brittle. During the sintering process the carbon diffuses throughout the entire compact, and the final product is one of uniform steel of controlled carbon content.

Carney, in his United States Patent 2,273,832, discloses a method for making powders of double characteristics. He mixes relatively large particles of a high-melting-point powder with relatively small particle-size powder of low melting point. This mix is sintered and ground. He claims that the powder so formed has a hard center with a malleable surface.

Spherical particles are thought by some to be more valuable than irregularly shaped particles. Opinion is sharply divided, but Gallup has received United States Patent 2,269,528 for making powders with spherical particles. He feeds a thin strip of metal to a heat source which melts the metal into droplets; these fall through a tower onto an inclined plate, from which they bounce through an outlet in yet smaller spheres.

### **Iron Powders**

The production of iron powders is receiving a great deal of attention at the present time. It is evident that the trend of powder metallurgy is such that there is a great demand for a satisfactory iron powder at lower prices than now prevail. The demand has sharply increased since the war, and the need of a domestic source has been accentuated by the reduction in the supply of Swedish sponge iron; however, it is probable that at no time did we import enough Swedish sponge to supply present requirements, or even a high percentage of the needs which are growing and will further increase as manufacturers become aware of the advantages of powder metallurgy.

The importance of this product has not gone unnoticed. As early as 1927 the Bureau of Mines investigated and ran pilot plant experiments in an attempt to make a good iron powder by a number of processes. Some of their work revealed processes that appeared to have commercial value. A number of commercial projects were started and failed, not for lack of funds (they were backed by such companies as Ford and United States Steel) but primarily because they encountered mechanical difficulties which could not be overcome. In the last few years, some commercial progress has been made. One company is producing large quantities of powdered iron

of a quality which is comparable with Swedish sponge, but which sells about seventy per cent higher. For many years some iron of a superior grade has been produced but at a price some six times as great as the imported. New companies have begun production in the last two years; they produce a series of powders which sell for from ten cents to over a dollar a pound, and some carbonyl iron is being made in the United States which sells for from one to three dollars a pound. Previously these powders were imported from Germany.

The Government has become aware of the significance of iron powders and has recently authorized the expenditure of \$600,000 for the solution of the problem of producing a cheap sponge iron.

R. A. Boyer<sup>4</sup> has recently described an electrolytic process for the production of iron powder from low-grade ore. The ore is leached, preferably with hydrochloric acid, and the leach subjected to electrolysis. After the iron has been removed the acid recycles to take up more iron. Since it requires one kilowatt hour to produce a pound of iron, conditions would have to be highly favorable if this method is to produce iron powder cheaply.

It would appear then that we shall have to rely upon a chemical process for the production of a cheap iron powder. This will probably take the form of a process to recover iron from such waste products as pickle liquor, or the perfection of some of the reduction methods which have failed in the past. New knowledge is now available which may solve the old problems arising in reduction methods. We have learned something about the significance of cold-welding, and more about what causes coherence of metals. It is this coherence—the angel of powder metallurgy technique in the molding process—which is the chief devil in the reduction process. Although the method calls for relatively low temperature, no way has yet been found to prevent the freshly formed metallic iron from adhering to the walls of the reducing furnace. The companies which failed in earlier times found that, as the process went on, a ring of metallic iron built up within the chamber; less and less iron passed this ring until finally the entire process had to be shut down to clean the furnace. Nor was there any simple way to accomplish this, since the scale clung to the walls with a tremendous force.

Since we now know the significance of clean surfaces in the phe-

<sup>4</sup> R. A. Boyer, *Steel*, **88**, 112 (April 6, 1942).

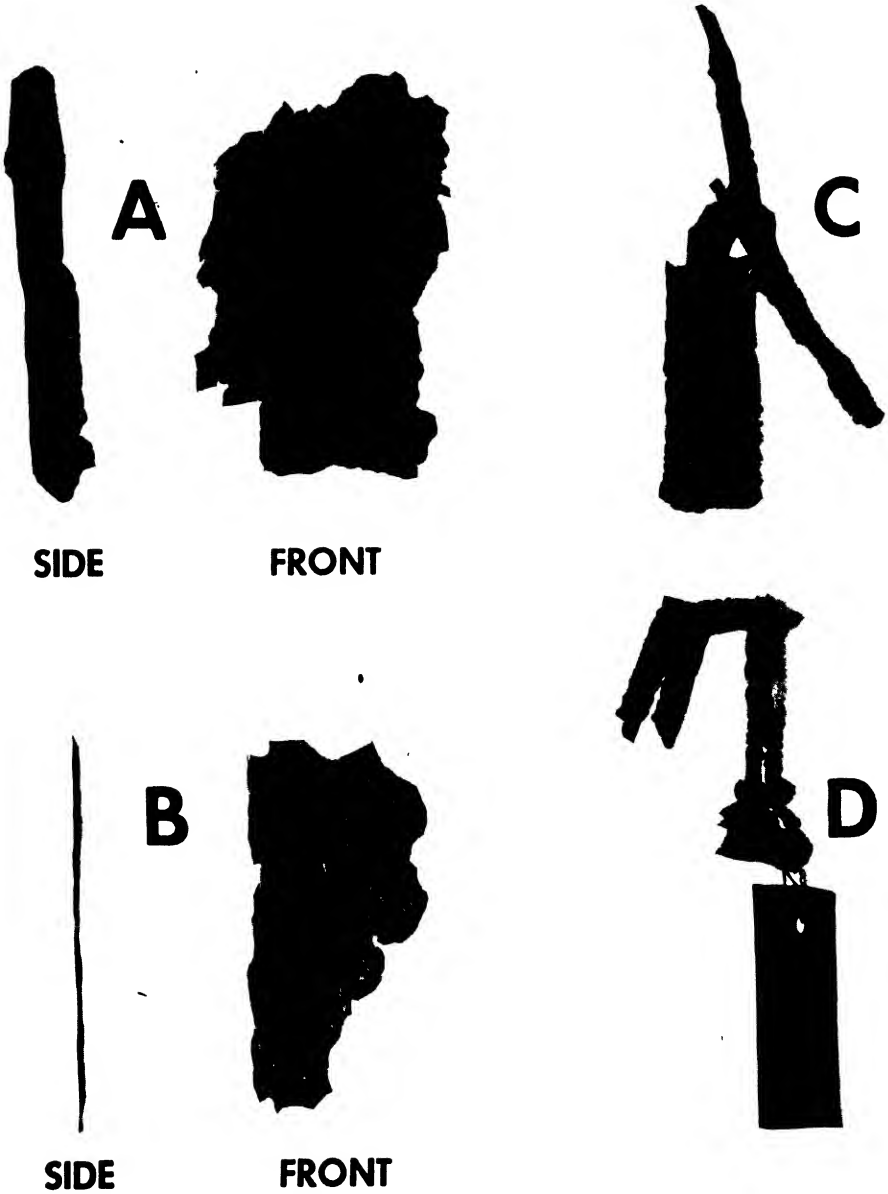
nomenon of cold-welding, it is possible that a new approach to the problem—some means of introducing an interfering phase—may make one of the previously unsuccessful reducing processes work in the immediate future.

The writer was called upon to solve just this problem in the production of iron from carbonyl. The process had been supposedly solved successfully in pilot plant tests, and even in short run tests in commercial operation; but after a longer run, yield dropped to zero, and all the iron made built up a wall within the reaction chamber, throwing the entire process out of heat balance, and making continued operation out of the question. The walls of the towers could be cleaned only with difficulty. The introduction of an interfering phase between the freshly precipitated iron and the reaction walls reduced coherence to a minimum.

On page 35 there is a photograph A, of the scale formed after a four-hour run before the walls had been properly treated, and B, the scale formed in *twenty* hours after treatment. Picture C is an untreated test specimen hung in the tower for four hours, and D shows a similar specimen which was treated and hung next to the control during this same period. Note how the iron was built up on the metal wire upon which both specimens hung, and observe that where the loop holding the treated specimen is attached no iron has built up, nor is any apparent on the treated specimen.

The solution of the carbonyl problem would not solve the problem of the oxide reduction process, since conditions are radically different, but an approach to this problem might profitably be made by application of the same principles.

A small source of iron is found in treated scrap. In some processes, such as hot-pressing and hot-forging, powder size and quality seem to have less bearing on the physical properties and size changes of the final product, than when cold-pressing followed by sintering is employed. In any case several companies have found it possible to "shred," that is further disintegrate, the metal chips and machine turnings from lathes and other mechanical operations, heat-treat them, and use them to mold finished products which apparently have all the physical strength necessary to perform the functions for which they are designed. Both General Motors and Moraine Products have found such powders satisfactory. Chevrolet uses shredded chips of S. A. E. X1112 steel coming from the screw machine



Carbonyl Iron Scale Found in Decomposition Tower

- A. After four hours, before wall treatment.
- B. After *twenty* hours, after wall treatment.
- C. Untreated Test Specimen, hung in tower four hours.
- D. Treated Test Specimen, hung with C four hours.



without other treatment than washing to remove lubricant.<sup>5</sup> Mo-raine ball-mills a low-carbon scrap and decarbonizes it in a controlled atmosphere to obtain a practically pure iron powder.<sup>6</sup> These companies do not make iron powders commercially, but only for their own use.

### Hydrides

No list of powdered metals would be complete without mention of the hydrides. Besides sodium, potassium and calcium hydride there are available zirconium, titanium and titanium-copper alloy hydrides in a powder form. These hydrides are still very expensive but their addition in small quantities to other powders has some interesting effects. "Nascent" hydrogen begins to be released at the lowest sintering temperature and has a remarkable effect upon the boundary phases of the finished product since it combines with and removes the last trace of oxide.

<sup>5</sup> Herbert Chase, *Automotive and Aviation Inds.*, **85**, 35 (1941).

<sup>6</sup> *Iron Age*, **149**, 49 (1942).

## Chapter 4

### Powder Specifications

As commercial developments grow, new methods of specifying the type of powder required by the producer will no doubt be originated. Unfortunately, most of the users of metal powders have arrived at their specifications by purely empirical means. A few of the general ideas are fairly well agreed upon, but the basic knowledge required to outline the specific qualities required to yield a product of known physical characteristics is not fully understood.

#### Chemical Properties

In general, the chemical properties of metal powder are less important than the physical. Oxygen is the impurity which has received the greatest attention from those working with all types of powder, but experience has shown that the importance of the oxide content is greatly modified by the form of the oxide impurity. If oxide occurs on the surface of the particle it does not interfere very markedly with the molding operation or subsequent strength of the product. On the other hand, occluded oxide will generally result in a product of low tensile and impact strength. It appears that surface oxide in the form of a thin film is displaced during processing, and the place from which it has been removed presents a fresh metallic surface to promote adhesion. This oxide may subsequently be reduced during the sintering operation if it is carried out in a reducing atmosphere. If the sintering takes place in a neutral atmosphere the oxide will of course remain unchanged, but apparently it does not materially affect the physical properties demanded of the product.

If the oxide is occluded, however, there is nothing in the production cycle which has any tendency to remove it, and a weak spot remains within every particle in which oxide is present. The subsequent treatment in a reducing atmosphere in a sintering furnace is not sufficient to reduce the occluded oxide. Most such oxides are present in metal powders which have been made by reducing an

oxide powder. The reducing action of the process proceeds from the outside of the particle toward the center, but not far enough to remove the last traces of oxygen. The treatment received during this reduction process is considerably more severe than that to which the formed product is subjected during sintering, and if this severe treatment is insufficient to reduce the oxide, it is obvious that the less severe conditions during sintering cannot do so.

The final product will be weaker than when made from a purer powder, but of course in some cases it is sufficiently strong to meet specifications. Sponge iron with an oxide content of approximately 4 per cent has been used successfully for many products, but even 1 per cent of oxide is too much for satisfactory iron products for the radio industry.

Some companies are developing processes for the manufacture of parts from iron with as high an oxide content as 7 or 8 per cent, but the general practice is to specify either a 99+ or a 96+ iron. The oxide content of other metals is generally less than 2 per cent and the purchaser frequently makes no specification on this point. Every effort is made to keep the oxide as low as possible. Much of the oxidation takes place during shipment and handling, after the powders have been brought to a high degree of purity. Most manufacturers take this into consideration. High-purity powders are frequently shipped in steel drums often evacuated or containing a neutral atmosphere.

The presence of carbon and silica may be serious in iron powders; but again the form in which the carbon is present is more important than its quantity. If the carbon is combined with the iron, the hardness of the iron is increased and its malleability reduced. This in turn lowers the cohesive strength of the compact. It makes it necessary to use higher pressures to obtain products, the green strength of which would be achieved under lower pressure with an iron purer in respect to carbon. If the carbon is residual and free carbon, it does no harm to most products, and may aid the production cycle by slightly lubricating the dies.

Silica is to be avoided because of its abrasive action on the dies and because it acts as an interfering phase and reduces metallic cohesion. For this reason manufacturers frequently specify a low silica content.

Sulfur should be low, since during sintering it may create an acid

condition obviously undesirable in the product, and furthermore it shortens the life of the equipment. The sulfur may combine with any oxide or the hydrogen of a hydrocarbon to produce sulfur dioxide, sulfurous or sulfuric acid, or hydrogen sulfide. Acid is of course undesirable because it is chemically very active. Physically, during sintering, the expansion of the gas may affect the porosity and the size and shape of the finished product.

Minor percentages of foreign metals, for instance manganese, in an iron powder, do not appear to require any special safeguard and, incorporated in an iron product, do not seem to affect its physical properties. Obviously, if the part is to be used in the radio industry, foreign metals would introduce new electronic or electrostatic properties and complicate methods of production.

### Physical Properties

Turning now to the physical specifications for metals, the prime requisite is that the particle size should be constant. Manufacturers usually specify this in rather broad terms for example, 100 per cent through 100-mesh, or 50 per cent through 325, but a closer attention to screen analysis and micro-count would obviate a number of irregularities which producers frequently encounter. Many of the characteristics which the producer thinks he is controlling by these broad specifications, cannot be controlled by them alone. The distribution curve of the sizes is of great importance in controlling the percentage of voids and the change in size—shrinkage or growth—from green to sintered compact. The apparent specific gravity of the powder is also largely affected by the distribution of sizes, and the flow is materially affected. The producer requires a powder which will have constant shrinkage or growth during processing so that the product coming from his mold will either shrink or grow to meet the specifications, often within a tolerance of less than one thousandth of an inch. Any change in the size of the powder which brings about even small variations in size during sintering will obviously produce parts which will not meet the specifications. The apparent specific gravity is important because commercial operations employ a volumetric fill for each mold. Therefore, if the apparent specific gravity varies, more or less powder will be fed into the mold, depending upon whether the gravity has been increased or decreased, and when pressure is applied, provided it is applied

to a constant pressure, the green compact will be shorter or longer than desired. If pressure is to a definite volume, the porosity and density will be affected.

Flow is important for the same reason. The feed is by volume. A powder which flows freely and rapidly can be fed to a cavity with great rapidity and with confidence that there will be little bridging and that all parts of the die will be filled with an even quantity of powder. If the flow is poor, it will be necessary to feed the cavity slowly in order to get sufficient powder into the die. An even fill becomes more difficult. In general, a powder with poor flow has a tendency to bridge, leaving spaces within the apparently full die.

It is for these reasons that attention to particle size is of great importance in purchasing powder.

The effect of distribution of size is made apparent if we consider a study of packing spheres of equal size. Regardless of whether these spheres are less than a micron or very large, if they are systematically packed one on top of another, there will be 47 per cent of voids.<sup>7</sup> If one sphere is packed on two spheres and this continued until the entire space is filled, there will be 41 per cent of voids. If they are packed one on three spheres, there will be 26 per cent of voids, and if one on four, this remains unchanged. A moment's study will show that any other form of packing is impossible and can be but one of those already considered or a combination of these. Therefore, if the particles are all of equal size, it is impossible by any systematic form of packing to reduce the voids below 26 per cent. Any chance packing of particles of equal size certainly will result in greater porosity than 26 per cent. To create, then, a more solid product, it is necessary to introduce a variation in the size of the particle so that the voids created by the packing of particles of one size can be filled by particles of a smaller size.

Theoretically, it is possible to reduce porosity to nearly zero by selecting the proper distribution of size. Practically, it is of course impossible to pack particles of very small dimensions systematically and we are left with haphazard arrangements.

A great many workers in this field have stressed shape of particle as a determining factor in flow, but the writer feels that here again distribution plays a predominant part in this phenomenon. In support of this he cites the fact that it is possible to reduce materi-

<sup>7</sup> R. Meldau and E. Stach, *J. Inst. Fuel*, 7, 336-54 (1934).

ally the excellent flow characteristics of a given powder by screening out of it a percentage of the fine or a percentage of coarse particles, thus changing the distribution of sizes. It is also possible to improve the poor flow characteristics of a powder by varying the size distribution of its particles. Since the same powder is employed in each case, the shape of the particle remains constant and the determining factor is entirely the size distribution.

It should be noted that if we limit the distribution to a few definite sizes, no matter how these may be varied proportionately from one to another, porosity will remain nearly constant. Fraser<sup>8</sup> has done some interesting experiments which illustrate this. He used particles of three different sizes, 1.3, 2.3, and 8.1 microns. Using 10, 10 and 80, the voids were 33.3 per cent; with 30, 40, 30, they were 33.8 per cent; with 40, 30, 30, they were 33.1 per cent, and with 10, 45, 45 they were 33.4 per cent. (Fraser's figures have been reduced to round numbers.) For commercial purposes it may be considered, then, that porosity will remain constant if there are but few sizes involved, regardless of their distribution.

In view of these considerations, we can see that particle size distribution plays a significant part in the manufacture of powder metallurgy products.

### Importance of Small Size

Powders larger than 100-mesh have no particular value in powder metallurgy, since it is concerned with surface phenomena. The finer the powder the greater the amount of surface. A cubic centimeter cube presents but six square centimeters of surface, but if reduced to -320 mesh, it presents a surface equal to 384,000 sq. cm. With such an increase in surface during pressing an enormous increase in friction results, producing an internal heat which promotes plasticity. Plasticity increases the surface-to-surface content, which increases the cohesive forces; and with the increase of surface *per se*, an enormous increase in surface-to-surface content is obtained. Against this can only be raised the objection that the entrained air is also increased, since each particle carries with it an air envelope. The smaller the particles, the more of them are in a given volume, and the more adsorbed air is present. But the gains far exceed the minor losses.

<sup>8</sup> H. J. Fraser, *J. Geol.*, **43**, 910-1010 (1935).

## Chapter 5

### Classification of Particle Size

#### Specifications of Significance to Manufacturers

As the name indicates, the entire industry of powder metallurgy is built around a raw material which may be any one of a number of metals, but is always a powder. Although the trade has far from any clear-cut specifications for powders in general, once consumers have discovered a powder whose empirical tests meet their needs, they set up specifications which must be adhered to rigidly. A change in average diameter or in distribution of sizes has a very marked influence on the physical properties of the finished product. Very often a slight change cannot be compensated for by varying a cycle of operations, though in many cases it allows great flexibility. In general, most powders used in the industry are 100 per cent through 100-mesh, and from 35–50 per cent through 325-mesh; but for different metals, and for different consumers using the same metals, there is considerable variation in the size specification.

Many other factors besides size are of significance. Shape, flow and apparent density are the most important physical ones, and the last two are a function primarily of particle size and its distribution. Here it might be well to say that authorities are in some disagreement about this, and have expressed the opinion that shape governs flow and influences apparent density.

It is the writer's opinion that size distribution is more significant than any other factor in respect to these two qualities. He has taken a fast-flowing powder, which might be designated as an 8-second powder, and by removing a selected size range, either retarded the flow rate or stopped it altogether. It is also possible to make a poor-flowing powder, or one which has no flow whatsoever, flow satisfactorily by adding one or more selected sizes to smooth out the distribution curve.

In general, the writer has found that a powder which comes closest

on analysis to paralleling the curve that statisticians call the Curve of Normal Distribution is the most satisfactory from the standpoint of flow and apparent density.

Since particle size and its distribution are so significant, it becomes necessary to consider with great care the methods employed for determining them. In the first place, 50 per cent of the powder may be smaller than the smallest mesh available with standard sieves. Nevertheless, the distribution of the size of the particles less than 44 microns is just as important as the distribution of the sizes which make up the other 50 per cent. Therefore it immediately becomes evident that we cannot rely upon sieves as a means of classifying powders. It is necessary to consider all the methods which have been suggested from time to time and which are in use in the industry to a greater or less extent.

We have set up experiments in the laboratory which will give the student experience with sieve analyses (see Experiments 1 and 2, pages 129 and 132), with direct-count methods (Experiment 4, page 140), and with methods based on Stokes' law (see Experiment 3, page 135). These by no means exhaust all the methods of measuring particle size but they represent the principal ones. First, it is well to consider the limitations of the several principal methods of size classification.

### Range of Testing Methods

The sieve will measure particles from about 60 to 100,000 microns; elutriation from 5 to 100 microns; sedimentation from 1 to 100 microns. An ordinary microscope will measure particles from 0.3 to 100 microns; the turbidimeter, which is one type of method based on Stokes' law, from 0.2 to 50 microns. The ultra-microscope is satisfactory from .05 to 2 microns, adsorption methods from molecular size to 10 microns, and the electron microscope<sup>9</sup> from 5 millimicrons to 1 micron.

Until much more work is done on these methods, especially in the region of overlapping size, they should not be depended upon for absolute size; but, in its own range, subject to the errors which we will discuss, each will give comparable results, which may be confirmed by any skilled technician and repeated within a reasonable

<sup>9</sup> See "The Electron Microscope," by E. F. Burton and W. H. Kohl, Reinhold Publishing Corp., 1942.



limit of error for powders which do not vary too widely from one another in shape or physical characteristics.

### Basic Error

Particle size is usually expressed in average diameter, average volume, or average surface (occasionally average weight). The diameter is determined and the volume or surface calculated from this figure. The assumption is usually made that the particle is a sphere, so that any diameter measured is equal to any other diameter of the particle. From the diameter a simple calculation will give figures to express particle size in volume, surface, or other terms if desired.

The patent fact is that, with very rare exceptions, metal powders are not spheres; nor are they cubes or any other regular geometric solids. In general they so far depart from such regular figures that the first problem confronting the investigator is to determine what dimension he should measure or which dimension his method has measured. It must be that one, or those dimensions, from which he can calculate the particle size in terms intelligible to those who employ the data.

### THE SIEVE

The sieve is the most common device used for determining particle size. The results of sieve analysis are expressed in terms of mesh. A report will read: "20 per cent minus 200 plus 325 mesh," which means that one-fifth of the sample examined was small enough to pass through a 200-mesh sieve and too large to pass through a 325-mesh sieve. Common practice gives this a further meaning, to wit: Twenty per cent of the sample has a diameter of 53 microns. In general this interpretation can do no harm and often is convenient. But the student should be aware that implied in this convenient statement is one questionable assumption, and one which is generally false. Though all the material was small enough to pass through a 200-mesh sieve with openings of 62 microns, and too large to pass through a 325-mesh sieve with openings of 44 microns, there is no basis for the assumption that the average size of these particles was the arithmetic mean of the two sizes. Indeed the same report would have been made had the particles been uniformly 61 microns or uniformly 45, nearly 28 per cent smaller.

The other implication, which is true only if the particles are true spheres, is that the sieve measures the true diameter. Actually it passes any particle which has any controlling cross-section with an area smaller than the openings in the sieve. Conversely, it retains any material which has one controlling cross-section too large for the opening. This does not mean, however, that the diameter of this area is the mean diameter of the non-existent theoretical sphere suggested by this statement.

It is to be noted that many observers report in terms of mesh sizes too small for sieve analysis. Thus if the analyst, by direct methods or those based on Stokes' law, finds 20 per cent of the powder to have a diameter smaller than 5 microns, he may report 20 per cent, minus 2500 mesh. Enough has been written about the false implications of the other procedure, *i.e.*, reporting sieve analysis in microns, that no further discussion is required to show the error of this. Despite this, there is still some excuse for reporting mesh in terms of microns because we actually can measure the size of large particles in microns. But since there is no available equipment for sieving beyond 400-mesh, to write of 625-mesh or 1250-mesh sizes is to measure concretely and report abstractly. This is basically an unscientific procedure.

The inherent errors in sieve analysis are in part introduced by particle shape. It is obvious that if the particles are in the form of flat discs, any diameter measured will represent only one dimension and not be the diameter of the theoretical sphere which the sieve is designed to classify. Obviously the sieve will be unable to pass any of the material the cross-section of which is larger than the sieve opening. If the material is predominantly disc-like, the diameter of the flat surface stopped by the sieve has little relation to the particle size in terms of a sphere, and the material retained on the sieve has an actual volume far different from that indicated. On the other hand, if the particle is needle-shaped, the short diameter will be the governing factor in the number of particles which pass through the openings in the sieve. Here again, any attempt to estimate the size of the particle based on this diameter will not represent the true particle size.

In commercial powders, no products are entirely flat discs or long needles, although some milled aluminum powder and some types of copper powder which are employed for commutation brushes ap-

proach the former shape. Most powders are extremely irregular and depart considerably from true spheres. However, the sieving method is suitable and comparable when the shape is not predominantly disc-like or needle-like.

Another error which the sieve cannot overcome is introduced by the tendency of some powders to form agglomerates. Such agglomerates break down in time; and at first glance it would seem that all that would be required to offset the errors introduced by agglomerates would be to shake the sieves for a sufficient length of time to assure the distribution of all interlocking particles. However, further thought on this subject will show that extremely long agitation will introduce an error due to the grinding action inherent in any period of shaking.

It is true that soft powders will stand a considerable amount of shaking without deformation. On the other hand, powders cannot be shaken at all without some attrition. Therefore, with every minute of shaking, the size of the particles decreases and theoretically, at any rate, it is possible to start with a powder 50 per cent of which will go through 325-mesh in the first 10 minutes, and shake it so long that by attrition 100 per cent of it will pass. So we see again that the sieve will not give us any absolute particle size results. Powder metallurgists have agreed upon how sieve analyses should be made, but attention to the details outlined will not eliminate these errors, but only standardizes them. In practice, the powder metallurgy industry usually employs dry-sieving and confines the period of agitation on a standard shaking machine to 20 minutes.

### MICROSCOPIC COUNT

If we turn now from the sieve to a consideration of the direct method, microscopic count, the first problem which faces us is the preparation of the slide. There is very little that can be told about how to prepare a slide. Only considerable practice will make it possible for a technician to produce easily counted slides which will check with one another. The methods which appear in the literature suggesting techniques cover an extremely wide range, but there is no reason to believe that long practice with any one of these will produce better slides than long practice with another; and there appears to be no recommended method of preparing a slide in which one can become efficient more quickly than another.

In Experiment 4, it is suggested that the student try to familiarize himself with the preparation of dry slides and with the preparation of a slide using a dispersion medium. Most students have had best results preparing dry slides, and a few have found the use of diacetone alcohol satisfactory. It is important that, however the slide be prepared, it truly represent the powder, and care must be used to prevent classification.

In the dry method, classification will definitely occur if any attempt is made to spread the powder by tapping the slide and dispersing it by vibration; the heavy and larger particles under this action will tend to separate from the lighter, and no field under the microscope will truly represent all the size ranges.

In the wet method, classification will occur if the motion of the liquid, due perhaps to the angle at which the slide is held, is great enough to float the lighter particles away from the heavier. It will also occur if, on drying the slide, the rate of evaporation is so great as to produce bubbles of vapor which tend to separate the fine from the coarse particles. With reasonable precautions and long experience, it is possible that satisfactory slides truly representative of the sample can be prepared, and the next question which faces the analyst is what he is to measure, that is, what dimension most truly represents the diameter of the particle.

#### THE MEASUREMENT OF THE PARTICLE

Particles tend to fall with the longest axis parallel to the slide and the flattest side down. Therefore they present a classified diameter to the observer. The length will appear in proper distribution, but the breadth generally will be the breadth of the flatter side. The thickness will rarely be presented to the observer, but it may be calculated by measuring the adjustment of the microscope necessary to bring first the top of the particle and then the microscope slide into sharp focus. This obviously introduces all the errors inherent in measuring the depth or thickness of a particle by means of variation in focus.

If, however, we assume that all three dimensions can be determined, then the diameter equals the cube root of the length times the breadth times the thickness. The three dimensions must be determined for each particle and a count of less than 5,000 in most cases will not be sufficient to produce verifiable results. This is a tedious procedure multiplied by three.

Some students of the subject suggest that the diameter may be assumed to be the length times the breadth divided by 2. This obviously is not a true diameter, but considering the number of errors which may arise in attempting to measure breadth it probably is not far different from the result obtained when all three dimensions are measured.

There is a third way of measuring diameter which has the advantage of cutting the observer's work to a single measurement. If a filar eyepiece micrometer is employed, it is fair to assume that particles will fall on each line in such a way that the line will be crossed by a statistically correct distribution of diameters. A few particles will fall lengthwise on the line and an equal number will be crossed through their breadth. There will be as many particles varying from one of these directions as from the other, and it is equally true that for each variation from the exactly parallel position there will be variations from the exactly perpendicular position. Therefore, a measure of the diameter at the point that the filar line intercepts the particle may be said to represent the average diameter of the particles under observation. But here again thickness is not fairly represented.

A fourth way of measuring particles is purely arbitrary, but extends the determination made by the sieve to sub-sieve sizes. In most powder classification work, the examiner seeks a continuous distribution curve from a size too large to be examined under the microscope to one very much too small to be measured with a sieve. If the diameter of the particle is assumed to be that dimension which would just pass through a sieve, we have a measure which is comparable with the sieve analysis. Assume a wedge-shaped particle. The diameter to be measured would be the long dimension of the base, because such a particle would be stopped from passing to a smaller sieve only by this long base dimension. If the wedge were extremely long, it still would eventually stand on end when shaken, and pass through any opening large enough to pass the base.

Assume a particle pointed at both ends and semi-elliptical; then the critical dimension would be that across the middle of the particle. Pursuing this line of reasoning, only a single measurement need be applied, namely, that diameter which would classify the material by a continuation of the sieve analysis beyond the fineness attainable by sieves.

It is evident that in no case are we interested in absolute particle size, but primarily in distribution of particle size, and that this method will meet these requirements.

#### SIZE DETERMINATION BY SEDIMENTATION

Turning now from the methods employing the sieve and direct count, we come to a consideration of those based on Stokes' law. These may be divided roughly into those which use air as a mechanical means of separation and those which use a liquid.

Stokes' law in its simplest form may be stated as that law of physics which says that, other conditions being equal, a sphere will fall through a viscous medium at a rate directly proportional to its diameter. The viscous medium may be air, or any other gas, water, or any other liquid. Common practice in powder metallurgy is to employ air or water.

The first error inherent in the application of Stokes' law to particle size determination lies in the fact that the law applies to spheres, and metal powders are not spheres. Here again, as in the use of a sieve, particle shape introduces an error which is difficult if not impossible to compensate for.

#### Separation in a Liquid

Let us consider first the sedimentation method which the student will employ in Experiment 3. A small quantity of powder is suspended in water and allowed to settle, and the amount settled is measured at various intervals. From the data it is possible to draw a sedimentation curve. A sedimentation curve is one in which the amount of material settled is plotted against time. Later we shall discuss in some detail the arithmetic involved in the calculations and the conclusions which may be worked out from the findings, but for the moment it is desirable to consider how closely this method can give accurate results.

We have considered the error introduced by shape. Next we must consider that introduced by agglomerates. It is extremely difficult to be sure that the addition of any dispersing agent will truly break up the agglomerates, and there is no way of deciding that a dispersing agent has been entirely successful. In addition to the interlocking action of irregularly shaped particles, which tends to hold them together despite vigorous agitation, there may be an electrostatic force

between particles of small size which tends to cement them. Vigorous agitation itself may introduce agglomerates by virtue of the gas bubbles caused thereby. A slight rise in temperature may cause a slight evolution of gas and particles tend to form around these fine gas bubbles. There is an upward pressure of gas bubbles which causes an eddying motion in the settling tube, and this counteracts the tendency of the particles to fall. It is to be noted that it is possible to minimize the production of gas, and in no case need the student expect to observe any noticeable evidence of it. The gas bubbles are not numerous and are very small. Agglomerates too, in a sample which has been treated with a dispersing agent, are difficult to detect. There is a question whether either of these effects is entirely eliminated. Obviously a cluster of particles surrounding a gas bubble will not obey Stokes' law. Unfortunately, agglomerates do not fall either as a large particle or as independent small ones, and again are outside of Stokes' law.

Moreover, particles of less than 3 microns develop a Brownian movement which again vitiates the application of the law; and finally in some cases we have what the writer chooses to call the ring effect. If a large cylinder is filled with dispersed particles slightly larger than colloidal size, they will not fall to the bottom evenly dispersed, so that one may expect to see clear liquid at the top of the cylinder and a well defined sediment at the bottom, with less and less dispersoid as one goes from the base of the receptacle to the top. This appearance may develop for from 10 to 24 hours, but thereafter the solution will present a series of what appear to be clear rings of water sharply marked off from rings of suspension above and below the water.

No full explanation of this phenomenon is available, but it shows clearly that small particles do not fall independently, but in relatively massive groups, and this action may occur long before it becomes apparent to the observer. These several factors make sedimentation in a liquid subject to errors which obviously make any determination of particle size by this means of only relative value and do not give absolute particle size.

### **Separation in Air**

In separation by means of air, the principle source of error is again inherent in the powder, which still refuses to conform in shape to the

sphere; therefore all calculations based on Stokes' law must be more or less invalid. The method itself, however, introduces one serious error. Air flows over and through the dry particles and keeps them in continual agitation as it removes the lighter portion from one sample compartment to another. During this separation and agitation, there is necessarily a considerable pounding of one particle against another—a far greater grinding than occurs during a sieve analysis. The attrition which takes place must increase the number of fines considerably, and at the same time reduce the size of large particles to smaller dimensions. Thus the final weighed classifications do not truly represent the size of the particles as introduced into the sample chamber, but as changed during the analysis.

Another somewhat minor error is introduced by the electrostatic force of the particles as they float from one part of the apparatus to another. The fine particles tend to cling to the sides and are extremely difficult to collect.

In spite of these faults, both the sedimentation and the air methods are extremely useful. So long as the student is aware that he is not determining absolute size, it is unlikely that any false conclusions will be drawn, and there is no doubt that for sizes below those which can be determined by the sieve, these methods are extremely useful. They are quicker than the direct count method, and the results can be more readily reproduced by different technicians and by those with less skill or experience than is required for accurate microscopic count. The Roller air separator is an apparatus which may be found in many powder metallurgy laboratories and upon which many specifications are based. In the newest machines a number of modifications have been introduced to reduce to a minimum the errors inherent in air separation.

### **The Wagner Turbidimeter**

A close rival of this apparatus is the Wagner turbidimeter. This machine uses a light analyzer to determine the rate of fall of particles in a liquid suspension. Added to the errors which occur in any sedimentation method are those introduced by the application of light. Materials differ in the way in which they absorb and scatter light rays. Therefore, besides introduction of false values by variation from the sphere, we have variations in the recorded size introduced by the varying reaction of different materials to light. How-



ever, if the turbidimeter is used for a single material, such as a copper powder or an aluminum powder, it will serve very well for the particular powder under examination. Note, however, that serious errors are introduced when the data determined on analysis of one material are applied to another which has entirely different characteristics toward light.

More simple methods of determining sedimentation curves can be devised in any reasonably well equipped laboratory and are subject only to the errors inherent in any method based on Stokes' law.

### **The Balance Method**

The commonest of these is the balance method. In this method the pan on one side of a balance is extended so that it floats freely in a large cylinder containing the powder dispersed in water. The mixture is agitated long enough to assure a perfect suspension and the suspended pan is balanced. At intervals several minutes apart, the increase in weight is recorded. This, of course, is due directly to the increased number of particles which have fallen upon the pan during the interval. From these data a sedimentation curve may be drawn.

### **The Pipette Method**

The pipette method, a variation of which is used in Experiment 3, page 135, is somewhat more tedious and requires a more experienced technique, but has the advantage that no special balance need be added to the laboratory equipment and only simple, readily available material is required. A series of long test tubes marked off 20 cubic centimeters from the base, and with another mark 20 centimeters above the first, are set up in a test tube rack. Into these is introduced an approximately 2 per cent suspension of the powder under examination. The tubes are agitated and then the suspension is allowed to settle. The material in the 20-centimeter column is withdrawn with a pipette from different tubes at intervals and discarded. The 20 cubic centimeters of suspension remaining, to which has been added now all the powder which settled during the interval, is filtered and the filtrate weighed.

The increase in weight gives the analyst the amount of material which has settled in the interval and from this he may draw a sedimentation curve. This is an outline of the method suggested by

Tickell.<sup>10</sup> The writer has made some changes in this method in the interest of economy of time and of increased accuracy.<sup>11</sup> While the published method is easy to follow when using light powders, which may be quantitatively removed from the long test tubes employed, when using heavy metal powders it is extremely difficult to wash them from the base of the test tube to the filter paper. After about 30 washings some of the powder is still found in the test tube, and all methods of washing with streams of water fail unless great volumes, considerable force, and an awkwardly long wash-bottle tip are used. To overcome these difficulties, the writer prefers to weigh the material which is still suspended in the 20-centimeter column at the end of the desired interval. Although the material removed by an ordinary pipette may be readily weighed, it is difficult to remove this and retain it safely in the pipette quickly enough to avoid introducing a time error. A specially devised pipette is explained in Experiment 3, and illustrated in the frontispiece.

### Adsorption Methods

Particle size distribution cannot be measured by adsorption methods, but some key to the total surface per unit of powder can be determined by this method. The principle may be applied by passing a gas through a powder and measuring the quantity of gas adsorbed, or by mixing the powder with a solution, usually a dye, and determining the amount of material adsorbed from the solution. All other conditions being equal, the quantity of gas or material adsorbed by the powder bears a direct relation to the surface exposed to the gas or solution. The results must be compared to some standard powder, the surface of which has been determined or estimated by some other method. The result, if expressed in terms of surface, defines an entirely different surface from that defined by other analytical measurements. It bears no relationship to the diameter of the particles, since it measures surfaces within the theoretical sphere. The surface of a porous particle, when measured by other methods, is the peripheral surface and does not include that presented by the pores. The efficiency of adsorption depends upon the total surface exposed to the testing medium; a dense particle with a diameter of 10 microns

<sup>10</sup> Frederick G. Tickell, "Examination of Fragmental Rocks," pp. 11-15, Stanford University Press, 1939.

<sup>11</sup> See Experiment No. 3, page 135.

would present a considerably smaller total surface than one of the same diameter, but porous. Therefore it must be understood that adsorption methods do not give any index to the actual size of particles.

### **Special Microscopes**

The ultra-microscope can be used to measure and classify particles within its range, and is subject to the same criticisms which have been raised in regard to an ordinary microscope. This range is necessarily small. For determining geometric properties, shape, size, etc., or size distribution of very fine particles, we now have a new and remarkable tool in the electron microscope.<sup>9</sup> Its wider use will give the powder metallurgist a much better picture of the shape of the particle on which the entire industry is built than anything previously available. The picture that may be obtained by the use of this microscope is so clear that all questions of doubt regarding shape can be resolved by appeal to the equipment.

### **Control with a Micrometer**

An interesting method of control, in regular use in some of the large sugar refineries, has recently been brought to the writer's attention. Dealing with powdered sugar, all minus 400 mesh, it is necessary to make sure that no particles larger than this go to the consumer. It has been customary to take a catch sample which at the end of an hour amounts to more than 10 lbs., and reduce this by quartering to a gram, which is spread on microscope slides. The slides are studied to determine whether the previous run contained any over-size particles. The procedure is time-consuming and requires a skilled technician.

A new method of control which has been introduced is extremely simple and can be readily performed by the operator in charge of the screens. It is only necessary to make a thin cream of a sample of the fine sugar by mixing it with a thin mineral oil. A drop of this is placed on a micrometer and the micrometer slowly closed down, applying pressure with a ratchet only. It is easy to examine many samples in a few minutes and the micrometer will show the largest particle present in the sample. The writer has applied this to copper powder, 100 per cent through 325-mesh, and to a number of other metal powders, but has not attempted to standardize the procedure or to check it against other methods of measuring particle size.

The results, however, have been promising enough to justify some research work on the application of this technique to control methods by firms who must produce powders all below a certain maximum particle size.

### **The Hydrometer Method**

Rogers<sup>12</sup> describes a method of determining particle size by a method which appears to be simple and quick. He points out that the hydrometer does not measure the specific gravity at a point, but only the average specific gravity over a rather wide depth. The volume and the dimensions of the hydrometer must be known, as well as the cross-section of the cylinder in which the measurements are made. If sufficient care of a number of points of technique are controlled, good and reproducible results may be obtained, though the inherent errors cannot be avoided. Stated in its simplest form, the hydrometer is used to measure the specific gravity of a one or two per cent suspension of the powder in water at various time intervals. From these readings a cumulative figure showing the percentage finer than a given size may be calculated. This method appears to have a number of points in its favor.

<sup>12</sup> Morgan F. Rogers, "Powder Metallurgy," Edited by John Wulff, p. 204, A.S.M., 1942.

## Chapter 6

### Cohesion

Before any attempt is made to interpret the results obtained by pressing and sintering, it is necessary to understand the forces which come into play during these processes. There is probably no more disputed factor in metallurgy, and certainly not in powder metallurgy, than cohesion. What force binds together a fused mass of crystals? More pertinently, what force makes one surface cohere to another? It is best to say at once that we do not know. Any number of scholarly papers have been presented on the subject, but the writer is unaware of any which, when faced with a specific instance, does not fail to explain the phenomenon fully. The theories may generally be classed as: (1) those which attempt to explain this factor in terms of a difference of potential between the surfaces; (2) surface-tension activity; (3) an interatomic force; (4) mechanical interlocking.

#### Theories

If we examine these theories from the point of view of plain language interpretation, we will see that only the last avoids begging the question. There is little to support the theory that mechanical interlocking of two surfaces is a major factor toward producing a bond strong enough to resist rupture. However, it must be taken into consideration when considering surface relations. It does have a minor influence.

As for the others, it is impossible to attach a physical interpretation to the terms "surface tension" or "interatomic force." When we say "surface tension" we *explain* nothing. We merely label an observed phenomenon; and when we talk of "interatomic forces" we interpret an unknown in obscure terminology.

As to the theory which attempts an explanation on the basis of a difference of potential, in many cases it is possible to measure a surface potential on two surfaces and to discover that the potential is

either positive or negative on both surfaces and of equal magnitude. It has been claimed that these measurements are for a gross surface, and the force postulated is one arising from a difference of potential between discrete particles. This again would seem to be begging the question since, if it is impossible to discover any difference of potential, we are talking metaphysics when we postulate an unmeasurable force.

The writer prefers to describe the phenomenon as an example of surface tension and frankly admits that this is merely a label for the phenomenon. That such forces are familiar to every physicist and can be measured makes this a useful method of approach and has the further advantage of employing familiar terms. To go further than this and attempt to define what surface tension is, except as it functions, is to enter the realm of pure speculation.

We are, however, familiar with the factors which influence surface tension, and thus we can examine powder metallurgy phenomena in a familiar medium. The outstanding fact of powder metallurgy is that the surface forces are brought into play on a vastly increased scale. Compared to the force displayed in gross surface phenomena, this is so much greater that some phenomena appear to be different in kind rather than in degree. We can grasp the magnitude of the increased surface if we consider, for example, the surface of a particle 1 cc. in volume. This will present six square centimeters of surface. If we subdivide this particle sufficiently to pass through a 325-mesh sieve, we have increased the total square surface from 6 to 384,000 square centimeters. Whatever surface forces operate have been multiplied by 64,000 times; and from our point of view whether these forces be due to interatomic attraction, to difference of potential or any other theoretical consideration, the patent fact is that they have been increased many thousand fold.

It must not be thought that such force may be observed only when particles are of this small magnitude. It is true, however, that there are phenomena which, within our present knowledge of the subject, appear to be different in metal powders than in massive metals. Some of these may be touched upon later. For the moment let us consider those phenomena which may be observed in massive metals. The most significant of these Jones<sup>13</sup> has labeled "cold-welding."

<sup>13</sup> W. D. Jones, "Powder Metallurgy," p. 2, Longmans Green, London, 1937.

### Cold-Welding

If two halves of a material freshly parted are brought into contact, they cohere with sufficient strength to require considerable force to separate them again. Several precautions must be observed, and a number of factors influence the strength of the bond.

One of the most interesting experiments that has been reported is that of G. A. Tomlinson,<sup>14</sup> who lightly draws a glass bead across the surface of a clean glass plate. In the course of pulling the bead across the plate a series of sharp jerks may be noted when the bead momentarily resists the pull of the experimenter. On microscopic examination of the plate a sharply etched dotted line is discovered as the result of the bead adhering to the surface of the glass and actually tearing out particles from the surface.

We are, of course, familiar with the phenomenon of "wringing" gauge blocks.<sup>15</sup> A gauge block is optically flat, and when placed in contact with a similarly prepared surface it requires a force of 90 lbs. to the square inch to separate the blocks. Part of this may be attributed to atmospheric pressure, but this leaves a residual force to be accounted for by, shall we say, "cold-welding"? It is a good phrase. Glass optical flats placed in contact require a force of 654 lbs. to the square inch for separation; and it has been suggested that under a microscope a glass optical flat appears to have a much smoother surface, more free of the marks made by the lapping operation, than do gauge blocks. Thus there is more true surface-to-surface contact in the latter case. But these figures fade into insignificance when we read the report of Beilby,<sup>16</sup> who found that metal filings adhere to glass or porcelain with enormous strength, and cites as an example gold leaf adhering to glass with a force of from 0.93 to 23.32 tons per square inch.

We need not prepare any elaborate experiment to become acquainted with this phenomenon. It is merely necessary to shear lead, tin, zinc, cadmium, copper, or gold, and place the sheared surfaces in intimate contact a few moments after shearing. The experimenter will find in most cases that it requires considerable force to separate them where they have been joined only by light pressure of the hands. Or we may draw out a fine glass thread and break it

<sup>14</sup> G. A. Tomlinson, *Proc. Roy. Soc., London*, **A115**, 472-83 (1927).

<sup>15</sup> Lord Rayleigh, *Ibid.*, **A156**, 326-49 (1936).

<sup>16</sup> G. Beilby, "Aggregation and Flow of Solids," London, 1921.

after it has cooled. If the two portions are placed parallel to each other and not too far apart there is sufficient attraction (surface tension) so that they leap the gap and cohere with considerable force. If the threads are fine, it will be impossible to separate them without breaking them in cross-section.

### **Clean Surface**

With these experiments as evidence, we become aware of the fact that strong cohesion occurs without the aid of heat or pressure. It must be said here that clean surfaces are essential to produce this phenomenon. Here it may be well to define what is intended by the term "clean". This is most easily done by employing the definition of its antonym. Dirt may be defined as matter out of place. A freshly sheared metal may fail to cohere if it has been wiped with even the cleanest of fine linen. A trace of grease on either surface will prevent cohesion entirely. On the other hand, slight surface oxidation does not always prevent cohesion.

Even after lead has been parted for a period of hours, when placed in contact it will usually cohere provided that no dust or any other material has fallen on the surface. It is possible too, after a relatively thick oxide film has formed, to disturb the oxide enough to recover the forces of cohesion. It is necessary only to rub the surfaces to cause sufficient attrition to displace and redistribute the oxide. This is because, as the attrition removes the oxide, it exposes the freshly cleaned surface, and it is probable that the cohesion is due to the contact of such surfaces. Soft lead permits the oxide to be driven below the surfaces. In harder metals, once an oxide is formed it becomes much more difficult to bring back into operation the forces of cohesion. This may be explained on the theory that though fresh surfaces are exposed as the oxide is redistributed, the fresh surfaces cannot be brought into contact because of the rigidity of the harder metal. The oxide remains as an interphase. In the case of lead and more plastic materials, surfaces may be deformed sufficiently to make contact in spite of the oxide, which is probably forced below the soft surfaces.

### **Plasticity**

The effect of plasticity is particularly observed if experiments are performed with Celluloid or mica. Lightly placed together, the



irregularly split mica or Celluloid coheres very slightly but, if the sheets are bent back and forth and manipulated so that advantage is taken of their flexibility to materially increase the contact, the cohesion is great, in the case of mica amounting to 550 lbs. per square inch.<sup>17</sup> Therefore we may say that the importance of two factors is illustrated by these experiments: first, a clean surface is essential; secondly, plasticity is desirable.

In this connection it should be noted that it is not easy to produce a clean surface. In the above experiments clean surfaces were obtained because they were freshly made. In some cases even a fresh surface may not be clean of oxide in the sense in which we are using the term "clean." For instance, if cast iron is broken, it is impossible not only to make the fresh surfaces cohere, but to amalgamate them in a mercury bath. Cast iron is so rigid that after our studies of the above phenomena illustrating the significance of plasticity, it need not surprise us to find that we cannot make its surfaces bond to each other. However, mercury which is entirely fluid can make intimate contact over the entire surface; so some other factor is at work. We say that mercury will not "wet" cast iron, but this is true only because of the oxide and adsorbed gas film which forms immediately on exposure of the fresh surface. If the cast iron is broken under the surface of a mercury bath, the fresh surfaces immediately amalgamate.

### Interpretation of Solder Flux

Continuing our discussion of producing a clean surface, we find that it is possible to wash freshly prepared surfaces with benzol or alcohol, and in some cases even water without in any way reducing the surface tension phenomenon. At the same time, such washing will not produce by itself a clean surface. Every solderer is aware of the need of a flux. The function of a flux is to clean while not itself wetting the surfaces being prepared. It absorbs any interfering film and leaves behind a clean surface. If the flux itself wetted the material it could not serve the purpose, for some of the flux would remain on the surface and would itself introduce foreign matter as an interfering phase.

These experiments show that great care is required to obtain surfaces which exhibit cold-welding. The manufacturer of parts molded

<sup>17</sup> J. M. Macaulay, *J. Roy. Tech. College, Glasgow*, 3, 353-71 (1935).

from powder must take every precaution to employ powders free of grease, and even surface oxide films are to be avoided. He frequently resorts to a preliminary treatment of powders just before they are used—treating them at elevated temperature in a current of hydrogen, for instance—to secure as perfect a surface as possible. The operators of metal spray guns take pains to prepare a satisfactory surface before spraying it with metal, and the manufacturers of friction plates electroplate the steel backing with silver or nickel before sintering the powdered metal to its face, in order to secure a perfect bond between the steel and the graphite bronze section of clutches and brakes.

On the other hand, with the perversity of the inanimate, welding takes place when it is least wanted. Without lubrication, moving parts not only heat but frequently “freeze.” The faces of tapered and ground valve plugs often freeze in valve seats, and the several processes for the reduction of iron ore to produce sponge iron failed largely because the freshly formed iron clung to the walls and to previously formed iron until it literally filled the exit end of the furnace.\* So, too, newly made carbonyl iron will freeze to the walls of the reaction chamber and build up until it is closed, unless special precautions are taken.†

### The Function of Heat

Considerable has been written about the subject of cold-welding, but it is obvious that although the strength of a pressed product is not the result of fusion, the heat treatment does do something to increase the cohesion. If the strength is primarily due to surface tension, then the heat treatment must improve the surface-to-surface contact of the particles. We know definitely that in the formation of practically all products of metallurgy there is an optimum temperature which gives products of the greatest strength, and that beyond this temperature a sharp decrease in strength occurs. This does not accord with the classical concept that a fused material recrystallizing will have greater strength than materials merely brought together under pressure, but it does fit in with our own approach; and with this in view we may examine what effects of heat treatment improve the surface-to-surface contact and make surface tension more effective after heat treatment, than after cold-pressing alone.

\* See page 33.

† See page 34.

### Strength in a Solid

Before going into this, however, it might be well to examine the cause of strength in any solid metal product. Why is the tensile strength of one ingot greater than that of another? In the final analysis the strength must depend upon either the strength of the crystal or the strength of the boundary. Although there is at least one exception, namely tungsten, it is generally claimed by physicists that the strength of a solid metal depends on the strength of the boundary. In other words, the boundary phase is always considerably stronger than the individual crystal. It is much easier to break a coarse-grained than a fine-grained metal. In a metal with extremely fine grain, the proportion of boundary to crystal phase is considerably higher than in a coarse-grained one. If we fracture a metal, examination under a microscope will show a crystalline surface, but the crystals have all been broken off smoothly. We are seeing a cross-section of the crystals where they have broken. The boundary phase will, of course, have broken at the same time.

Contrary to this, if we fracture a piece of tungsten we find an extremely irregular surface studded with whole crystals which have not failed; the failure has taken place in the boundary, and the crystals were literally pulled apart, one from another. We do not break them as we do when we break or fracture most metals: we break the boundary phase.

Now, in a fine powder, every particle carries with it an envelope of gas, whether it is the gas which formed the film during the production of the powder or whether it is the gas picked up during transportation. Although pressing will displace some of the air films, it cannot remove the atomic envelope entirely. But as heat is applied, the gas is driven off, and as this happens, a more intimate contact between particle surfaces is created. Although we know little about it, it also appears evident that the effects of surface tension are increased as the temperature is raised. Other phenomena occur during the heating. We must consider recrystallization and crystal growth.

We know that at all times these two phenomena take place more or less simultaneously, and that at elevated temperatures they occur at an appreciable rate of speed. Even at such low temperatures as may be derived from steam under low pressure, the crystal growth of copper tubing is pronounced, and frequently such tubing fails. Examination shows that there has been what is commonly called

granulation. Granulation may be described as the growth of crystals already present. The crystal grows at the expense of the boundary phase. What has happened is that we have increased the ratio of crystal phase to boundary phase, and since the crystal phase is considerably weaker than the boundary phase, a small external force will rupture the metal at this weak point. In considering what happens when we heat a compact, we must face the fact that such crystal growth will undoubtedly take place at elevated temperatures, but at this point we have very little crystal-face to crystal-face boundary phase. There is the third phase of the gas envelope. Simultaneously with driving this envelope from the particle, we increase the growth of the crystal, which now takes the place previously occupied by gas. Thus, instead of weakening our boundary phase and in spite of the fact that we are increasing the size of the crystal we have actually produced a more perfect boundary phase. It is probably true that only in unique cases do we induce recrystallization, as it must start from a fresh nucleus, and such nuclei form rapidly from impurities only at high temperatures.

Oxide, for instance, constitutes such an impurity, and each particle of oxide would be a starting point for the growth of new crystals. However, new crystals cannot form unless we have a liquid phase, and in many cases no liquid phase is present at the temperature of sintering. So much for crystal growth and recrystallization.

### **Network Phenomenon**

We have now to consider another phenomenon not uncommon in powder metallurgy. This may be classified as the network phenomenon. In many cases—the outstanding example being the cemented carbides—the temperature of sintering, although not high enough to melt the predominant metal, is sufficiently high to melt the metals of the mix which are present in small quantities. Thus, in making cemented carbides, the nickel or cobalt in quantities from 3 to 13 per cent and intimately mixed with the carbide, is subjected to a temperature which is high enough to melt it. It naturally flows to fill in cavities between the carbide particles, and when the product has again reached room temperature, the once-fluid metal freezes in this new position. Thus it acts as a net in which the harder carbide particles are confined.

When it is desirable to depend on the network phenomena, it is

very important that we consider the physical affinity of the metal used to form the network. We are all familiar with the fact that many materials wet each other easily, while others seem to repel each other. Thus it is possible to pour mercury from glass vessel to vessel without leaving any drops adhering to the walls, whereas water always leaves a residuary deposit; this we call wetting. Cobalt clings closely when fused to tungsten carbide, but it has little affinity for titanium carbide. Therefore in tools made of tungsten carbide cobalt is used as a binder, whereas in tools made of titanium carbide nickel is used, as it has an excellent affinity for titanium carbide but a poor one for tungsten. Cobalt and nickel, however, are readily soluble in each other. Many modern tools are made of combinations of tungsten and titanium carbide, and it has been found desirable to use a mixture of cobalt and nickel as the cementing medium for these.

### Plasticity

We have not yet considered another phenomenon induced by heat. With rising temperature, we have increased plasticity. This means that the particles which are already under enormous stress reach a stage where the plasticity is sufficient to concede deformation to partially meet this stress. Thus the particles are pressed in more intimate contact, and this also increases the effect of surface tension.

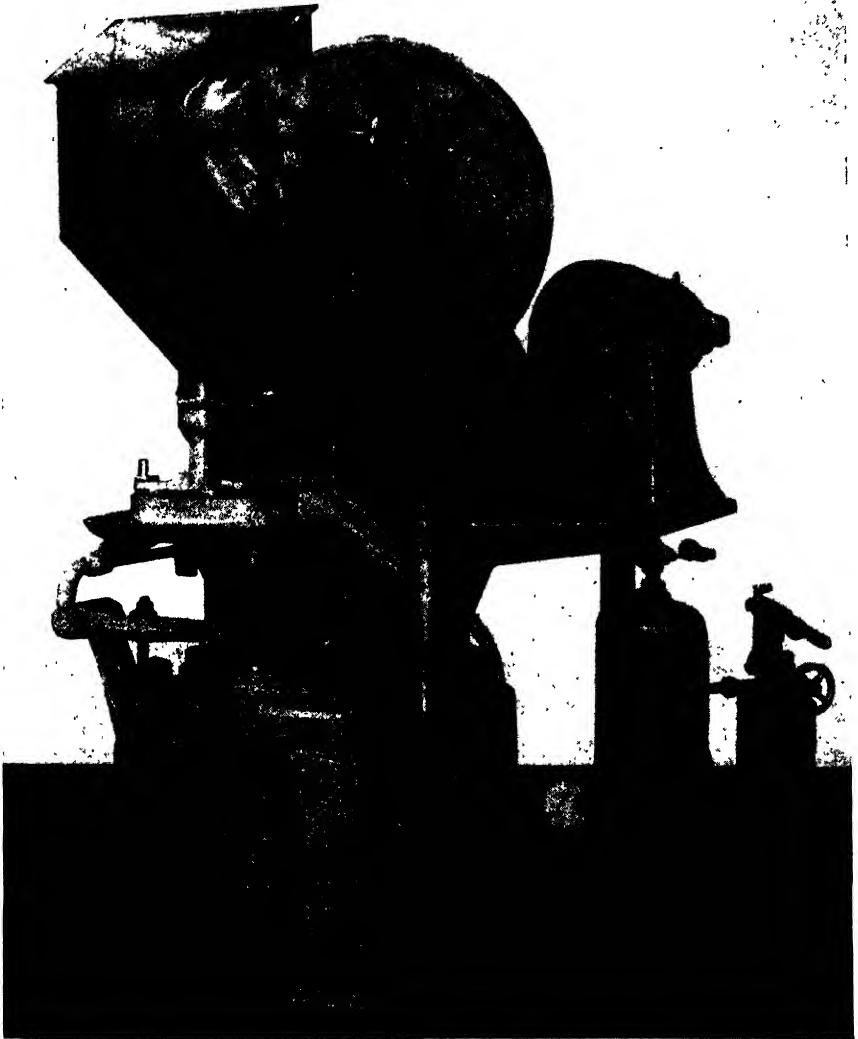
Summarizing the effect of heat, we may say:

- (1) It accelerates and increases the effectiveness of surface-tension reactions.
- (2) It removes the interfering gas phase.
- (3) It promotes the growth of crystals to fill the voids vacated by the gas envelope and promotes crystal interlocking or keying.
- (4) It may produce better mechanical interlocking by building a fluid network.
- (5) It increases plasticity and thus induces better surface-to-surface contact.

### Pressure

We may now consider the effect of pressure upon powders. The motion of the press as it descends into the die sets up enormous frictional effects which produce heat at the surface of each particle and a considerable degree of attrition. This grinding of particle against particle removes microscopic, perhaps molecular, irregularities

and so scrapes the surface of each particle as to create a number of fresh, clean surfaces. This in itself makes possible a greater area



*Courtesy F. J. Stokes Machine Co., Philadelphia, Pa.*

A new model compacting press; it applies a pressure of 20 tons from both top and bottom, and has independent ejection and hydraulic or pneumatic density equalizer and release.

effective for surface reaction. Not only are the physical irregularities of each particle reduced, but surface impurities are displaced and the adsorbed gas is squeezed from the mold. This does not mean that

under any pressure it is possible to remove the last trace of gas, but the major portion is removed.

During this squeezing action the internal temperature is raised sharply, and this promotes plasticity. Since the plasticity aids in deformation of the particles, a keying action develops; and if the timing of the pressure is good and the particle size such as to promote flow, bridging and voids are filled in. Even when bridging occurs, the size of the void is greatly reduced. We may say then, that the effects of pressure are:

- (1) Frictional cleaning and the production of fresh clean surfaces.
- (2) Removal of most of the gas film.
- (3) Reorientation of surface impurities.
- (4) Breakdown of bridges.
- (5) Increased plasticity resulting in better surface-to-surface contact.

We saw that the favorable effects of heat reach an optimum at some narrow temperature range determined by the particular metal or mixture of metals treated. Beyond a certain temperature, crystal growth becomes excessive, and below a certain temperature it does not reach its full effectiveness. The favorable effect of pressure approaches a maximum, and a study of pressures shows that beyond a certain pressure the increase in density, tensile strength or hardness is so slight, regardless of how much pressure is employed, that it is uneconomical to increase it beyond this point. The effect of such pressure is greatest with very fine particles. Working with copper particles all smaller than two microns, Trzebiatowski<sup>18</sup> produced compacts with a density greater than 8.7 for pressures of 200 tons per sq. in. This was within 3 per cent of the maximum density attainable in solid copper. However, at 150 tons per sq. in. his density was over 8.6 and at 100 tons per sq. in. was nearly 8.5. Thus, his maximum density at 200 tons per sq. in. was but little better than densities achieved at 100 tons per sq. in. On the other hand, the density increased from 7.5 to about 8.5 as pressure increased from 50 to 100 tons per sq. in.

An examination of the results of Goetzel's<sup>19</sup> work, which is a study of a number of copper powders, indicates that in general there is a

<sup>18</sup> W. Trzebiatowski, *Z. Phys. Chem.*, **B24**, 75-86 (1934).

<sup>19</sup> C. G. Goetzel, "The Influence of Processing Methods . . . On Copper Powders," Plate 37, Columbia University, 1939.

sharp increase in density from between 5.5 and 6.5 at 10 tons per sq. in. to over 7.5 at 45 tons per sq. in. At 70 tons per sq. in. density had nearly reached the maximum he could obtain from these powders, slightly under 8.5. Thereafter under pressure as high as 190 tons per sq. in. density increased less than 0.3 per cent. One powder which he was able to compress to greater density reached 8.7 under 70 tons and was no denser at 190 tons. In commercial practice it would seem that seldom would there be justification for pressing beyond 100 tons per sq. in., as the relatively slight gains in density as pressure exceeds this figure can hardly compensate for the extra cost of equipment and tremendously increased wear on the die and other parts. On the other hand, it may often be desirable to use pressures above 50 tons per square inch since below 100 there is a noticeable gain in density as the pressure is increased.

### Hardness

As density increases with increased pressure, there is a steady but not so pronounced increase in hardness. Trzebiatowski<sup>18</sup> found in his study of copper, that the hardness increases, for example, from 70 Brinell at 10 tons per sq. in. to a little over 100 at 50 tons per sq. in., 160 at 100 tons per sq. in., and 180 at 150 tons per sq. in. At 200 tons per sq. in. there is no change from this maximum figure. Goetzel<sup>20</sup> found a Brinell hardness of about 20 for cold-pressed compacts made under 10 tons per sq. in. pressure, and this increased to about 80 when the compacts were made at 45 tons per sq. in. With 70 tons the hardness increased, but to less than 100 at both 70 and 100 pounds compacting pressure. At 190 tons the Brinell of the coppers he examined averaged about 120. One powder, which reached the highest Brinell (95) at 45 tons per sq. in., was but little over 100 at 100 tons per sq. in., and at 190 tons per sq. in. was still less than 115.

Hardness is not directly related to porosity, but the two curves are similar. This is not surprising if we accept the usual explanations for increasing hardness. Hardness is due to crystalline deformation or plastic deformation. Crystal slip sets up internal stresses and distorts the normal crystal face arrangement. The pressure exerted in a mold does just this. It does distort and deform, and it is thus logical to expect that pressure will increase hardness; but there comes

<sup>20</sup> *Loc. cit.*, Plate 56.



a maximum beyond which no further distortion is possible. Basically, work-hardening is an effect produced by similar crystal deformation, and the frictional abrasion which takes place as the particles work against one another in the mold is another activity which promotes hardness through work-hardening.

An excellent example of work-hardening is easily observed if hardness is tested by means of a scleroscope. If the test piece is not moved after the steel ball has rebounded, and the same spot is tested again, the second reading on the scale will be considerably higher than the first; and additional tests of the same spot show repeated increase in hardness.

### **Pressure and Heat**

The densities and hardnesses we have discussed are those obtained by pressure alone. In practice it is necessary to heat-treat the compact after it is pressed; in other words it is put through the sintering process to give it the high coherence necessary to meet the tensile strength requirements. This treatment has a marked effect on the hardness and density. As might be expected, the sintering operation acts as any annealing treatment and, according to the temperature employed, effects a reduction in hardness not directly related to the pressure employed to make the cold compact.

But the effect on the density is directly related to the pressure. The density of the sintered compact may increase or decrease from that of the cold-pressed compact. In general a compact made under low pressure may retain the same density, or the density may increase under heat treatment. A cold compact made under high pressure will decrease in density during the sintering operation. The trapped gases can find no channels through which to escape, and literally blast their way out, forcing the closely packed particles apart.

### **Flow**

The commercial manufacturer of metal products must be constantly aware that although loose powders may exhibit all the characteristics of a fluid, compacted powders do not follow hydrostatic laws. Pressure is not transmitted equally in all directions; and compacts vary from end to end in tensile strength and hardness. This is particularly true of those made at low pressures, but it sometimes

occurs when pressures as high as 100 tons per sq. in. have been used. To reduce this variation as much as possible, it is necessary that the pressure be exerted equally and efficiently at both ends of the compact. Even then, the weakest spot will be found farthest from the ends of the rams. If the compact is pressed at both ends, this weak spot will be in the center, and similarly the center will be the softest and least dense.

### Time of Sinter

The strength of the final compact is the resultant of several forces. There are the effect of pressure; of temperature; to a minor extent, of period of heating; and very noticeably the effect of occluded gases. The effect of the period of heating is very marked during the first short period. It is also somewhat dependent upon the metal under study; each may have a characteristic curve at different temperatures or for compacts formed at different pressures. In general, it may be said that there is a sharp increase in favorable characteristics during the first five minutes of heat treatment. There is little change for the next 15 minutes, and then a sharp decrease in the case of iron, though no change is noticeable in copper or bronze. Thereafter, for six hours the curve of most metals remains relatively flat, and after this length of heating a slight decrease occurs which is hardly appreciable even after 10 hours.

In the case of copper heated to 715° C., copper and tin, or copper and zinc alloys, there may be a slight increase in density and tensile strength, for instance, which progresses for six hours, and thereafter there is a sharp decrease. With pure copper compacts pressed at 24 tons per sq. in., the typical density reaches 7.8 after 15 minutes of heat treatment, and after 30 minutes it drops to 7.7. Six hours later this has increased to about 8.1; but after 10 hours it has fallen to nearly 7.0. In the meantime, the tensile strength of pure copper at the end of 15 minutes is up to 7½ tons per sq. in. At the end of 30 minutes this has reached nearly 10 tons, and after six hours is about 12 tons. If the heating period now continues for 10 hours, the tensile strength falls to less than 7 tons per sq. in.<sup>21</sup>

Goetzel's<sup>22</sup> work does not show this for all copper powders. Two of the powders he examined showed no change in density after being

<sup>21</sup> F. Sauerwald and E. Jaenichen, *Z. Elek. Chem.*, **31**, 18-24 (1925).

<sup>22</sup> *Loc. cit.*, Plate 41.

heated for 2 or 16 hours, but 8 others showed a constant increase with time from an average of about 7.8 at the end of 2 hours to about 8.1 at the end of 16. His results were obtained on copper powder compressed at 25 tons per sq. in. Compacts made at 45 tons per sq. in. showed practically no change in density whether heated for 2 hours or 16, though in most cases there was a very slight increase. The writer's experience with bronze compacts pressed at 40,000 lbs. per sq. in. shows that for periods of from 5 minutes to 6 hours at a temperature of 800° C. there is no change of density within the limits of error. For hardness Goetzel reports a slight increase in hardness with increase in sinter period, but this is very slight. The writer too has found in many cases little change in hardness due to increased periods of heating, after the first 15 minutes.

Iron heated at 870° C. has a tensile strength of 5 tons per sq. in. at the end of 15 minutes. At the end of 30 minutes this drops slightly but rises sharply to 6 tons per sq. in. after the first hour. After the fourth hour this has further increased to about 7 tons and it falls off at the end of 6 hours to a little over 6 tons. At the end of 10 hours no change is observable. The density curve is practically parallel. The density at the end of 6 hours, 10 hours or more and 15 minutes is just under 7.0, while at 30 minutes it is about 6.8 and at the end of 4 hours is about 7.1.<sup>21</sup>

### Occluded Gas

Many of the contrary effects on heated compacts may be attributed to the effect of occluded gas. In general, a lightly pressed compact has a greater density after heat treatment than an excessively highly pressed compact. In fact, when light pressures are used, shrinkage is a common phenomenon and the finished compact has smaller dimensions than the green. On the other hand, a compact which has been subjected to 25 or 50 tons pressure usually expands when heated. If we consider that enormous forces, strains and stresses are set up in a pressed compact, and if we visualize the fact that the occluded gas must escape through whatever pores remain after pressing when heat is applied, it is easy to understand why these effects are produced. A lightly pressed compact leaves relatively large pores, and the gas may escape without deforming the channels left after pressing. On the other hand, a compact which has been subjected to high pressure contains no passageways large enough for the

gas to escape through, and the expanding gas pushes the particles apart in its effort to escape. This internal pressure naturally expands the compact and causes a larger and more porous product than that originally formed. Note again, however, that the final shape and density of the product depend upon several other factors, some of which possibly come into play to offset the effect of gas expansion.

### Source of Gas

We now consider the source of this gas. The largest portion of it undoubtedly arises from the gas envelope which clings to each particle. It should be noted, for instance, that if we heat particles of copper or iron, -325 mesh, for several hours in a vacuum at 440° C., allow them to cool in a vacuum and then expose them to air, they will pick up appreciable quantities of gas. For instance, at the end of 1 hour, 5.5 milligrams of air will be occluded by 100 grams of copper powder and 20.5 mg. of air by 100 grams of iron powder. At the end of 20 hours the copper will pick up 8 mg. of air per 100 grams and the iron 23 mg. per 100 grams.<sup>23</sup> This air consists of oxygen, nitrogen and moisture, and if the product is sintered in carbon or in a hydrocarbon, reaction with the air will take place, which may form carbon monoxide, carbon dioxide, methane and hydrogen, or a mixture of several of these gases. If the reduction takes place in a hydrogen atmosphere, more water vapor probably will be formed. Note too, that if graphite has been added to the mixture, the hydrogen may produce hydrocarbon gases, and that the carbon itself may react with the occluded air to give carbon monoxide and dioxide. Ruer and Kuschmann took care to produce a gas-free powder, and during exposure to the air it was not subjected to the agitation which occurs when powders are shipped. Commercially produced and transported powders contain more air than this, for though there is no reason to expect a greater increase in the quantity of surface film, a certain added amount is entrained in agglomerates.

In addition to the gases adsorbed and trapped by the press or entrained in agglomerates, there are gases which are occluded by the metal during its production. Thus, in the case of refined copper there is occluded as much as 2 cc. of gas per 100 grams of metal; reduced copper occludes 3 cc. of gas per 100 grams of metal, and

<sup>23</sup> R. Ruer and J. Kuschmann, *Z. Anorg. Chem.*, **173**, 233-61 (1928).

electrolytic 8 cc. Analysis of the gas shows that that from refined copper consists approximately of 60 per cent sulfur dioxide, 15 per cent hydrogen, 20 per cent carbon monoxide and 6 per cent nitrogen. The gas from reduced copper analyzes 10 per cent sulfur dioxide, 35 per cent hydrogen, and 55 per cent carbon monoxide.

In electrolytic copper the gas consists of 10 per cent sulfur dioxide, 40 per cent hydrogen, and 50 per cent carbon monoxide. In the case of nickel, the volume of gas per 100 grams of metal is enormously high except in the electrolytic product. Thus, the cube nickel contains 500 cc. per 100 grams of metal; granulated nickel 800 cc.; Mond nickel 100 cc.; and electrolytic 8 cc.

The analysis of the occluded gas in the cube nickel approximates 2 per cent carbon dioxide, 90 per cent carbon monoxide, 4 per cent hydrogen and 4 per cent nitrogen; granulated nickel gives a trace of carbon dioxide, 93 per cent carbon monoxide, 5 per cent hydrogen and 2 per cent nitrogen; electrolytic, 20 per cent carbon monoxide and 80 per cent hydrogen. Mond nickel gas is 3 per cent carbon dioxide, 72 per cent monoxide and 25 per cent hydrogen.<sup>24</sup> (For convenience figures have been reduced to round numbers.)

The gas which may be evolved by pressure alone from a carbonyl iron divides roughly into 66 per cent carbon dioxide and 33 per cent carbon monoxide. The gas may amount to a considerable quantity.

The effect of the presence of gas or the formation of gas during sintering is dramatically illustrated by the work of Trzebiatowski<sup>25</sup> who compared the effect of heating identical powders compacted under different pressures, as it applied to porosity. He used copper powder, all smaller than 2 microns. One set of compacts was made under 200 tons per sq. in. pressure and one set under 40 tons per sq. in. The former had a density of 8.6 at room temperature, and the latter a density of only 7.1. But when these compacts were heated to 400° C. the density of the high-pressure compact fell to 7.5 and that of the low-pressure one rose to slightly more than this. At 600° C. there was no appreciable increase in the density of the low-pressed compact (7.6), but the compact pressed at 200 tons per sq. in. was now less dense than the low-pressed compact had been at room temperature — 6.9. It is evident that the latter permitted the gases to escape easily and the voids were filled by deformation of the copper, whereas the gases in the high-pressed compact had forced

<sup>24</sup> W. Hessenbruch, *Z. Metallkunde*, **21**, 46-55 (1929).

<sup>25</sup> W. Trzebiatowski, *Z. Phys. Chem.*, **B24**, 75-97 (1934); **A169**, 91-102.

channels of escape that left the heated compact porous. Working with gold powders, Trzebiatowski reported similar results. Gold pressed under 200 tons per sq. in. had a density of about 17.5, but under 40 tons per sq. in. its density was slightly under 16.0. However, at 400° C. the heavily pressed gold had a density of but 16, which was exceeded by the low-pressed compact at this temperature. At 500° C. the density of the low-pressed compacts had increased to about 16.5, but that of the high-pressed compacts had dropped to only 12.

These examples illustrate the fact that there is no point in raising the pressure to increase the density of products. The final density of a powder metallurgy product is not determined by the pressure under which it is cold-molded.

### Hot Pressing

Within recent years there has been a steady progress in the industry toward hot-pressing of compacts to replace cold-pressing and subsequent sintering. This has come about naturally, as powder metallurgy has turned with increasing vigor to the production of mechanical parts that must have strength and often density, and that must conform to dimensional specifications within close limits. The older process nearly always gave products of a greater or less porosity, and invariably resulted in units with dimensions which differed in the sintered compact from those of the cold-molded unit. As shapes of more complicated form were made it became increasingly difficult to provide dimensions in the cold compact which would finally shrink or grow in all directions during sintering to meet the rigid size specifications. Moreover, it was learned early that products molded at elevated temperatures had greater strength than those which were compressed cold and subsequently sintered.

To a considerable extent these difficulties have been overcome by double-pressing with or without further sintering. The use of sizing dies has been common practice in many factories. After the compact has been sintered it is pressed in a die, sometimes before it has cooled, more often after it has reached room temperature. This process reduces porosity and adds strength. It also forces the unit to meet the proper size specifications. But it does add to the cost of the process, and many laboratories are working on a means to avoid this step by combining pressure with heat.

There are many problems that must be met before a method of

combining heating with pressing can be successfully worked out. Many of the powders used are subject to severe oxidation at elevated temperatures and it is difficult to design equipment which will prevent this.

One manufacturer is successfully producing cemented carbides by this process, however, and there are no doubt many others who are not discussing their methods for publication. The method used to make the cemented carbides—which it is said may be employed equally well to produce excellent iron or steel products of great density, good tensile strength, and a Brinell hardness equivalent to that of a hard steel—is simple in principle, but not so easy to operate.

The powder is compacted under about 20,000 lbs. per sq. in. pressure into a slug with sufficient material to make the finished product and roughly of the size, but not necessarily the shape, required of the commercial unit. This green compact is placed over the die in the bed plate of a press in which it is to be converted to finished form, and insulated from it with a thin sheet of mica. Two electrodes are brought against the sides of the green compact and the current allowed to flow until the temperature is about two-thirds of the melting point of the material in use. It is said that this heating operation takes less than a minute for small units. A variation is to heat with an induction coil. In any case the operation takes place in a vacuum to prevent oxidation. When the green compact has reached the proper temperature, the press ram impresses upon it with the force necessary to form a product of desired qualities, in most cases about 60,000 lbs. per sq. in. The ram forces the compact into the die, carrying a thin sheet of mica with it, which is subsequently removed, as the knockout punch pushes out the finished product. No finishing operation is required. Cemented carbide tool bits made by this process are said to have qualities superior to those made in the usual way.

The wear on the dies is considerable, but not nearly as great as in some of the proposed processes which heat the powder, as such, in a roughly compacted form within the die cavity. The hot compact is in contact with the walls of the die so short a time that they do not heat excessively and means may be provided to cool the dies between pressings. Ways have been found to overcome the excessive wear on electrodes, but these too are a problem not fully solved.

In principle, a process proposed by Jones<sup>26</sup> and worked out in great

<sup>26</sup> W. D. Jones, *Metal Ind.* (London), p. 69, January, 1940.

detail is not greatly different, except that Jones heated the cold-formed compact in a hot die cavity and used the rams as electrodes. He then removed the hot compact to a third die and press for final pressing. He suggested the use of an induction coil to heat complicated shapes. Although he used neither vacuum nor a reducing atmosphere, Jones got very good results from copper-nickel alloys, mixed copper and nickel and mixtures of copper with tin or zinc to form bronze or brass products, pressing at only 10,000 lbs. per sq. in. at 1750° F. He obtained ideal densities and good tensile strength.

Very different is the process employed by General Motors to produce an iron ring from steel scrap, which is described by Macconochie.<sup>27</sup> The shredded scrap is compressed cold to a shape roughly approximating that of the finished article. It is then sintered and as it emerges still hot from the sinter furnace is compressed in a finishing die and press to final form. This might be called hot-forging, or hot-coining.

### Coining

A great deal of experimental work has been reported on both hot-pressing and coining, the term used for pressing after sintering. In general, we may say that both of these processes improve density and strength. As several examples of actual practice with hot-pressing have been mentioned, something further should be said about coining, which has long been common practice but about which little has been published. Hardy and Cremer<sup>28</sup> reported on the effect of repressing electrolytic copper powder. The original compacts were pressed at 60,000 lbs. per sq. in. and sintered at 950° C. in hydrogen for half an hour, after which they were annealed for half an hour at 660° C. At this point they had a tensile strength of 23,000 lbs. per sq. in. When the compacts were repressed at 60,000 lbs. per sq. in. the tensile increased to 27,000 and the elongation from 17 to 25 per cent; repressing at 80,000 lbs. per sq. in. gave a tensile of 29,000 and an elongation of 28 per cent; at a pressure of 100,000 lbs., the tensile reached 30,000 and the elongation 30 per cent.

Such gains in strength are not unusual when repressing is employed, whether on copper, bronze or other metals and alloys, and in addition repressing is an excellent way of controlling dimensions.

<sup>27</sup> A. F. Macconochie, *Steel*, **110**, 98 (1942).

<sup>28</sup> Charles Hardy and George D. Cremer, *Mining and Met.*, p. 509, October, 1942.



## Chapter 7

### Manufacturing Problems and Machines

The manufacture of parts from metal powders presents a number of problems that have not yet been solved, nor is any help to be found in the science of metallurgy or powder metallurgy, as theory has not yet caught up with practice.

#### The American Pattern

In this respect powder metallurgy follows the usual pattern of development in the technical field. A few scientific facts come to the attention of imaginative practical men who see the possibility of basing a method of production upon them. By trial and error they evolve a technique which results in a marketable product. Others follow their lead. More products come on the market. Trial and error methods improve techniques and products, and scientists become interested in studying them and trying to find the underlying principles which cause success or failure. But scientific investigations are slow and tedious; only small kernels of valuable information come from the laboratories. These are avidly seized upon by the manufacturers, who use them to make further progress in processing methods and to evolve new products.

Until this period there is a great deal of duplication in the many rival plants, which keep their knowledge secret. But human nature being what it is, in many cases these secrets are in duplicate. Each company believes that it alone knows the answer to certain problems, but often each has found the same or a very similar answer and deceives itself in believing it to be unique. Gradually this fact comes home to the producers, who reluctantly recognize that an interchange of information involves little sacrifice in exchange for a great gain. More information reaches the researchers, who are quick to use it. Industry becomes aware that its rule-of-thumb progress is slowed by lack of information about basic principles, and subsidizes research programs to investigate the scientific principles underlying practice.

The exchange of information by publications and by conferences increases in tempo. Patents take the place of secret processes. The number of problems given to scientists to solve begins to exceed those studied by the practical trial and error technicians. Companies which have been most successful establish fellowships and research programs to continue to maintain their leadership. Slowly scientific explanations and laws are discovered, with implications too great to be fully absorbed by industry; and it is then that theory forges ahead of practice.

### **Status of Powder Metallurgy**

This is the pattern of American industrial progress. Powder metallurgy has not yet come of age. It is still at that stage when information is being exchanged reluctantly but more rapidly, when scientists are beginning the slow task of determining underlying laws and principles, when patents are replacing secret methods, but where practical experience—the “know-how”—is still well ahead of scientific knowledge.

### **A “Must” in Research**

Together with the immediate problems of making products for the market, any manufacturer who hopes to keep his place in the competitive race must have a basic program of research into the field of hot-pressing. Today the demand from the industry for porous products is being fairly well satisfied. The demand for dense, strong products is only beginning, and a big market will be opened still further with the production of larger, stronger parts which can be made by molding, thus avoiding the need of highly skilled labor, the loss of material in chips and turnings, and the use of complicated metal cutting machines. A great many such parts are already being made, some by the conventional process of molding and sintering, with or without a subsequent hot- or cold-coining, and a few by hot-pressing. But it appears that products which can be made only with difficulty by conventional methods, may be fabricated more easily with this newer technique; however, this is still in an experimental stage. Except for the very few who have solved some of its special problems, hot-pressing must be the basis for a program of research and development; commercial production must follow the beaten path. Enough problems will be found here.

### First Question

When a producer receives a contract for a part, he must first make sure that the contract calls for enough units to justify the economic advantages of the powder metallurgy technique. If but a few units are required, it would probably be more economical to machine them than to mold them. The cost of dies and of designing the proper die is high, and this cost must be charged against the products. Only an order for a considerable quantity of units can so distribute the initial outlay as to warrant acceptance.

### The Powder

Provided the order is large enough to make this method of production economical, the manufacturer must select with care the powder or powders he expects to use. Before any experimental work is begun, he should make certain that he can secure enough of the identical grade of powder upon which he does his experimental work. It is advisable, whenever possible, to purchase and have delivered all the powder required to fulfill the initial order and any contingent orders, and at the same time to contract for future deliveries of exactly the same grade for as long ahead as the need can be foreseen. If purchase and delivery of powder for the first order would tie up too much capital or storage space, the producer should secure as much as he can in advance and contract for the balance under very careful specifications. If, before the order is filled, a substitute powder must be used, trouble invariably arises. It means at the very least changing the pressing cycle and probably the pressure, but sometimes it means redesigning the dies. A few changes of this kind may easily mean the difference between profit and loss on a given contract. *Too much attention cannot be given to making certain that the powder on which the work is begun shall remain standard throughout the order and reorders.*

It is perhaps as well to await the results of the rest of the investigation before studying whatever changes in powder mix may be possible. Enough may be learned from the part of this book devoted to student experiments to show that slight changes in the composition of the powder mix cause considerable difference in the properties of the finished product. In some cases a slight change may make it possible to retain all the physical qualities desired and yet cause sufficient growth or shrinkage to reduce change in die design to a minimum.

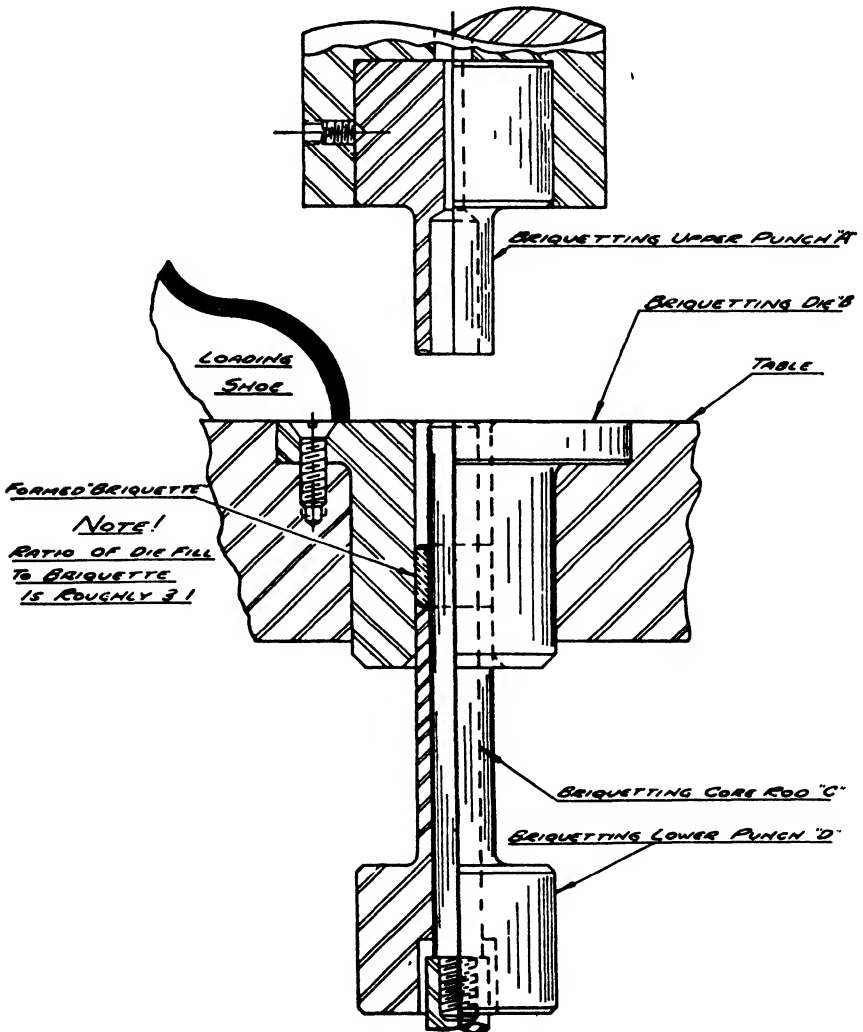
## Dies

Before the commercial die is designed, it is desirable to make an experimental die. This should be made as close to the dimensions of the production die as the knowledge of the manufacturer and die designer permit. In fact it should be identical with the production die, except that it may be made of somewhat cheaper material, since long life is not required of it. If the upper and lower punches are to affect the shape of the upper and lower faces of the product, these must be designed with all the care given the final die. In the die used in the experiments outlined later in the book, the upper punch is used later as a knock-out punch, and the die is designed without taper; but in commercial practice the knock-out punch pushes the pressed compact from the bottom out of the die through the top into which the powder is fed. A slight taper will add to the life of the die and conserve energy. Therefore, whenever possible, dies should be designed to take advantage of this. If a taper is to be employed in commercial production the experimental die should be designed with taper, and a special knock-out punch provided.

Attention to the design of this assembly will save endless difficulty later. Experimental units are to be made in it, and if the designer has brought sufficient knowledge to bear on this preliminary work, the errors in design may be slight enough to correct directly in the finished commercial die. If a complicated shape is to be produced, however, it may be necessary to make two, three, or even more experimental dies before the final one can be designed.

A great many different steels have been used for making dies. Each has its advocates, and only experience can tell which metal will retain its dimensions longest for a particular powder mix or cycle of operation, or which it is wisest to use for a given order. It is a good idea to try to get the material having the longest life, for the difference in cost between a long-lasting die and a poor one is negligible compared with the cost of cutting the cavity or shaping the punches. If the die outlasts the order, that is all to the good, for long after it has been put on the shelf a reorder may bring it into use again. It would be unfortunate if a false economy made its use impossible, for fear that it might become too badly worn before the end of the new order had been completed. A small additional cost for a higher-grade material might have made it out-last its immediate usefulness, and be of future service.

High-carbon, chromium steels are more commonly used for die cavities than most metals; but low-alloy tool steels, high in carbon



*Courtesy Chrysler Corp., Amplex Div., Detroit, Mich.*

Drawing of a Set of Briquetting Tools for Plain Cylindrical Bearings

and tungsten or vanadium are preferred by some manufacturers. The punches should be tough; oil-hardened tool steel is satisfactory for them. Sizing dies are made of hard, long-wearing steels, and occasionally it is advantageous to line them with carbides; but this

is an expensive die not often justified by their cost. Products sized in such dies have an unusually fine finish. But an excellent finish may be obtained by using a hard steel, if the minor detail of the final lap operation is supervised and it is made parallel to the direction of molding and ejection, instead of at right angles to it, which appears to be common practice.

If a hard or face-hardened steel is used for the experimental die it is well to support it with rings of softer metal either shrunk to the external walls or fastened to them as a guide. It is dangerous to use such dies without this support, as they may rupture in the press under operation; and when such a die splits, portions of it fly from the platen with sufficient force to injure severely, if not kill, any one who is struck by them. The same die set in the bed plate of a commercial press is protected by the platen, so that this problem does not arise. But if material of peculiarly low tensile strength, such as a cemented carbide, is used it should always be employed as a liner, for reasons of economy as well as safety.

When the experimental die is ready, the entire cycle of operation should be carefully tested. Units should be pressed under a variety of pressures, closely centered around the pressure which experience suggests as the best one. The wise manufacturer will not confine himself to the pressures in that immediate range, but will make some units well above, and others well below the theoretically correct ones. Sometimes interesting possibilities develop; but in any case the test specimens may be used as samples to put beside the commercial press when the run begins to show the operators what happens if they fail to operate at the pressure required.

### **Presses**

It is more convenient and economical to use a laboratory press than one of the regular presses for this experimental work. Such presses require no time to set up, and their use makes it possible to make samples whenever desirable, without having to wait until one of the regular presses has finished a run, and can be reassembled. There are a number of such laboratory presses on the market, some capable of pressures up to 20,000 lbs. per sq. in., and others of much higher pressures. For small products—those with a total surface area of two-tenths of a square inch—that is to say those with a

diameter of one-half inch, the small press suits the purpose best, since it makes possible a pressure of up to 100,000 lbs. per sq. in. on a unit of this size, operates under easy hand pressure, and can be handled more conveniently than larger presses; but if tests on larger pieces are to be made, a press with greater capacity is required.

It requires a little ingenuity to simulate the action of a floating die, or of pressing from both ends of a die with a laboratory press, but when this is absolutely necessary it can be worked out with the aid of shims and supports. Usually this refinement can be eliminated from the experiments. The operator must make allowances for the sectional variation in density, which will be greater in the test specimens than in the commercial product if pressing of the test specimen is from one end only. The pressure in a commercial press is applied with both an upper and lower ram, with or without a floating die. In most cases the experienced designer can allow for this. If a shape is too complicated to permit interpretation from a sample pressed only with the upper punch, the action of the commercial press must be simulated in the test press.

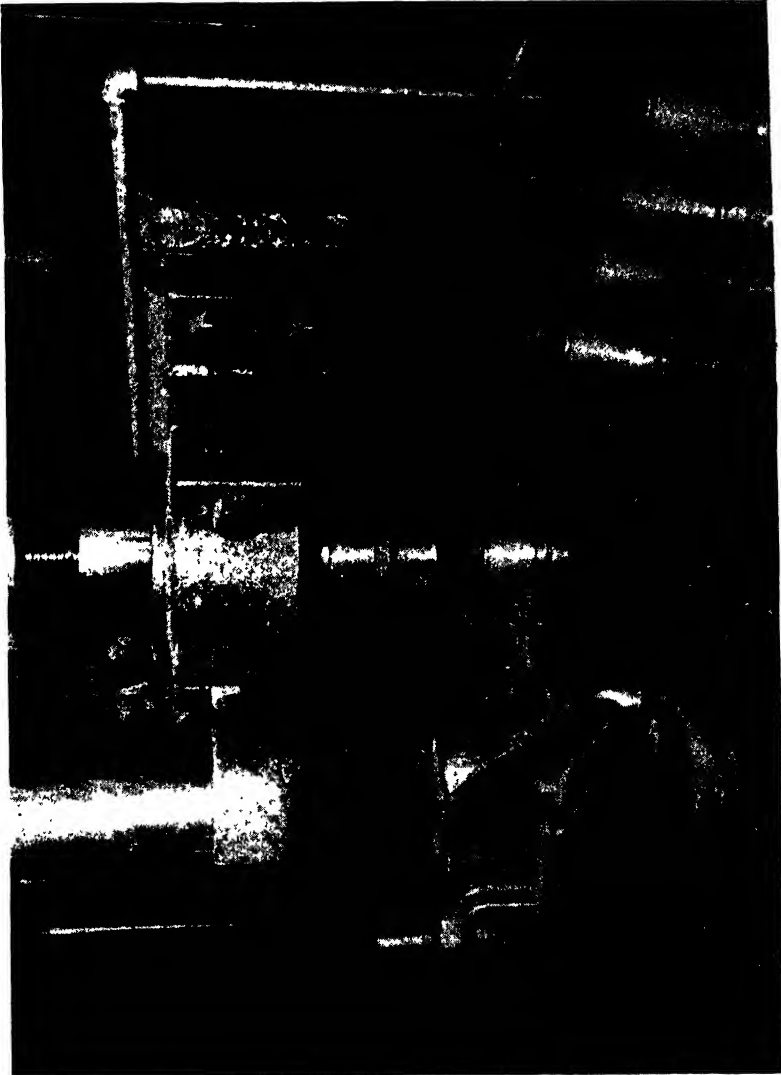
One thing that the test press will not duplicate is the speed of the pressing operation, and final adjustment for optimum speed must be worked out after the commercial cavity has been designed. Speed is not very important if pressure is to be brought to bear with one punch only, but in a floating die, or in a fixed die in which pressure is brought to bear from both the upper and the lower ram, the relative speeds require considerable adjustment.

Commercial pressing is done in a variety of presses, from simple tableting machines to gigantic hydraulics. The presses in use may be mechanical, hydraulic, or a combination. In general, small parts to be made at great speed and at relatively low pressure are best made in mechanical presses; parts which may be molded more slowly, larger parts, and those made at relatively high pressure should be made in hydraulic presses. But such conclusions should be interpreted in only the broadest sense. Mechanical presses make 6-inch parts at a rate of from 300 to 900 an hour operating at about 20,000 lbs. per sq. in. They can produce small bushings as fast as 12,000 an hour, and some small parts have been produced at the rate of over 30,000 per hour. Hardy<sup>29</sup> reports a very small part turned out at the incredible speed of 240,000 units per hour.

<sup>29</sup> Charles Hardy, *Wire and Wire Products*, 17, No. 5, 248 (1942).

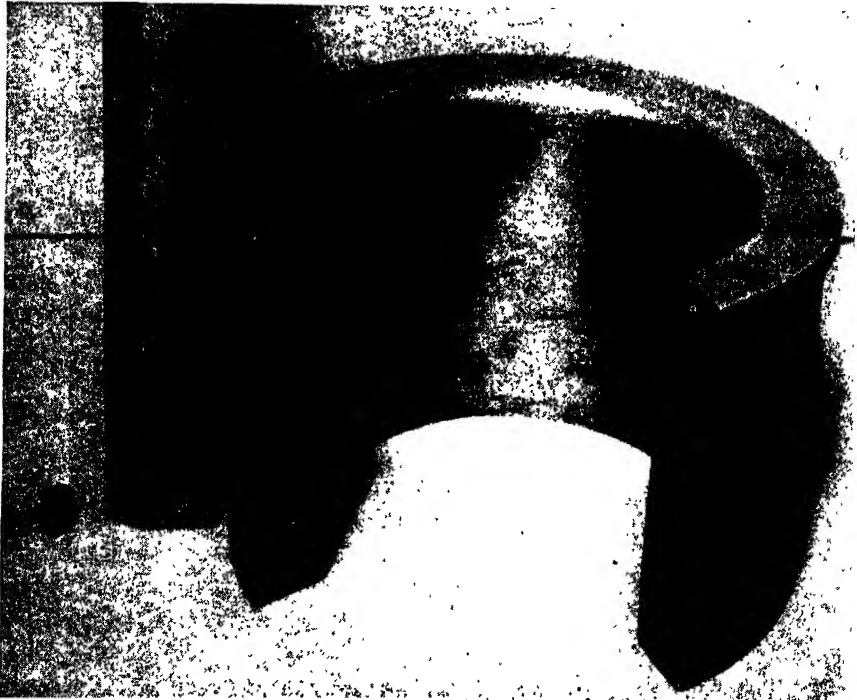
Standard Briquetting  
Equipment

*Courtesy of Chrysler Corp.  
Detroit, Mich.*





Where speed is less important and a part must have perfectly uniform density throughout its depth the hydraulic press is favored. Thus, for shallow core-rings, one manufacturer uses a mechanical press which produces an incredible number of units per hour, but for his long core-rods he employs a hydraulic press, which operates more slowly but turns out units which "track" perfectly. Radio cores are often used in pairs; and since they control inductance



*Courtesy Hard, Metallurgical Co., New York, N. Y.*

A Railroad Bearing Weighing 25 Pounds Made by Powder Metallurgy Technique

by linear movement, the density at any distance from the end must be as close as possible to that at any other point. At high frequency, slight differences are multiplied many times, but when a single core is used, this is readily compensated for. When a pair of cores is used in the same tuning circuit, it is essential that the variation in density from the ideal shall be of the same order and at the same point on each rod; otherwise they fail to "track." To meet this demand for perfect uniformity, the hydraulic press has

been found more satisfactory than any mechanical press yet available.

But a hydraulic press also must be used where really high pressures are required, nor is speed sacrificed to too great an extent. One manufacturer makes a press—his standard 375-ton press—which will produce 12.5-sq. in. parts under a pressure of 60,000 lbs. per sq. in. at the rate of 1200 per hour; greater speeds and higher pressures may be obtained from larger presses.

### Sinter Atmosphere

Having made a number of units under various pressures and measured them to compare their size with the specifications, the next step for the manufacturer is to determine the proper heat cycle. He is probably unable to vary the atmosphere in which this sintering is to take place, since he is already committed to one of the several gases available for the purpose. However, certain gas-producing equipment is adjustable and can be made to afford sintering atmospheres of various compositions.

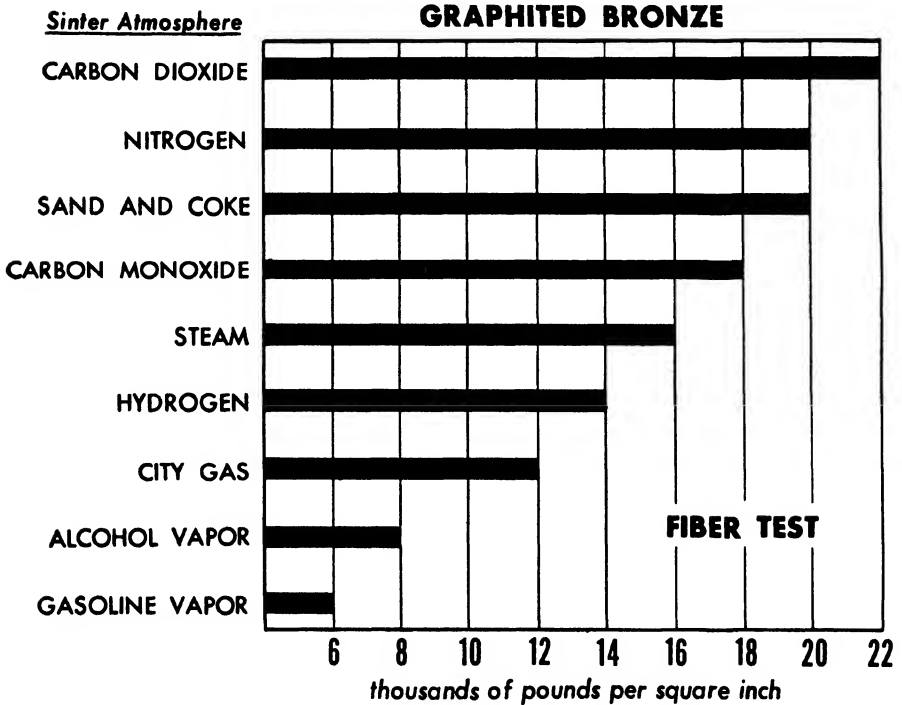
The simplest sintering method seems to be the one used in the experimental work at the end of this book, but it is difficult to adapt such a procedure to commercial practice. A slight variation of this—packing the pressed compacts in a carburizing compound—is feasible and is still practiced in a few instances; but control of such atmosphere is only approximate since, as time passes, the percentage composition of the gas generated by carburizing compounds differs considerable.

City gas, natural gas, hydrogen or carbon dioxide led into the furnace from containers have all been tried; the first three are still employed, though the cost of pure hydrogen is usually so high that its use is uneconomical unless the product is of great commercial value. Properly prepared flue gas may be used, and nitrogen may be employed, but of course the latter has no reducing action upon any oxide which may be present, and which would probably be removed, at least in part, in a reducing atmosphere. Modern gas-producing units are now available and one is well described by Webber<sup>30</sup> which permits control of the gas-air ratio to give a sintering atmosphere of varying composition. A unit may be provided to

<sup>30</sup> H. M. Webber, "Powder Metallurgy Conference Papers", p. 302 edited by John Wulff, A.S.M., 1941.

remove the moisture from the gas. By partial or complete combustion this gas may vary from 4.0 to 11 per cent carbon dioxide and from 17 to 1 per cent hydrogen. The balance of the atmosphere is primarily nitrogen after the water vapor has been removed.

Recently producers who feel that hydrogen is essential for their purpose have favored the use of dissociated ammonia, which is considerably cheaper than hydrogen and often does all that may



### SINTER ATMOSPHERE VS. STRENGTH

be expected from pure hydrogen. Dissociated ammonia produces a mixture of about 75 per cent hydrogen and 25 per cent nitrogen; it is formed by leading ammonia from containers over a heated catalyst. Completely designed units are available for the conversion of ammonia to hydrogen and nitrogen. If an atmosphere of high nitrogen rather than of hydrogen is desired, the hydrogen may be burned and reduced to as low as one-half per cent, calculated after the water vapor has been removed.

The importance of the atmosphere is often overlooked by manufacturers, who seem to feel that any atmosphere free of water vapor and oxygen will yield products of the same physical properties. This is far from being the case. The accompanying graph\* will show the importance of this consideration as it affects strength.

Test units of graphited bronze were prepared and then sintered in the atmosphere under investigation. They were then subjected to a fiber strength test with the results shown. Note that some units were more than three times as strong as others. Even those sintered in hydrogen were more than 35 per cent weaker than those sintered in carbon dioxide.

The significance of this should not be lost upon the student or manufacturer. Often factors which may be considered negligible in common metallurgical practice must be carefully considered when employing powder metallurgy techniques.

Except in rare instances a manufacturer will not change the composition of his atmosphere for the sake of one product. He will only accept orders which he can fill with little variation from the procedure he uses for all the rest of his products; and changing a sintering atmosphere is a major change, which is seldom warranted. But it is not so difficult to change the temperature of the furnaces, nor the time during which parts remain in them. Thus a fabricator has two more variables to investigate before redesigning his die, and fixing on the final cycle of operations. These are time of sintering, and temperature of sintering.

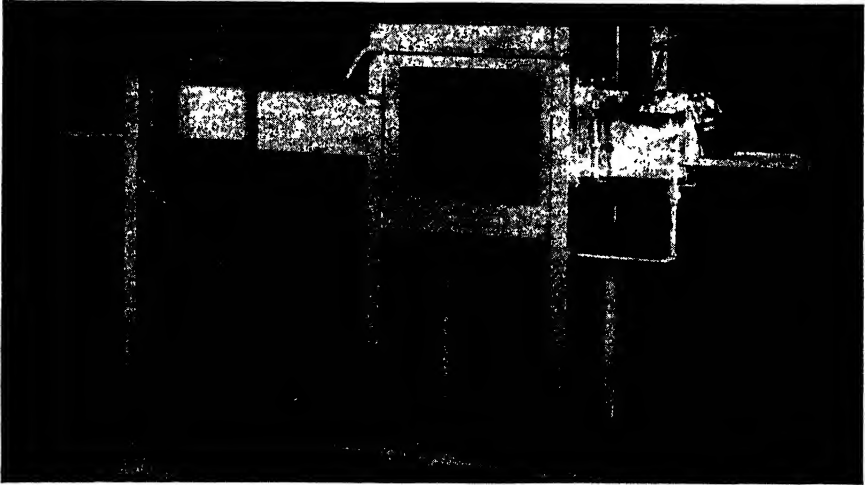
## **Furnace**

If he is making a part it will probably be a bronze, brass or iron one. A study of the conventional curves in Chapter 11 will acquaint him with some of the effects he may expect by varying these factors. A great variety of furnaces is available, and any of these fitted with good controls is suited for the purpose. Electric furnaces are used almost exclusively; these vary from tube furnaces in which a silica or Alundum tube is circled with Nichrome or molybdenum wire, to box furnaces heated by elements in the sides, top and bottom of the walls. A bell-type furnace in which a stack of parts is placed on the floor and pressed from above during sintering has

\* The writer is indebted to H. E. Hall, Metals Disintegrating Co., Inc., for the data on which this graph is based.

been used to weld friction-powdered parts to a steel backing. When this furnace is used, the material is stacked under the press head, and the heating elements in the walls of a bell-shaped cover are lowered over them. It seals to the floor of the furnace and a blower is designed to circulate the atmosphere and make uniform heating possible.

The most modern development in sintering furnaces is the continuous furnace. Broadly, this is of two types. Both are long, box-shaped structures with heating elements in the walls and top. The horizontal box is divided into zones in which the temperature



*Courtesy Huray Metallurgical Co., New York, N. Y.*

A Pilot Plant Sintering Furnace

is separately controlled, so that rapid preheating may take place in the first zone, conditioning in the second, annealing in the third, and in a fourth zone cooling may take the place of heating elements; any zone but the heating and cooling zone may be eliminated. The two types differ in the type of conveyor used for the continuous operation. In one type the work is placed on a conveyor which carries it through the several zones on an endless belt at the desired speed; in the other, the flooring is a series of rollers, and the work is pushed from one end to the other on a flooring which does not move along with it, but consists of a series of touching rollers. The rollers are operated by external means, which roll the work along.

So many factors enter into the design of a furnace that it is difficult to decide its dimensions unless all factors are known. A typical furnace for iron compacts operates at about 2000° F., and has a heating zone 10 feet long and a cooling zone about 30 feet long.



*Courtesy of Chrysler Corp., Detroit, Mich.*

Heat-Treating Furnace

The writer favors the web belt hearth rather than the roller, but the wear on the web—which is continually heated and cooled—is high, and the web is not well suited to heavy work. On the other hand, the difficulty of sealing a roller type hearth in which each roller bearing must be lubricated and insulated from the furnace heat presents obvious difficulties; and if anything happens to a roller

or its bearing the furnace must be cooled and taken down before a repair can be made. A belt conveyor can be completely replaced without stopping operations.

### **Heat Control**

The temperature at which the furnace is to operate must be decided largely by the material to be sintered. The important thing is that the heating be uniform throughout the mass of the product, that it be reached quickly, and be maintained for the least time necessary to obtain a product with the properties desired. The effects of temperature and time are more fully discussed elsewhere in this book. Once these factors have been decided upon, too much emphasis cannot be placed on the importance of adhering to the schedule. This is more true of temperature than of time, but no carelessness may be permitted in regard to either factor. Good control instruments on the furnace are essential if uniform results are desired. Care should be exercised to cut down the lag between the cut-off-and-on of current as far as possible. Too often control instruments which cut the current when a certain temperature is reached do not become effective upon the temperature until it has exceeded the cut-off temperature by as much as 100 degrees; similarly, though the current begins operating when the temperature drops to a given point, the furnace may cool many degrees more before the heating becomes effective.

### **Final Steps**

When the operator has set his heat cycle he may proceed to test his product at once, or may decide to put it through a sizing die first. Enough has been written about dies, so that nothing further need be added here. Study of the product at this point reveals how close to specifications the manufacturer has approached. He may find it necessary to make several adjustments in each step of production, or he may find that simply changing some minor dimension of his die will give him what he wants. But whatever the case, this is the time for him to make his changes and prove them; he cannot afford to wait until he begins to fill the order. Before beginning commercial operation he should produce a number of units following the exact cycle he has set to make absolutely certain that it is exactly correct.

## Chapter 8

### The College Course

Without a full knowledge of the circumstances, the facilities of the school, and the previous education of the students, it is impossible to lay out a detailed course of instruction in powder metallurgy. However, a few basic principles have been evolved, and a typical working outline has been planned and tested by the experience of several semesters and finally put into a form which has met with the approval of a great many serious students.

First of all it must be recognized that powder metallurgy is not in itself a science or a skill, but is rather a system of techniques based on the sciences of chemistry, metallurgy and physics and on the application of mechanics, laboratory and plant practices. The student coming to the course should be equipped with a knowledge of these sciences and with training in these skills.

#### Objectives

One cannot hope to create any powder metallurgists qualified to become consultants and guides to the development of the industry by means of any single series of lectures and laboratory experiments. The objective of any course of instruction must necessarily be considerably more limited. The student who has successfully completed a course in powder metallurgy should be skilled in the techniques of evaluating the raw material employed in this field—powdered metals—and in those of evaluating the products of powder metallurgy. He should be familiar with and have at least laboratory experience in methods of producing various types of metal powders, as well as familiarity with the processing of typical metallurgical products.

Tentatively, a few general rules expressing the relationship between the forces involved should be thoroughly known to the student. The relationship between the quantity of graphite in a powder mixture and the strength, per cent elongation and hardness



of the product, for example; the effect of varying the pressure of formation while making a green compact, as it relates to the physical properties of the finished product; the effect of varying the time of heating or the temperature of sintering upon the physical properties—all these should be part of the knowledge of any student who has finished the course.

In view of the fact that these rules of relationship are held only tentatively by experts in the field, the student must be impressed with the importance of using research methods to check any proposed plan of operation, and he must know that he cannot rely upon these rules as one can upon a well authenticated theory. Therefore he must be trained in short-cut methods of research as applied to this particular field.

### **Basis of Instruction**

The mind of man is so constituted that it reasons from observation. Observation should precede general deductions. In planning the course it is well to give the student an opportunity to make his own generalizations and lay down tentative rules of relationship rather than to offer the student generalizations which he may later try to check in the laboratory. All modern pedagogues agree that one learns best by actively experiencing, and it is a grave question if any scientific knowledge can become a permanent part of the equipment of the student if gained through mere hearsay, the lecture, the printed page or demonstration, regardless of the skill of the lecturer or of his authority.

The science of chemistry has long recognized this, and a chemistry course that is not at least in part a laboratory course is an anomaly rarely found in any first-class educational institution. On the other hand, so long has it become an accepted practice to combine laboratory work with lectures when teaching the sciences, that too often the laboratory merely supplements the lectures instead of integrating them to produce a single total effect. This course attempts such integration.

### **Demand for Course**

A recent survey of 90 firms engaged in the powder metallurgy industry showed that approximately 20 per cent thought that the colleges would do an essential service to the industry if they gave

courses in powder metallurgy. About 5 per cent felt that enough men were available so that training of new men in this field was unnecessary. Another 5 per cent thought that, though it was highly desirable, no adequate training could be provided to fit the peculiar requirements of powder metallurgy; and the balance of 70 per cent agreed that such courses were highly desirable. There may be short periods in the near future when there are more men trained in this technique than the industry can immediately absorb; but powder metallurgists of experience are convinced that the field is expanding at a very rapid rate. There can be little doubt that it will absorb many more men than are now equipped to serve in it.

Since it is difficult to plan for other schools and other conditions, the program outlined here is offered as a well proven plan of instruction, which is sufficiently basic that it may be adapted to meet the limitations of most institutions, and of course it may be expanded to take advantage of greater facilities than those available where this course was tested.

### Student Qualifications

First we must determine what student is properly prepared to take this course. What previous education should he have had? Our first semester was limited to students who already had a chemical engineering degree, and those were favored for acceptance whose previous training had included some specialization in metallurgy, or whose industrial experience had been in powder metallurgy or a closely related field. As courses were repeated, these strict requirements were somewhat liberalized. Men who had not acquired a chemical engineering degree were allowed to substitute for it considerable experience in the industry or a related industry; and we have found that, where such experience was preceded by an education equivalent to a bachelor's degree in the physical sciences, the students did quite as well as chemical engineering graduates without such experience. On the other hand, it became clear that men with less scientific background than this, regardless of how experienced they were in one or more phases of the powder metallurgy industry, invariably found it extremely difficult to keep up with the class, and none of them ever finished among the upper 20 per cent.

With the single exception of the application of Stokes' Law,

there appears to be no need for any great facility in higher mathematics, but to do well in the course, before entering one should have had a thorough grounding in simpler mathematics, be familiar with graphic presentations, and able to write a concise, clear statement of proceedings and conclusions.

It would seem better, therefore, to establish the general rule that a chemical engineering degree is a prerequisite for those not already engaged in the industry, and that the requirements be liberalized to make acceptable lesser educational attainments for those already engaged in the industry in a research, development, or executive capacity.

### **Number of Students**

Having decided on the qualification of the students, the question arises of how many students should be in a single class. It has been our practice to accept 16 students and divide them into four squads. Experience has taught that more than 16 students cannot be properly supervised and will not get the full benefit of instruction. Four squads seems to be the optimum number that can be efficiently handled. Four men to a squad are not absolutely necessary. Three men could probably perform the squad assignment in the same laboratory periods and indeed would get more experience than the larger number; but two in a squad are too few to handle the assignments properly.

### **Laboratory Schedule**

It is poor economy to assign the same experiment to all students in the same period. This would require a duplication of equipment that cannot be justified, certainly not under war conditions. With considerably less equipment, staggering the assignments achieves the same purpose and has the added advantage that after the first few sessions the official laboratory instruction is supplemented by the instruction of fellow students, so that a squad which has not yet performed a given operation gains assistance from members of those squads which have performed it, and in turn gives instruction in experiments it has performed, but which have not yet been performed by their fellow students. This has a two-fold educational advantage. Direct instruction of student by student is often more effective than that by a teacher and is much more detailed than a

teacher can possibly supply. In addition to this, the student who takes on the role of instructor gains a greater knowledge of the subject he is teaching than he received from merely performing the experiment. Very often, in attempting to pass on what he has recently learned, he uncovers gaps in his own knowledge which he was unaware of; and of course the official instructor is there to fill these gaps, to the advantage of both students.

Some of the experiments are necessarily unrelated, each squad performing the same experiment under identical conditions when it is assigned; but as far as possible the experiments have been planned to supplement each other and, when the results are all in, to supply sufficient data to develop one of the rules or theories of basic significance. In other words, the class as a whole is made into a research staff which proceeds to investigate the simple relationships which may be formulated in the field of powder metallurgy.

For example, instead of having each squad mold a compact under a pressure of 40,000 lbs. per sq. in., and then study its physical properties with a view to comparing the care exercised in the procedure and the general accuracy of their laboratory technique, it is assumed that qualified students are reasonably accurate in their laboratory technique, and that the purpose of the pressure experiment is to determine the relation of the pressure employed in forming the green compact to the physical qualities of the finished product. Thus, Squad A will make compacts under 20,000 and 100,000 lbs., Squad B under 30,000 and 80,000 lbs., and Squads C and D under other pressures. When the results are correlated we shall have discovered by a research procedure the effect of varying the pressure from 20,000 to 100,000 lbs., and the class will itself have worked out the rules of relationship between pressure and physical properties.

Experienced teachers, fully aware of the fact that a post-graduate degree is no guaranty of efficient laboratory technique, may feel that results based on a single experiment by inexperienced students cannot be correlated so that a rule or theory can be evolved from such questionable data. There is some justification for this criticism, and it holds true particularly in the first few laboratory sessions. However, the knowledge that each squad is an important factor in the work of the whole class, and that only by its own careful work can the work of all groups have any value, acts as a tremendous

incentive to careful work. Indeed, we have found that, once the students have direct experience with the equipment they will be using throughout the term, they rapidly learn to do their work with the care and precision with which their undergraduate training should have equipped them. Notice too, that with four squads, each working with a variable at two or more points, the correlated results consist of 8 to 12 points covering a fair range of the variable. When we plot these points, although a few may be in error and fall out of the normal line of the curve, it becomes quite evident which they are. Either the work is so haphazard that no general curve can be drawn, or there is no general curve. We have of course, for background to check the "research" work of the class, the published work of a number of researchers, and it is rare indeed that the students do not produce curves which, in spite of some points obviously out of line, closely parallel the work of the authorities. So far as is possible, the lectures are timed to reach a discussion of the factors examined by the students in the laboratory at about the time the last squad has finished the experiment on this factor. In this way the lectures and the laboratory work are well integrated.

Considering now a class consisting of four squads, the point of immediate consideration is the minimum equipment required in the laboratory.

### Laboratory Equipment

Except for a few special pieces of apparatus, a good general chemical, physical, or mechanical laboratory is well equipped to offer the facilities needed for the experimental work in a powder metallurgy course. On the other hand, few of the standard apparatus or chemicals are required. In their place an assortment of powdered metals must be on hand in sufficient quantity to perform the necessary experiments.

Assuming that four squads will work in a 3-hour laboratory period, the minimum equipment desired will be outlined.

Sufficient tabletop space should be provided to permit about 6 ft. of table frontage per squad. This space should be cleared of all fixed apparatus. There should be provided a table with a heat-resistant top approximately  $2\frac{1}{2}$  feet by  $1\frac{1}{2}$  feet, close to wherever the sintering furnaces are established. In addition, there must be space for one analytical balance, two bulk balances and two pan balances.

These should be on a table sufficiently stable to resist vibration, and far enough from the other laboratory activities to guard them against shock. A space of not less than 12 inches should be provided at each side and in front of each balance so that students may work at them with equipment, notebook and weights free of interference.

In addition table-top space will be required for setting up a certain amount of other apparatus which will be discussed as we come to it. In all, however, in addition to the above, there is required table-top space of about 100 sq. ft. for permanently located equipment. Each squad should be provided with a locker and there must be storage space for metals and chemicals used by all students. A dark room for microscope work is highly desirable, but if an efficient lighting arrangement can be provided, this is not essential. One good microscope with a Euscope attachment and an automatic arc light is necessary, with a system which will enlarge about 500 times and a micrometer slide to standardize the enlargement.

A ball mill, with driving mechanism, is required for experiment 1. Manufacturers will find it useful for mixing powders, reducing their size, and recovering raw material from faulty green products.

For testing hardness, one Rockwell tester is necessary, and we have found that equipping it with a  $\frac{1}{4}$ " ball and a 60-kilo weight will give tests which can be read on the B scale to cover the hardness found in most of the laboratory products. Two hydraulic presses should be provided, each with a low-reading pressure gauge from 0 to 6,000 lbs., and a high-reading gauge with a range up to 20,000 lbs. per square inch. Not less than two high-temperature furnaces with an opening approximately  $7\frac{1}{2}$  in. wide and a height of  $5\frac{1}{2}$  in. are essential, and it is well to have a third oven with an opening approximately 4 in. square. These ovens should be able to maintain a temperature of 2,000° F. and should be equipped with thermometers of the thermocouple type. One small constant-temperature drying oven, capable of being maintained within a few degrees of 110° C. is necessary. In addition, each squad should be furnished with a sheet of oilcloth 3 feet square, a thick glass plate about 14 in. square, a small spatula with a steel blade approximately  $2\frac{1}{2}$  in. long, and a large spatula with a steel blade 8 to 10 inches long. Two micrometers are also necessary, one reading from 0 to 1", and one from 0 to 2".

A cylindrical combustion furnace with a supply of hydrogen and a silica tube, together with the usual equipment of a reduction train is required for the reduction experiment. Half a dozen small specimen wide-mouthed bottles (approximately 6 ounces) with corks are necessary and in the general stock to be issued as required, there should be a reserve supply of assorted wide-mouthed bottles ranging from this size to  $\frac{1}{2}$ -gallon bottles.

Each squad should be provided with a standard 25-ml. flask.

Each squad should have six small metal boxes for sintering. These should be  $2\frac{3}{4}$  in. square and  $1\frac{1}{4}$  in. high, each with a well fitted cover. Each squad should have one die for making compacts; a description of this will follow later.

In addition to two large desiccators and a small one, tongs and asbestos gloves, labels, corks and bottles, the general supply should have special copper boats used for the reduction experiment, tared watch glasses, microscope slides and microscope cleaning paper and such general supplies as are necessary to keep a laboratory in good order.

The break tester may be considered part of the hydraulic press equipment, and one should be provided for each hydraulic press.

A special sedimentation apparatus has been worked out for the determination of particle size and this should be set up permanently.

Two flow-test apparatus should be permanently set up and each one provided with a stop-watch and a 100-ml. standard flask.

We have found that a press such as the Carver Laboratory Press or the Hydraulic Press Manufacturing Company's laboratory press is heavy enough to do all the experimental work quite satisfactorily.

### Laboratory Dies

The dies for this course require some detailed description. A 3-in. cylinder ( $\frac{1}{2}$  in. I.D., 2 in. O.D.) is provided to receive the powder mixture. For the bottom of the die a disk  $\frac{1}{2}$  in. thick is provided with a rod  $\frac{3}{8}$  in. high which exactly fits into the base of the die cylinder. To compact the powder, a  $3\frac{1}{2}$ -in. rod which fits the hole with a clearance of less than 0.0001 in. is backed by a disk approximately equal to the outside diameter of the die cylinder. For each die a horseshoe-shaped arbor not less than  $1\frac{1}{2}$  in. high should be provided, on which the die cylinder may rest, its opening clear of the inside walls of the die. In operation, the die cylinder is placed on the supporting base and the projecting  $\frac{3}{8}$ -in. rod closes

the opening tightly. It is then loosely filled with the powder mix to be formed into a compact and the compression rod carefully inserted by hand. The pressure of the hand is sufficient to compact the powder slightly and permit the plunger to enter far enough to support itself. Thus assembled, the die is placed between the plates of the hydraulic press, which are brought together until the gauge indicates that the required pressure has been reached. The pressure is now released, the plates parted and the base of the die removed. In its place is put the arbor. The plates of the press are again brought together and the upper plate exerts pressure upon the plunger. This now acts as an ejector, since there is nothing under the hole in the die. The formed compact is pushed out between the arms of the arbor and is ready for subsequent examination and treatment. The plunger is removed, the arbor replaced with the base of the die, and the die is ready to be loaded with powder for the formation of another compact. (See Frontispiece.)

Experience has shown that dies with an outside diameter of as great as 3 in. may break under high compression loads. To avoid the danger which arises from the rupture of a die, we have found it desirable to shrink supporting rings around the cylinder. These take the place of the bed plate which customarily protects a die on a commercial press. The cost of making dies of this depth to the precision required is very great, and we have been able to substitute bushings such as those made by F. A. Baumbach Company and the Danly Machine Company, both of Chicago. These are guaranteed to  $\pm 0.0001$  in. variation from 0.5 in. I.D. These companies also manufacture dowel pins which can be attached to a disc to make the compression element, and such rods may also be used in connection with a disc to form the base of the die. They are guaranteed to  $\pm 0.0002$  in. They should be purchased over-size and lapped to fit the die compartment with a minimum of clearance. While a die of the kind described will cost from \$80 to \$125, the assembled unit made from the standard parts suggested can be purchased for less than \$5.00 and after the necessary work, including lapping, has been finished, the cost should still be very moderate.

### **Break Tester**

The break tester is a piece of equipment which we have designed to fit our special needs and which has proved entirely satisfactory. In testing the strength of powder mixtures it has been the custom



in the trade to make 1-in. bushings and subject them to direct pressure between the plates of a hydraulic press. At the point where the nipple fails, the pressure is read and reported as compression strength. In order to compare the strength of the products made in the laboratory course directly with figures obtainable from other sources, it would be necessary to make such bushings.

Besides the cost of the dies required, a number of other objections arise. Measurement and calculation required to determine change in volume would be much more complicated. A specially designed arbor would be required in order to make Rockwell tests, and a greater furnace capacity would be needed to sinter these larger test units.

The core made by the die described above is a simple cylinder  $\frac{1}{2}$  in. in diameter and from  $\frac{3}{4}$  to  $1\frac{1}{2}$  in. long; only two micrometer measurements and very simple arithmetic is required to determine its volume. It occupies little space and a great number of cores may be sintered simultaneously. It fits into the V-arbor of the Rockwell tester. Against this is the fact that data on this type of product are not available. It is very necessary however, that the student be acquainted with the "strength" of the products he makes. Of course the ideal test for strength would be the determination of tensile strength; but before this can be measured, we must have a product of such shape and size that an extremely complicated die would be necessary, or a larger product than the core made, and subsequently machined to a standard size and shape. For all these reasons we have measured strength in terms of the break test—a purely arbitrary test which, however, we find gives consistent results and which may be used as an empirical measure of strength.

The break tester has been designed and redesigned to meet various objections. In principle it consists of a broad knife-edge which presses upon the supported core. Pressure is steadily increased upon the knife-edge until it reaches such a point that the supported core fails. This pressure is read from the gauge and reported as break strength.

We are aware that the figure represents a combination of sheer and fiber tests; but its simplicity, the ease with which it may be applied and the consistency with which duplicate results may be obtained, make it admirably suited to the purpose of the course.

It is made clear to the student that he is reporting only a number, not pounds per square inch, and no confusion of terms arises.

The apparatus which has finally become standard for our courses consists of a base into which a slot 0.1270 in. wide at the top, 0.132 in. at its base and 0.5625 in. deep has been cut. This slot extends throughout the width of the base which is 2 in. square. The core rests on this slot. The knife-edge 0.1250 in. thick, also extending the length of the base, is 0.5 in. high, supported in a block of metal 0.5 in. thick, upon which the upper plate of the hydraulic press impinges. The knife-edge is carefully centered so that the blade always descends directly over the slot. Two rigid guide plates 0.375 in. thick are attached to the upper plate and extend beyond the top of the base element when the knife-edge rests lightly on the supported core. We have made them 1.438 in. deep, measured from the base of the upper plate. To add rigidity to the assembly to give certain guidance to the knife-edge, and to hold the test piece in place until the knife-edge grips it, the corners of the base extend like four cubical columns above the top of the slot for 0.5 in. These columns are 0.3125 in. square. (See Frontispiece)

### Flow Tester

Flow tests of various kinds have been designed by some manufacturers for their own use. They usually consist of a funnel and a vibratory system. The powder is placed in the funnel, the vibrator started, the mouth of the funnel is opened, and the time required for the load in the funnel to discharge itself is determined by means of a stop-watch. Such a system has many advantages; for example, with it positive figures may be obtained on the poorest-flowing powders. Unfortunately, the equipment has not been standardized; and although the principle is employed by a number of laboratories, unless these laboratories have consulted, their flow figures are unrelated. Any system which will measure the flow or, conversely, the bridging and lack of flow of a powder, is satisfactory provided the flow figures are directly related to other flow figures determined on the same equipment.

In the interests of economy, the system adopted in our course is extremely simple. A standard 60° 4 in. funnel is sawed off  $\frac{1}{8}$  in. from the point where the stem begins. A 6 in. wooden plug is provided to close the opening. The funnel is supported by a ring

stand at a height sufficient for the opening of the funnel to be inserted into the mouth of a 100-ml. standard volumetric flask. To determine the flow of a metal powder, 200 cc. of the powder are poured into the closed funnel. The plug is removed and the time required to fill the flask to the meniscus is measured on a stop-watch.

This is entirely satisfactory for powder which has either fair or excellent flow. Time covers a range of from 4 to 18 seconds. It is not satisfactory for powders with poor or bad flow, as long before the 100-cc. mark has been reached, the flow ceases entirely. In fact, in the case of some powders of very bad flow, when the plug in the funnel is removed, after the first few grains of powder fall no further powder will pass through the opening. Therefore, it has been necessary in some cases to report simply "no flow" for a number of powders which the student encounters. A vibratory system would give some figure on most, if not all, of these powders.

### **Sedimentation Apparatus**

For the particle size determination by the sedimentation method adapted to this course, only minor pieces of special equipment are required. The sedimentation method is more flexible than others, and a technique can be quickly developed which will produce results easily checked by other operators. The first cost of installing the necessary materials is much lower than the purchase price of a Roller air separator or a Wagner Turbidimeter. But the difficulty of operating the so-called pipette method is very great when heavy metal powders are tested. This method is entirely satisfactory with such materials as clays and lightweight powders, but when metal powders are subjected to the test, the operator has great difficulty in removing the settled portion of the powder to the filter. Only after repeated washings, each taking considerable time, is it possible to clean thoroughly the very long test tubes which are required by the method. This repeated washing multiplies the possibility and the extent of the errors which may be introduced in transferring a solid from one vessel to another, and the shape of this particular vessel demands a special delicacy of technique. In order to cut down these errors, and primarily to increase the speed of the test, we have designed a simple system which has proved very satisfactory. As the test is customarily run, a weighed quantity of powder is uniformly suspended in a liquid medium (we employ

water) and the suspension is allowed to settle through a 20-cm. fall. At various intervals of time, what remains in suspension above the lower mark of the 20-cm. drop is removed and the quantity of material which has not settled in the time interval is removed to a filter paper and weighed.

In our system we weigh the particles which still remain in suspension after various intervals of time, and we therefore filter the column of liquid in which the unsettled particles remain above the lower mark of the sedimentation chamber. Thus there is no need to remove the residue from the long, narrow sedimentation vessel. To accomplish rapid and efficient removal of the suspension, we use a vacuum which pulls the liquid in the sedimentation chamber into a separatory tube equipped with a stop-cock. The separatory tube is, of course, of sufficient capacity to take the entire column of suspension, and is long enough so that any splash which occurs cannot reach the closure. We use a tube 11 in. long from top to center of stop-cock. The I.D. at the top is  $1\frac{1}{4}$  in.; the walls are thick enough to withstand vacuum. About 10 in. from the top the sides taper to the stop-cock, which has a  $\frac{1}{8}$ -in. bore and exits through a stem 2 in. long whose I.D. is  $\frac{1}{4}$  in.

A U-tube is designed with the short end bent to impinge the incoming liquid just below the stopper against the side of the separatory tube, and continues through the two-holed stopper in a wide bend. The long end of the U-tube is long enough to reach the bottom of the suspension vessel easily ( $13\frac{1}{2}$  in.). It is made of  $\frac{1}{4}$ -in. tubing drawn to a  $\frac{1}{8}$ -in. opening at the tip. Through the other hole of the stopper a right-angled tube connects to the source of vacuum. The end entering the funnel is cut off short just below the stopper so that it cannot draw any of the incoming liquid out of the funnel, and a water aspirator serves as a satisfactory pump for the source of vacuum. A good light source is required, which should illuminate through the suspension the lower mark of the 20-cm. column. (See Frontispiece.)

In operation, the two-hole stopper is inserted into the top of the funnel. The stop-cock is closed, and the vacuum turned on. When the proper time interval has elapsed, the operator plunges the long arm of the U-tube rapidly to within  $\frac{1}{2}$ -in. of the lower mark of the settling column and then lowers it with care to the mark. The vacuum remains on and the long tube may be washed by permitting

it to draw up 4 or 5 cc. of wash water in three or four successive operations. All the suspended particles have now been removed to the funnel, with the exception of a very small quantity which adheres to the sides of the suspension vessel. This error, which is common both to this method and the usual pipette method, cannot be eliminated; but experience shows that it is negligible and well within the limits of error inherent in the mathematical treatment required by this method of analysis. When all particles have been removed to the separatory tube and the U-tube washed, the two-hole stopper is removed from the top and the tube placed in a jaw-clamp above a filter paper. The stop-cock is opened to feed the filter. A rapid rinse with a wash bottle cleans down the sides of the tube and the transfer of the heavy particles is easily effected.

With most metals it is unnecessary to use any scrubbing technique, but if great accuracy is required and the particles have a tendency to cling to the walls, a long rubber-tipped scrubber may be employed efficiently. Any method for determining the weight of the suspended particles may be employed; but for the purposes of the course we find it convenient to select a metal to test which does not readily oxidize and which therefore may be weighed directly as pure metal. If it is desired to test a readily oxidized metal, it will be necessary to convert to the oxide or the sulfate by the usual methods of burning and then calculate the metal from the incinerated residue.

We have found it convenient and sufficiently accurate for our purposes to balance all the filter papers employed, usually six, against a single paper which becomes the tare. We subject the tare to as much washing as the volume of water which passes through the test papers, and we dry all test papers at the same time as we do the tare. In this way any change in weight which may occur in the filter papers which hold the metal particles occurs simultaneously in the tare paper. In order then to determine what weight of particles have been caught in the filter paper, it is only necessary to place the tare on the weight side of the balance before weighing the test papers containing the powder. The weight found tells directly the weight of the particles and no correction is necessary for the filter paper.

Undoubtedly this is not a precise weight, but since time is a factor, and since we cannot hope to develop a perfect technique in a

single experiment, we have found that sufficiently accurate results are obtained by weighing exactly to but 3 decimal places. The several errors inherent in this technique will not appear in the third decimal place, and are very much smaller than the inherent error introduced by the assumption that the particles under examination are spheres. We therefore feel justified in ignoring the errors of technique.

In addition then to the special separatory tube and connections, the sedimentation method requires, as is clearly suggested in this discussion, a test-tube rack, which will hold six test tubes 1-in. in diameter, each with a capacity of about 120 ml. These tubes should be marked at a point where the volume is 20 ml. and at another point exactly 20 centimeters above the first mark. In this way we provide a 20-centimeter fall for the particles into a space sufficient to hold 20 ml. of volume.

In addition to the suspension vessels and the U-tube assembly, four of the specially designed separatory funnels with proper clamps to hold them are required, as well as beakers to catch the filtrate, and a wash bottle. Two stop watches should be sufficient for the necessary timing of the six suspension vessels. Four of the separatory tubes should be enough, since the early suspensions can be filtered and the tubes ready for use again long before the 30 and 60-min. tests are ready for siphoning. A small pair of sharp scissors is necessary for taring the filter papers. It will facilitate the technique if the aspirator is connected to the siphoning apparatus by not less than four feet of heavy-walled tubing. Seven 50-cc. beakers should be marked 1 to 6 and T, respectively, so that the filters with their filtrate may be dried simultaneously with the tared filter in a drying oven at 105° C., and a desiccator filled with calcium chloride should be provided, of sufficient diameter to take all these beakers for cooling.

This completes the equipment required for the sedimentation test.

### **General Instructions for Making Cores**

It is advisable to state some general instructions covering the cycle of production of the cores which are so much a part of the course. The core is the representative product made by the student, and in making and studying it he learns all that he can learn about

the production of metal parts from powders. Difficulties which naturally arise in making more complicated shapes than the simple core can be discussed intelligently after the student has himself made enough simple cylinders to learn something of the peculiarities which arise in their production. Because of the relatively low temperature required and the ease of securing a uniform powder, the bronze core forms the basis of core experiments. However, so that some comparison may be drawn, we have introduced a few other experiments which have as their end product a brass core, an aluminum core, iron and iron combination cores, and occasionally others. Under special conditions an instructor may decide that for the particular field on which he wishes to place the emphasis of his course, cores of some other type should receive the major emphasis.

Of special interest to instructors should be the fact that the results of the work of one class can be sharply varied from that of another class by changing the general instructions for one simple point, or by changing the raw material, using in one class a different particle size distribution from that used in another. The most markedly different results may also be obtained by changing the percentage of graphite, either eliminating it entirely or increasing it beyond the standard suggested in this text. Somewhat different results may also be obtained by changing the heat cycle. We have found it desirable to bring about such different results by standardizing the cycle in one case to a straight single heat at 1500° F., while in another case we heated at 1500° F. for 15 minutes and then introduced a soaking period of 60 minutes at 900° F. Another variation is to preheat at 900° F. for from 15 to 30 minutes and then sinter at 1500° F. for 15 minutes, following this with a soaking period or not, as desired. Other variations will suggest themselves to the instructor.

The simplest cycle and method of standardization is to employ a copper powder 100 per cent through 100 mesh, 50 per cent through 325, a tin powder 100 per cent through 100 mesh and 70 per cent through 325, in a proportion of 10 tin to 90 copper. To this for the standard should be added 2 per cent of graphite.

### **Pressure**

The standard pressure of 40,000 lbs. per sq. in. is a desirable one, since it permits considerable variation all well within the range of a

good laboratory press. The effect of varying the pressure is a valuable and highly instructive experiment. Since the die is  $\frac{1}{2}$ -in. in diameter, it presents an area of 0.1964 sq. in. For practical purposes this may be treated as 1/5th of a sq. in. of surface, so that a dial pressure of 8,000 lbs. is equivalent to 40,000 lbs. per sq. in. The standard laboratory press dial pressure ranges from 0 to 20,000 lbs., and these cores may be made on it under a pressure up to 100,000 lbs. per sq. in.

### Heat Treatment

In a three-hour laboratory period the length of time required for an experiment is a limiting factor. A 15-minute heat treatment at 1500° F. permits maximum use of the time available. It should be pointed out to the student that one cannot begin to time the heat treatment until the cores have actually reached the sintering temperature. For a particular furnace, the load of a sintering box and other factors, this period may vary slightly in each experiment. The time required to bring the temperature of the contents to sinter point must be determined experimentally. This can readily be done by inserting a thermocouple into a properly charged sinter box and timing the period required for the temperature within the box to reach the temperature of the furnace as read by a furnace thermocouple.

Using a box of the dimensions we employ and charging this with 5 to 10 cores and charcoal in an oven set 100° F. above the temperature at which it is desired to sinter, we have found that it takes  $\pm 20$  minutes for the charge to reach the desired temperature. The temperature of the furnace naturally will drop when it is loaded. This is partly caused by the opening of the door and partly by the cold load which has been placed on the floor of the furnace. For this reason, whenever possible we keep the empty sintering furnace about 100° F. above the desired temperature of 1500° F. If many boxes of cores are charged at the same time, the temperature may actually drop to as low as 1100 or 1200° F.; but well before the 20-minute period has elapsed, the temperature will have evened out at 1500° F. and we find that the interior of the box still reaches the proper temperature within a few minutes of the standard 20 minutes.

A study of the effect of time of heating on the physical qualities of a powder metallurgy product indicates that this effect is less



noticeable than that of almost every other factor in the cycle. With negligible variation, cores with the same physical properties are produced, whether we heat for five minutes or sixty. Therefore it is not necessary to complicate the procedure by reading the temperature within the box for each experiment and measuring the time of heating from the point when the contents of the box are actually at the proper temperature. It may happen that, when only a few cores are heated, the contents are subjected to sinter temperature for over 20 minutes, or that, when a number of boxes are introduced into the furnace at the same time, some reach the sintering temperature only 10 minutes before they are withdrawn. Little difference will be observed in the physical qualities of the two sets. It is for this reason that we have found the 15-minute heat period desirable.

### **Packing**

It should be pointed out that the medium in which the cores are sintered has a very marked effect on the physical qualities of the products. Elsewhere in this book there is listed the variation in strength which occurs when sintering is carried on under different atmospheres, all other factors being equal.\* It is true that when the sintering atmosphere is hydrogen, a bright, unmarked product is obtained, and that when the cores are imbedded in charcoal the products are sometimes roughened and in almost all cases dull. Nevertheless, the simplicity and safety of using charcoal far outweighs its minor disadvantages. We have found that the results obtained by using charcoal can be closely duplicated; and there is no reasonable objection to this practise so long as the student knows that the products would be somewhat different if the sintering took place in an atmosphere of hydrogen or other gas, and therefore that they may not be directly compared with the experiments of operators who employ a material other than charcoal.

It is of course true that slight carbonizing occurs when iron cores are sintered in charcoal. However, the period of heat treatment is so brief that this results only in a minor skin effect and varies from product to product so slightly that it does not interfere with the gross physical tests made on these products.

\* See page 86.

### Handling Powder

The first step in making cores is to mix the necessary powder. This is accomplished best on a piece of oilcloth with spatula manipulation. Too much emphasis cannot be placed on the importance of a thorough mix. Without proper care, there is a marked tendency for the graphite to segregate; and if a poor mix is made, such segregation will result in products of irregular physical characteristics. When the powders have been sufficiently mixed the die is loaded. Care must be used here to avoid segregation and breakdown of the mix. This is best achieved by filling the  $\frac{1}{2}$ -in. hole in the die with a broad-bladed spatula. The large one used for mixing is employed for this purpose. It would of course be possible to fill the opening in the die cleanly and neatly by transferring powder to the cavity with the aid of a creased paper; but as the powder ran down the paper automatic classification would occur. The apparently clumsy method of loading the die is deliberately used to reduce classification and segregation to a minimum. When the die is filled, the excess powder is removed and the upper part of the die, the plunger, is pressed slightly against the powder so as to enter the cavity for approximately  $\frac{1}{4}$  in.

Note that the filling of the die is volumetric. No attempt is made to make each loading of a uniform weight of powder. The emphasis is on the volume, as it is in commercial operation.

### Further Details

The die is now transferred to the platform of the press and with a regular full stroke the pressure is raised to the desired point—in the standard an 8,000 lb. per sq. in. dial reading. At this point the pressure is quickly released. Now the arbor replaces the base of the die and, still using a steady stroke, the formed core is ejected from the die.

We have found it advisable to use a punch to mark the cores so that they may be identified. A quick, sharp blow will usually indent the core deeply enough to identify it after it has been processed without damaging the green core. An occasional core may be lost by striking too hard or at an angle, but once the student has gained experience, this rarely occurs. When the proper number of

cores have been made, they are measured and transferred to the sintering box. The lower level of this box has previously been filled with about  $\frac{1}{2}$  in. of charcoal. The cores are set horizontally side by side about  $\frac{1}{4}$  in. apart. The box is now filled with charcoal and closed with a cover which should fit tightly.

We have found that if the box is marked with ordinary chalk, the identification persists throughout the heat cycle; and since this can easily be erased, it serves admirably for this purpose. The box is now placed in the furnace which for the standard is operating at  $1600^{\circ}$  F. It is necessary to emphasize to the student that the temperature required must be closely controlled during the last 15 minutes of heating; and he must watch his rheostat closely so that the temperature, which usually drops below  $1500^{\circ}$  F. when the oven is loaded, is brought back to it immediately and carefully maintained as long as possible through the preliminary heating period, and definitely through the 15-minute sintering period. He should be advised to throw out and repeat any experiment in which the heating has got out of control, as there is probably no factor affecting physical characteristics which has a more marked effect than a variation in the sintering temperature. When the time of sintering has elapsed the box is removed and allowed to cool to room temperature. It is important to observe that no opportunity for oxidation must be permitted until the temperature of the cores is so low that oxidation is negligible. The box must remain tightly sealed until cold.

We have found it convenient to then dump the contents of the box on a  $\frac{1}{4}$  or  $\frac{3}{8}$ -in. sieve and shake the cores free of charcoal. The charcoal may be used repeatedly, and actually improves in quality as it is used. When it is new, it contains a high percentage of volatiles, and though the box may be filled when inserted in the furnace, much of the charcoal is burned away, occasionally to such an extent that some of the cores are exposed to the atmosphere and may be oxidized while cooling. Eventually however, the loss in volatiles is so reduced that this never occurs. We have found it good practice to add small quantities of fresh charcoal to each charge of used charcoal, so that shortly after the beginning of a term there is completely satisfactory packing material available.

The procedure from here on need follow no definite pattern, except that before any tests are made the cores are measured and

weighed. Thereafter it is easiest to run the Rockwell test first and conclude with the break test.

### **General Instructions for Tests**

The length and diameter of the green cores are measured with a micrometer. If the diameter of two or three cores is measured it will be found that all the cores made of the same material under the same pressure will be of the same diameter to the third decimal place. The length, however, may vary considerably. The lengths of the five test cores should be determined and added together. The weight of the five test cores should be made in a single operation, and for our purpose weighing to the first decimal place is quite close enough. To determine the volume and specific gravity, the cores should be considered a single cylinder, as if they consisted of a single core with a diameter equal to the average diameter and a length and weight equal to the sum of the length and the weight of all five cores.

After heat treatment the cores are measured and mathematically treated in exactly the same way as were the green cores.

### **Hardness**

No special precautions need be taken for the Rockwell test, except that the operator should be sure that the fin, or flash, which sometimes is formed in the pressing operation and is rarely destroyed during sintering, is removed before the core is placed in the arbor. This is easily accomplished with a coarse file. The hardness of each core should be determined at three points. The first point is determined near one end. The core is then turned  $60^\circ$  and the next reading taken in the center. It is then turned another  $60^\circ$  and the final reading taken at the other end. As the student will quickly discover, in many cases the hardness of one end will be of a different order of magnitude from that at the other end, the center hardness invariably lying somewhere between the two. The high end readings and the low end readings should be grouped separately. The five sets of readings should be averaged and the report on the hardness of the cores should show the average high, the average low and the center hardness. As a check on the quality of the technique, the report should also include the absolute high reading and the absolute low reading.



*Photo by New York City WPA; Courtesy Board of Higher Education of N. Y. City.*

#### Determining Rockwell Hardness

### **Averaging Results**

Students have a tendency, in determining averages, to add together all their results and divide by the number included. Early in the course it is desirable to point out that irregular results should not be included in the average. If, for example, four of the cores show a hardness varying from 57 to 63 and the fifth core shows a hardness of 80, the 80 should not be included when the average hardness is calculated. It is obvious to an experienced technician

that the core showing the hardness of 80 is not representative, and that some error in technique has produced it. What is said here of hardness applies equally well to any quantitative determination, particularly to the break test.

### **Break**

The only precaution required in determining break-strength is to see that the pressure is applied with even strokes. The operator has a tendency to slow up the speed of applying the pressure when he gets to the point at which he expects the break to occur. The best results are obtained if the student operating the lever leaves observation of the dial reading to another member of the squad. Any irregularity in applying pressure will cause irregular results, but it is very easy to cultivate a steady up and down motion on the lever and a full stroke at about the rate of one per second.

### **Flow**

A few general rules should be laid down for the technique employed in the examination of metal powders. The flow test has already been discussed in some detail. No special precautions are required here, but the test may be facilitated if one student operates the stop-watch while another pulls the plug on the funnel and calls off the moment when the powder has filled the receiving vessel to the mark.

### **Apparent Specific Gravity**

In order to determine the specific gravity of powders and closely check results, the technique must be precisely duplicated. Any variation will result in the introduction of more or less powder into the weighing flask, and a slight error here is multiplied at least four times in the result. In order to avoid classification, the same method is resorted to as in filling the die cavity when making cores: a broad spatula is used to feed the powder into the small mouth of the 25-cc. volumetric flask. The flask must remain stationary during this operation. We fill the flask roughly to the mark. Usually the level will not be even but at an angle of approximately  $45^\circ$ . The mark will bisect the line of surface. At this point the flask is lifted from the table and held just below its mouth with two fingers. Four sharp strokes are then applied to the flask with a pencil at

four opposite points on the bulb. This will cause the powder to settle, usually to a point just about where the neck begins. The neck is then filled to the mark with as much care as possible, and here a small spatula with a  $\frac{3}{8}$ -in. blade may well be employed. Care should still be taken to avoid classification, but even extreme classification of the few centigrams of powder required to complete the filling is hardly detectable.

Care should be taken to wipe the mouth of the flask as well as all external surfaces, and it is then weighed to the second decimal place. The tare is subtracted, and the net weight of the powder, multiplied by four and divided by 100, gives the specific gravity. Specific gravity should be reported to the second decimal place from not less than two tests. Two tests are usually quite sufficient, and the results will generally check exactly in the second decimal place.

### The Experiments

The laboratory experiments can be classified in two groups. One group has been devised to familiarize the student with the principal methods of producing metal powders and the techniques employed to classify and report on them; the other to illustrate the established methods of making powder metal products, to measure product characteristics, and to learn the effect of varying procedures.

The experiments in the first group are duplicated by all squads with but minor variations. With reasonable attention to detail, and a procedure equivalent to the accuracy demanded by commercial analytical methods, comparable results with close checks may be attained.

In the second group of experiments, major variations have been introduced. No squad exactly duplicates the work of another. These experiments follow a research plan. In each of them all but one factor (the function of which is under study) is exactly duplicated. This plan makes it possible to determine the function of a single factor over a wide range.

The procedure for each experiment is outlined for "Squad A." The variation to be employed by the other squads follows. These squads follow the procedure exactly but introduce the variation assigned. The full results of the experiments are not complete until the work of all squads has been reported. The reports will be correlated in the lectures.

To make the coördination of results significant, it is essential that the *ONLY* variation introduced into each experiment be the one planned. Constant attention to detail, and care and accuracy in measurements and calculations will make possible consistent results which illustrate the function of the one factor under examination, unmarred by the effect of faulty technique. In order to study the effect of variations in the production cycle as quickly as possible, all the first group of experiments on product characteristics are made with bronze mixtures. Thereafter a few experiments have been added to familiarize the student with products of different compositions.



## Chapter 9

### Laboratory Cost for the Course of Training

Under present conditions, the cost of an item is often less important than the right to purchase it and the chance of getting it delivered in time to be of service. This situation will not change until the end of the war, and any attempt to predict prices at that time is futile. But as the Government has sponsored courses in powder metallurgy in the past and is prepared to continue to do so, there is no reason to believe that, if a course is planned in a district not served by other agencies, it will not be granted a high enough priority rating to enable the necessary equipment and material to be secured. Men trained in this industry are needed to further the war effort. Industrialists who are converting some of their facilities to powder metallurgy processes also should be able to get whatever priorities are required to set up their laboratory control and development, for the conversion would not be permitted unless the new output were planned for war purposes. Such newcomers will badly need the help of the laboratory.

It would appear then, that while we fight through the long duration, we shall continue to equip both educational and industrial laboratories for development work in this field. Before the installation is approved, some idea of its cost must be known. To help calculate this, an estimate is offered in this chapter. Costs are current costs, and will vary from those used here; but items have been liberally priced, and any business man familiar with the trend of prices at the time he makes his budget should reach a fair approximation with the help of these figures. Only round numbers have been used.

The estimate is made for a student laboratory and the particular experiments outlined in this book, but a little selective discrimination will make it of use to the industrialist.

It is assumed that four squads will take the course, comprising a single section of sixteen men, who will use the laboratory during a

three-hour period. It is assumed that the laboratory is fitted with work benches and lockers; water, air, gas and electric outlets; and the usual facilities, including furnace tongs and gloves, bottles and corks, glass tubing, rubber tubing, and the oddments which must be available wherever research or development work proceeds. Some of the equipment listed below will already be available in any laboratory working on chemical or metallurgical projects, but the rest should be filled in, if the course is to be given efficiently.

*General Equipment*

No.	Equipment	Cost
1	Arc light system for microscope	\$ 65.00
1	Aspirator	1.20
1	Ball Mill and Motor Drive	110.00
30	Balls, Steel $\frac{1}{2}$ -in.	15.00
1	Balance, Analytical	130.00
2	Balance, Bulk	100.00
2	Balance, Pan	25.00
7	Beaker, 400-cc.	1.50
11	Beaker, 50-cc.	2.00
1	Bottle, wash, for gas	2.00
2	Bottle, wash, for distilled water	2.00
3	Bottle, dropping, 50-ml.	1.00
24	Box, Sinter (made in local shop)	5.00
2	Break Tester (made in local shop)	10.00
	Cover glasses, microscope	1.50
1	Cross-hair eyepiece for microscope	5.00
1	Cylinder, Measuring, 250-cc.	1.00
1	"      "      "      25-cc.	.50
2	Desiccators, large	} 21.00
1	Desiccators, small	
4	Die assemblies (made in local shop) 6 Dowel Pins \$1.55; 4 Bushings \$6.20.	15.00
1	Drying oven	50.00
1	Euscope	45.00
1	Pkg. Filter paper 11 cm., discs	2.00
2	Flasks, volumetric, 100-cc.	1.00
4	"      "      25-cc.	2.00
2	Funnels, 4-in.	1.00
7	Funnel	2.50
2	Funnel Rack	8.00
2	Furnace, capacity 2000° F., Large	340.00
1	"      "      "      Small	140.00
4	Glass plate	1.00
2	Hammer	1.00
4	Jaw clamp	5.50
4	Micrometers, 0-1-in.	30.00
4	"      "      0-2-in.	40.00

*General Equipment (cont.)*

No.	Equipment	Cost
1	Microscope	200.00
	Microscope Slides	1.00
1	Microscope stage micrometer	8.00
	Microscope tissue	.50
1	Mortar and pestle	3.00
4	yds. Oilcloth	1.50
2	Press, Laboratory, double dial	450.00
4	Ring stand	7.50
1	Rockwell Hardness Tester	400.00
1	Scale, transparent millimeter	.10
1	pr. Scissors, small	.50
4	Sedimentation tubes, to order	12.00
2	Sieves, set of 3-in.	120.00
1	Sieve, 80-mesh, 8-in.	5.00
1	“ 100 “ 8-in.	5.00
1	“ $\frac{1}{2}$ -in. “ 8-in.	5.00
1	Sieve shaker (designed to hold two sets of sieves at one time) Light Cenco type	90.00
4	Spatula, small	2.00
4	Spatula, large	4.00
2	Sets Stamping dies	3.00
4	Temperature indicator	40.00
6	Test tube, small	.50
6	Test tube, large	1.00
1	Test-tube rack, small	.50
1	Test-tube rack, large	1.00
4	Thermocouple	15.00
1	Tubular Furnace	60.00
1	Tube, Silica	10.00
4	pr. Watch glass, tared	4.00
4	Watches, stop	54.00
1	Weights, set of analytical	15.00
4	Weights, set of, 500 to 1.0 grams	44.00
1	Valve, Reduction, for Hydrogen tank	25.00
	Miscellaneous	25.00
	<b>Total</b>	<b>\$2795.80</b>

*Raw Materials*

Quantity	Equipment
1 Tank	Hydrogen
12.5 Kilo	Standard Copper
2 “	Fine Copper
2 “	Coarse Copper
4.5 “	Iron powder (hydrogen reduced)
1.25 “	Standard Tin

*Raw Materials (cont.)*

Quantity		Equipment
1	Kilo	Copper oxide
4	"	Ferro silicon
1	"	Aluminum
5	"	Charcoal
1.5	"	Zinc
300	gm.	Lead powder
500	"	Graphite
500	"	Sterotex
60	"	Chromium (subsieve)
60	"	Copper (subsieve)
100	"	Salicylic Acid
100	"	Camphor
2	Liters	Acetone

In round figures, then, all the equipment necessary for the course will require an expenditure of about \$2800.00. An industrialist can equip his laboratory for about \$1900.00.

To facilitate ordering, the quantities of necessary expendables have been listed, but in view of the price fluctuations of these supplies, the cost has not been itemized. It may be said however, that at current prices the cost of the expendable material for sixteen students per term should not much exceed \$50.00.

## Chapter 10

### General Instructions to Students

In all experiments in which powdered metals are fed into a die, and compressed in a hydraulic press, great care should be taken to protect the die, which is an expensive precision instrument. It has been made to dimensions with a tolerance of .0001 in. It takes nearly two weeks to produce a die of this kind, and damage to it will seriously handicap instructions. *Always check and make certain that the LUBRICANT has been added to the powder, when required, before it is placed in the die.* A single pressing of unlubricated powder may completely destroy the usefulness of the mold. When in doubt, ask the instructor *first*.

Make seven cores for each experiment. Five are to be measured before and after sintering to determine dimensional changes due to the heat treatment. The weight of the same five cores (weigh all five in one operation) in conjunction with volume measurement is used to calculate the apparent density. Report all densities in GRAMS PER CC. Measurements of only five green cores are required.

Use the same five cores to determine hardness. Be sure to remove any flash or fin from the cores before placing in the anvil of the Rockwell tester.

Use the same five cores for the break test.

Use the two extra cores to check irregular results.

One hundred and fifty grams of powder should be sufficient to produce the seven cores required. The mold is filled volumetrically, *without* tapping to settle the powder.

The plunger should be set in carefully by hand and the upper plate of the press brought slowly in contact with the head of the plunger. Pressing is to a given pressure and as soon as that pressure is indicated on the dial, the force of the press should be released.

This makes a green compact. It is still in the die in contact with the plunger and the base of the mold. Remove the base and re-

place it with the anvil or arbor provided for the purpose. Now slowly bring the top of the press in contact with the top of the plunger, which now becomes an ejector. Do not use the ejecting action jerkily, but with a smooth motion eject the compact. Green compacts may be handled with reasonable freedom, provided they suffer no undue shock and care is taken not to crush the edges. As they are made, identify the cores with a number punch.

After a sufficient number of green compacts have been made and whatever measurements required by the experiment are taken with the micrometer, the cores are ready for the sintering operation.

Most of the products made during this course are subject to oxidation, and this factor must not be permitted to affect the experimental data. To avoid this, the green cores or compacts are set in a loose bed of charcoal in a sheet-metal box. They are separated from one another by a small ridge of powdered charcoal and then completely covered by the reducing agent, after which the lid is placed on the top of the metal container and the cores are ready to be placed in the furnace. Identify the samples by marking each container with chalk.

Be sure to identify each group of cores so that after they have been removed there is no danger of confusion. Obviously, if the heating time ends only shortly before the laboratory period, the material in the furnace will be too hot to handle and the containers should be removed from the furnace and put aside so that subsequent work can be continued on them at the next laboratory session. Be sure to place these apart from any inflammable material.

In removing the containers with the compacts from the sintering furnace to the soaking oven, if soaking is called for, be sure to reduce the interval to a minimum. You will note that many of the pressures used for making the green compact are 40,000 lbs. per sq. in. The die has been designed with a diameter of 0.5000 in. After it is ejected the compact may have a greater cross-section than when in the mold. The pressure required on the hydraulic press to obtain the pounds per square inch directed in the experiment is calculated from the area of the plunger and the *compact* before it is ejected, not on the area of the ejected core.

The rate of heating and cooling, and particularly the temperature of sintering, have a marked effect on the properties of powder metallurgy products. It is advisable that one member of the

squad give his entire attention to *control of temperature*. The furnace should be holding at a temperature about 100° F. higher than required before putting in the box of compacts. It will cool somewhat on loading the cold box and on opening the door. Fluctuations in current supply or other factors may vary the temperature. The operator can keep a good control with the rheostat and if necessary by opening the door slightly.

In general instructions call for heating 15 minutes, but tests have shown that the inside of the box does not reach indicated temperatures in less than 20 minutes. Therefore the full time in the sintering furnace is 35 minutes.

### Timing the Work

In general the cores prepared during one laboratory period are not ready for tests until the following one. However, it does not take a full period either for preparing cores or testing them. Plan the work so that the cores for one experiment can be prepared in the same laboratory period in which tests are made on those produced during the preceding session. Work as a squad. Each student must participate in all parts of each experiment.

### Checking Apparatus

Most of the equipment used may be employed without fear that it will introduce errors. Before using a micrometer make sure that it is accurate. If in doubt, see the laboratory assistant. Before reading temperatures, be sure the thermocouple is properly located in the furnace and is recording properly.

### Test Methods

Most of the techniques employed in powder metallurgy are common laboratory procedures. A few require special emphasis or have been adopted as standard for the course. It is important that the standard methods be employed by all students as closely as possible so that the results will be useful for comparative evaluation.

**Sampling.** In studying powders it is important that the sample be representative. Every effort must be employed to avoid mechanical classification. The *entire* sample should be thoroughly mixed and quartered down to a quantity slightly in excess of that required for the test. Mix by folding in and heaping up on a glass

plate or oilcloth sheet with a broad spatula. Then spread the sample in a thin layer. Divide it into four equal parts and place on one side opposite quarters. Thoroughly mix as before the other two quarters, spread thin, divide and discard, mix and repeat until the well quartered sample is just large enough for the purpose.

**Sieve analysis.** Sieves are expensive precision instruments. They should be handled with care, and always left clean. When nesting or parting them, be sure to place no strain on the fitting edges. Do not attempt to clean the surface with a brush; use a blast of filtered air. A 50-gram sample shaken 20 minutes is standard procedure. Report per cent unaccounted for when reporting analysis.

**Apparent Density.** The apparent density of powders is determined by filling a tared 25-cc. flask to the mark and weighing. It is obvious that the method of filling the flask will markedly influence the result, since it is easy to pack the flask with more or less of the sample. The following method will give results which check to the second decimal. Fill the flask to the mark without agitation and without disturbing the flask in any way. Lift the flask by the neck with one hand. Strike it four sharp blows with a pencil, one in front, one in back, one on the left and one on the right side. Replace the flask gently on the table top. The level of the powder has now fallen well below the mark. Without further agitation fill to the mark and weigh.

**Rate of Flow.** The rate of flow of metal powders is an arbitrary figure derived by timing the seconds elapsed to fill a 100-cc. flask to the mark through a 60° glass funnel from which the stem has been removed. About 200 cc. of powder is held in the funnel, which is unstoppered over the flask and the interval for filling timed. Tap the *flask* slightly during the flow but not sufficiently to pack the powder. Do not force the flow. Do not disturb the funnel in any way. *If flow stops before 100 cc. has passed, the powder is reported as having zero flow.*

**Break Test.** The only special precaution in using the break tester is to make certain that the pressure is applied at an even rate. It is best to have one man watch the gauge while the other applies the pressure so that the operator of the lever is not tempted to slow his motion as the pressure seems to be reaching the breaking point. After using the break tester broken cores should be thrown away so as to *keep the table top clean.*



**Hardness Test.** Use an arbitrary scale, adapted to this course. To avoid confusion, identify the hardness as a "K" hardness. This is derived from a  $\frac{1}{4}$ -in. ball, a 60-kilo weight, and the readings of the B scale on the dial. Five cores should be tested and the average reported. Three readings are made on each core. Take a reading near one end. Turn the core through  $60^\circ$  and take a reading on the center. Turn through  $60^\circ$  and take a reading near the other end. Cores produced under certain conditions differ from end to end. Report the three readings separately. Negative readings up to minus 20 have some meaning; beyond this, no value. Readings below minus 20 should be reported merely as "very soft."

### **Notebooks and reports**

Rough laboratory notes should be kept in a loose-leaf book, preferably on standard  $8\frac{1}{2}$  x 11-in. cross-section paper. Each page should have the student's name, section, squad and date (on which the notes were made) in the upper left-hand corner. In the right-hand corner should appear the experiment number. These notes must accompany reports. Notes should consist primarily of statistical data with only enough text to identify the record beyond question. When making laboratory notes, time may be saved if data are recorded by one member of the squad for all members. Stack the loose-leaf sheets with carbon paper inserts. At the end of the experiment each student takes one carbon copy, which should carry all the data required as the basis for his individual report. These data should accompany the report.

Reports are due one week from the date on which an experiment is completed. The experiment number and the student's name, section and squad must appear on each page heading. This information and the title of the experiment are to appear on the cover sheet of each experiment. Two or more experiments must not be clipped together. Separate pages of each report should be clipped together with the rough notes. **DO NOT USE STAPLES.** Use easily removable clips.

Make reports as brief as possible. Make them complete. The bulk of the reports will be on cores made with variations or under varied conditions, and the object of the experiment is to compare the characteristics of these variations with one another and with cores made under standard conditions. When possible use the following form.

Name, Section, Squad, Date \_\_\_\_\_  
 (submitted)

Exp. No.\* \_\_\_\_\_

Object:

Procedure: (Unless a variation not appearing in the text has been introduced, procedure may be omitted.)

	<u>5% Tin</u>	Standard†	<u>12% Tin</u>
Density (before sintering)			
Density (after sintering)			
Density (% change)			
% Elongation			
% Diam. Change			
% Volume Change			
Hardness	{ Maximum Minimum Average high Average low Average center		
Break	{ Maximum Minimum Average		

Observations: (Only when necessary and not covered by above table.)

Discussion: (When necessary, or to explain unexpected result).

Conclusions: (BRIEF) NOTE: *Cum grano salis* Conclusions cannot be drawn from one experiment. Perhaps procedure was faulty, perhaps a gauge or thermometer was wrong. Perhaps a calculation was in error. (WATCH DECIMALS!) Be sure to attach graphs when called for.

\* Experiment 8 is used as an example here.

† The data headed "Standard" should appear in its proper place. Here, since the standard bronze had 10% tin it is reported between the higher and lower tin content bronzes. Had the tin experiment been on 12% and 15% tin the standard would be reported in the first column.



## **Chapter 11**

### **Experiments, Precautions and Discussion of Results**



### **Experiment 1. Effect of Time and Volume on Ball Milling**

*Equipment.* Ball mill of 1 gallon capacity  
1-in. steel balls, sufficient to fill the mill one-third full  
Pan balance, capacity 1 kilo  
Set of weights, 500 grams to 1 gram  
Bulk balance  
Set of weights 50 grams to 0.05 gram  
Set of 3-in. sieves from 80 to 400-mesh  
Sieve shaker  
One 8-in. sieve 80-mesh  
One 8-in. sieve  $\frac{1}{4}$ -in.  
A flow tester  
25-cc. volumetric flask  
One kilo of ferrosilicon crushed to 80-mesh

*Procedure.* Screen through 80-mesh sieve and weigh 300 grams of ferrosilicon and transfer it to the ball mill. The mill should turn at 85–90 rpm. At the end of 20 minutes the mill is stopped and the contents transferred to the  $\frac{1}{4}$ -in. sieve. This separates the steel balls from the crushed ferrosilicon. The sample is well mixed and quartered. Fifty grams of the representative sample are placed on the top of the nest of sieves and the nest shaken for 20 minutes. At the end of this period the contents of each sieve are removed to the balance pan and weighed to one decimal place. The size distribution is converted to per cent. The results are plotted in the form of a bar graph on a scale showing per cent undersized and oversized. Another sample is tested for specific gravity and a third for flow. All the tested material, both that from the sieve analysis and the other tests, is combined and returned to the ball mill which now is revolved for another 20 minutes. At the end of this time the product is again tested for size distribution, density and flow. All the tested material is again added to the original sample and put in the ball mill, which is revolved for another 20 minutes and the product again tested. The results are shown in comparative bar graphs, and it is possible from these data to draw

a conclusion as to the effect of the time of balling on powder characteristics. Flow and apparent specific gravity are plotted against time of milling. Three sets of bar graphs are plotted to show the effect of time on ball milling.

*Instructions to other squads.*

Squads B, C and D will follow these instructions with the following variation.

Squad B will use 500 grams.

Squad C will use 600 grams.

Squad D will use 800 grams.

*Discussion.* The experiment indicates that though there is no direct relation between fineness and flow, the coarser material has the faster flow. In general this coarser product has a better size distribution, that is, it more nearly approaches the statistical curve of normal distribution, and it appears that flow decreases as the distribution departs from this curve of normal distribution.

It should be remembered that flow is a function of particle size distribution and shape as well as of fineness, and the student is warned against drawing the conclusion that flow decreases with particle size. Any conclusion must be modified by these other factors.

The apparent specific gravity decreases with fineness. This conclusion is inescapable within the range and for the conditions of this experiment. To account for this we must postulate the increase in surface film air, adsorbed on the increased surface. Note that the particle surface increases as fineness increases.

The most valuable lesson from this experiment is that there is an optimum load and optimum time of milling that should be used to produce the maximum percentage of the desired size range of particles. The law of diminishing returns operates forcibly in respect to the time of milling. If, for example, it is desired to obtain a product all below 275 mesh, the increase in yield of particles of this size that may be obtained by milling over 50 per cent longer than 40 minutes is negligible. If the load is 400 grams, for instance, the 50 per cent increase in power consumption, and the added investment in equipment required to secure the same quantity of product, yield but about two per cent more of the desired product.

To obtain maximum yields in a minimum of time the load must be adjusted to the material. The greatest yield of desired size is not obtained from the largest load. If ball-mill operations of any kind are contemplated, the little time required for experimental work to determine the best load and time will be amply repaid. Though it will take longer and require some ingenuity, a study of the effect of varying the number of balls and the size and size distribution of the balls used will usually be justified.



## Experiment 2. The Effect of Raw Material Particle Size on Finished Powder Particle Size

- Equipment.* A tubular furnace with a silica tube  
A thermocouple in guard well with a temperature indicator, range 0 to 1000° F.  
A two-holed rubber stopper to admit thermocouple and gas inlet to the silica tube  
A one-holed stopper to permit exit gases to flow through a glass tube bent at right angles and drawn to a small opening, to permit the exit of the gas or steam  
A supply of hydrogen with a reduction valve to control pressure  
A gas wash bottle (use light mineral oil as wash)  
A mirror or bright-surfaced spatula for testing the end of the reaction  
A set of sieves (see Experiment 1)  
An 80-mesh sieve  
A set of weights, 50 to 0.05 grams  
A bulk balance  
A flow tester  
A 25-cc. volumetric flask  
A large, wide-mouthed jar  
A combustion boat. This boat is best made of thin copper sheet. It should be nearly as long as the furnace and should fill half the diameter of the silica tube.  
200 grams of copper oxide reduced to a size sufficient to pass an 80-mesh sieve  
A Bunsen burner  
Test tubes  
Test-tube rack

*Procedure.* The copper boat is loosely filled to a heaping load with the copper oxide, previously screened through an 80-mesh sieve. This is inserted into the center of the silica tube. The tube

is closed at one end with the exit jet and at the other with the thermocouple-well and gas inlet. The thermocouple-well should rest in the powder as near the center as possible. After the tube is tightly closed, the hydrogen is permitted to enter under about 5 lbs. pressure. After it has flowed for about 2 minutes the test tube is put over the tip of the exit and a sample of exiting gas taken. Holding the test tube down, it is conveyed to the lighted Bunsen burner, which should remain a safe distance from the exiting gases. If all the oxygen has not been swept from the tube, the contents of the test tube will burn with explosive vigor—otherwise a slow flame will creep down its edges. The lighted test tube should be brought back to ignite the exit gases. *Whenever it becomes necessary to relight the outflowing gases this procedure should be followed.* If this care is used the danger from explosions is practically eliminated. The rate of flow of the hydrogen can now be regulated so as to keep a small flame burning at the exit. Once the flame is steady the furnace may be heated. The temperature should be raised to approximately 600° F. and carefully controlled by means of the rheostat and opening and closing the top of the oven. During the reduction, the combustion tube should be turned from time to time so that the powder within the tube is not heated hotter in one spot than in another. Naturally, when the tube is turned the copper boat will empty, but it has served its purpose, which is merely to convey the entire load to the center of the tube. The reaction is highly exothermic and once reduction begins it will be necessary to lower the temperature of the furnace so that the temperature of reduction does not become excessive. A high temperature will cause sintering, and slight sintering is probably unavoidable. As reduction proceeds, the flame at the exit will be extinguished by the stream of steam which is given off. Care should be taken to see that the steam does not condense and run back into the silica tube. From time to time samples of the exiting gases may be taken in the test tube, as in the first instance, and an attempt made to relight the exit gases. After the violence of the reaction has been slowed, it will be possible to relight the gas in spite of the presence of steam; and it should be kept relit until the end of the experiment, except when the flame is temporarily extinguished in order to test for the end of the reaction. This is determined by placing a cold bright object such as a mirror in the

stream of exiting gases. The bright spatula serves this purpose well. When this no longer fogs, due to the condensation of small traces of steam, the reaction is complete; the furnace is shut off and the tube allowed to cool. During the cooling the flow of hydrogen is continued. Cooling may be facilitated by sponging the silica tube with cold water. However, do not judge the temperature of the copper by the external temperature of the tube. The thermocouple has not been removed and the hydrogen should be permitted to flow until the indicator shows that the powder is at room temperature. When this occurs, it is safe to stop the flow of hydrogen and remove the copper powder which has been reduced from the oxide. In order to reduce oxidation to a minimum, transfer the contents of the tube as rapidly as possible into a wide-mouthed bottle which has previously been filled with illuminating gas and continue the inflow of illuminating gas while making the transfer. Then seal the bottle tight so that the copper may be stored in this reducing atmosphere.

Break up the agglomerates carefully but do not grind.

Screen the powder through an 80-mesh sieve.

Determination of apparent specific gravity, flow and particle size distribution are required on both the oxide and the copper made from the oxide. The size distribution should be reported in a two colored bar graph; one color representing the oxide and the other the copper.

*Discussion.* In general, particle size distribution after reduction bears a close relation to the distribution before reduction. There is a slight growth of very fine particles and a slight decrease in the upper sizes. This evening out of the distribution curve improves the flow characteristic of the reduced powder. Heat alone, often spoken of as an annealing, will often effect this improvement. Observe the slight increase in apparent specific gravity. Such an increase is often very marked if reduced iron is annealed, a common practice in the production of some iron powders.

### Experiment 3. Determination of Size Distribution of a Metal Powder of Sub-sieve Size by the Pipette Method

*Equipment.* Sedimentation apparatus  
Analytical balance and weights  
11-cm. diameter circular filter paper  
Two stop-watches  
Small scissors  
Drying oven set at 105° C.  
12-in. desiccator  
Seven 50-cc. beakers  
Wash bottle  
Distilled water  
15 grams chromium powder, sub-sieve size, previously dried and retained free from moisture

*Procedure.* Tare six filter papers against one. Number these papers from 1 to 6 and place in the funnels racked to receive the material siphoned from tubes 1 to 6 respectively. The tare paper should also be placed in a funnel and washed with about 200 cc. of distilled water.

The volume of the six suspension tubes is listed. From the bottom to the lower mark is exactly 20 cc. From this mark to the upper mark is exactly 20 cm. The *volume* from the bottom varies for each tube.

Weigh and transfer to each tube 2 grams of the chromium powder. The two grams may be approximate but should be weighed to the third decimal place. Dilute each tube to the upper mark, and shake well to accomplish thorough dispersion. At the end of 2, 4, 8, 16, 30 and 60 minutes respectively, rapidly siphon the suspension to the low mark of tubes 1, 2, 3, etc. The siphon transfers the suspended material from the test tube to the special separatory tube.

Now transfer the contents of the separatory tube to the respective filter papers, retaining the residue on the paper and discarding the filtrate. The separatory tube should be thoroughly washed into the filter so that it is analytically clean and all the suspended particles are on the filter paper. The filter papers may now be

removed from the funnels, folded over the top and placed in the 7 small beakers provided for the purpose. The 7 beakers with their contents are transferred to the oven and are air-dried to constant weight. Three hours are usually quite sufficient for this purpose. Cool in desiccator. Using the tare filter as a counterweight on one pan of the balance, weigh each sediment respectively. From these data the percentage distribution of particle size may be calculated.

### *Calculation of Sedimentation Curve*

Let  $w$  = grams of solid between rings at start.

= cc. between rings (total volume minus 20 cc.) times grams per cc.  $\left(\frac{\text{gms. weighed}}{\text{total vol.}}\right)$ .

$w'$  = weight of material found on filter

then  $w - w'$  = weight of material settled in time,  $T$  and

$100 \times \frac{w - w'}{w}$  = per cent material settled in time,  $T$

With time on the abscissa, plot the percentage settled against time. This curve is a sedimentation curve. This is the basis from which to draw a distribution curve and a cumulative curve. The distribution curve shows the percentage of material corresponding to a given size. The cumulative curve shows the percentage of material larger than a given size.

*The Cumulative Curve.* Refer to the sedimentation curve. This curve shows the amount of material, on the ordinate called  $P$  settled in time  $T$ , which consists of  $S$ . Further,  $S$  equals

(1) Particles larger than a certain size and all those of that certain size large enough to settle from the top of the column in time  $T$ , and

(2) Particles smaller than these, but which reached the lower mark because they started nearer to it, and which would not have reached it had they started higher in the tube. These fell at the

rate  $\frac{dP}{dt}$  and in  $t$  time amount to  $t \frac{dP}{dt}$

From 1 and 2,  $P = S + t \frac{dP}{dt}$  (Equation 3).

If we draw tangents from the sedimentation curve they intercept  $P$  at  $W$  and we observe

$$W = P - t \frac{dP}{dt} \text{ (Equation 4).}$$

But from equation 3,

$$S = P - t \frac{dP}{dt} \text{ (Equation 5).}$$

therefore  $W = S$  (Equation 6) and the point of interception,  $W$ , defines the per cent of material of that size which would fall in a given time, if it all fell the uniform distance from the top to the lower mark.

This mathematical analysis separates the total amount of material falling through the column into that portion which fell from the top because it was large enough to fall the entire distance in the given time, from finer material falling at a slower rate, but reaching the lower mark nevertheless in the same time only because it was lower in the column at zero time.

We can determine as many points,  $W$ , as we wish by drawing tangents from any points we please on the sedimentation curve. But as yet we have defined the size of the particles only in terms of time, putting it this way: "Blank per cent of the powder is big enough to fall 20 cm. in 2 minutes, blank per cent big enough to fall 20 cm. in 4 minutes, and blank per cent, etc. . . ." It is of course a certain kind of classification, but a very awkward one. To make it have significance, we must determine the relation between the diameter of the particle in terms of millimeters, or better still microns, and the time it took to fall.

To transcribe these times to diameter size of the particles we resort to Stokes' Law\*, which states in effect that, all other conditions being equal, the size of a particle is directly related to the time

\* Stokes' Law applies to spheres. For accurate work it is necessary to determine a factor of correction to compensate for the error introduced by the departure of the particle under examination from the true sphere. Viscosity of water varies considerably with the temperature, and accurate work requires that  $K$  in the formula be corrected for this. In this experimental procedure, the purpose is to familiarize the student with the method and the technique, so with apologies to the precisionist, the student is permitted to ignore these refinements.

it takes to fall through a fluid medium. Stokes' Law may be written:

$$V = \frac{2}{9} g Y^2 \frac{(d - d_1)}{K}$$

where  $V$  is terminal velocity,  $Y$  the radius of the sphere,  $g$  the acceleration of gravity (981 cm. per sec.),  $d$  density of material,  $d_1$  density of the fluid, and  $K$  coefficient of viscosity of the fluid (all expressed in cgs units).

For water at 20° C. we have:  $d_1$  equals 1.0;  $K$  equals 0.010.

Expressing  $V$  in mm. per sec., and diameter ( $D$ ) in mm., we have

$$\frac{V}{10} = \frac{2}{9} 981 \frac{(d - 1.0)}{0.010} \left( \frac{D}{2 \times 10} \right)^2$$

which may be reduced to the expression:

$$V = 545 (d - 1) D^2.$$

Substituting our values (for chromium we may assume  $d = 7.0$ ) we obtain a constant  $C$ , and we can determine the size represented by a given time by substituting in the simplified equation:

$$D = \sqrt{\frac{\bar{V}}{C}}$$

in which  $V$ , velocity, is  $h$  over  $T$ , where  $h$  is the height of the column in millimeters between the marks, and  $T$  the time in seconds. Finally, convert the diameters determined in millimeters to microns.

All that is left is to draw a new curve of the points  $S$ , the per cent of material larger than a certain size, as taken from the intercepts of the tangents drawn from the sedimentation curve, by plotting them against the micron figures which may be substituted for the minutes formerly used. This is the cumulative curve.

From this, as from any cumulative curve, a distribution curve may be drawn, and this is required to complete the report of this experiment. The method of determining the per cent of material between any two sizes is almost self-evident. It is merely necessary to subtract the per cent of material larger than any desired size from the per cent of material larger than a size just as much smaller as is desired. For instance, we may find that 100 per cent of the

powder is larger than 3 microns, 95 per cent larger than 6 microns, 80 per cent larger than 10 microns, 78 per cent larger than 15 microns, etc. We find these figures from inspection of the cumulative curve. Then by subtraction we get:

Size	Per cent
Minus 3	0
" 6 plus 3	5.0
" 10 " 6	15.0
" 15 " 10	2.0

From the cumulative curve we may select any set of numbers we choose, guided by an interest in either the size interval or the per cent interval; but it is usually more important to fix the size interval and interpret the per cent for each size. Thus, if a cumulative curve shows that 100 per cent of the material is larger than 5 microns and 3 per cent larger than 40 microns, we might select 7 points 5 microns apart, plus 5, plus 10, plus 15, etc., or 10 points 4 microns apart, plus 5, plus 9, plus 13, etc. and read the percentage from the abscissa for each point. Then by subtraction we get the per cent for each range of size. The per cent is plotted against size on the abscissa of the new distribution curve.

The value of this curve lies in its ability to show at a glance what sizes predominate, or whether there is about the same per cent of each size in the powder. If several powders have been studied with a view to blending, the use of such curves is indispensable as a guide for determining the amount of each that must be contained in the final product to obtain one with normal distribution (usually preferred) or any special distribution desired.



#### **Experiment 4.\* Size Distribution of a Metal Powder of Sub-sieve Size by Micro Count**

*Equipment.* A microscope and lighting system  
A micrometer slide  
A projecting system such as a Euscope  
A transparent ruler marked in mm.  
20 microscope slides and cover glasses  
Microscope tissue  
50 cc. of benzol with dropper  
50 cc. light mineral oil with dropper  
50 cc. diacetone alcohol with dropper  
A fine-pointed stirring rod  
A low-temperature hot plate  
10 grams of air-dried copper powder of sub-sieve size (it is sometimes advantageous to employ the same chromium powder used in Experiment 3, and the results of this analysis may be profitably compared with the results obtained by the pipette method)

*Procedure.* The microscope is furnished with an eye-piece scale in cross-section.

Prepare 10 slides by dispersing a small quantity of the powder either dry or in benzol, a very light mineral oil, or diacetone alcohol. To aid in dispersing the powder use a very fine drawn stirring rod. Select four of the best slides. Focus one with the eye-piece. Remove the slide and replace it with a micrometer slide.

In order to calibrate the microscope, retain the focus carefully and throw the image of the micrometer scale on the Euscope finder. Measure the divisions by means of the transparent ruler and calculate the factor by which to multiply the millimeters actually measured to convert to microns. A convenient ratio is three millimeters equal two microns. Examine several fields to determine the limits that must be covered. If the powder varies from a few

\* Appreciation is expressed to Charles R. Rogers, whose paper, "Microscopic Particle Size Determination," delivered at the 1940 M.I.T. Powder Metallurgy Conference, forms the basis for this experiment.

small particles under four microns to but a few as large as 18, the student's judgment is required to tell him how he should break down the classification. It is well to use not less than 5, nor more than 10 classifications. In this case he might group the particles: 0-4, 4-8, 8-10, 10-15, 15-18; and for the purpose of calculation we may consider the particles in each group to have an average size, equal to the arithmetic mean of the range; 2, 6, 9, 12.5, 16.5, respectively.

The finder plate should be marked off in thirds in both directions, and this will leave 9 divisions on the finder plate. In the smaller ranges where the particles may run as many as 5,000 or more, count only all those of the given size within one-ninth of the field (multiply by 9 to determine the total number). It is particularly important, however, that the count of the larger particles be accurate, as their volume and weight are much greater in proportion to their frequency, and for these every particle should be counted. In commercial practice, at least 5000 particles should be classified. It is seldom necessary to count 10,000, but most operators count between 6000 and 8000. A count of about 1500 particles will be sufficient to acquaint the student with the technique and give results fairly comparable with those of other students.

As the count is made, record the results in columns. Add the count of the four slides. Divide the number in each size by the total number of particles to get the per cent distribution by number ( $N\%$ ).

$D^2 \times N\%$  gives the relative surface of each size. As only the relative surface is wanted, the formula  $\frac{ND^2}{4}$  becomes  $D^2$  upon cancelling  $\frac{N}{4}$ , which is constant. Per cent distribution by surface is figured from these data, by dividing the relative surface of each size by the sum of the relative surfaces. The sum used here as a divider gives the mean surface in square microns if divided by 100. A simple calculation may give the average diameter from this figure.

The relative weight or volume is similarly figured by the formula  $D^3N\%$ , since  $\frac{\pi D^3}{6}$  becomes  $D^3$  upon cancellation of  $\frac{N}{6}$ , a constant.

Report the percentage distribution by number of particles, by relative diameter, and by relative surface. Determine the average diameter, average volume and average surface of the particles. State the number of particles in 1 cc., the square centimeters covered by 1 cc., and the square centimeters of surface present in 1 cc. Plot the per cent distribution by number, by weight, and by surface in bar graphs.

It is to be noted that throughout these calculations we have constantly assumed that the particles are spheres. Density must be used to determine weight.

*Discussion.* Only practice can help the student prepare slides which are free of agglomerates and are representative. The student should practice making dry slides, and slides with the various dispersing mediums supplied. Until satisfied that a slide is a proper sample of the powder under study he will only waste time measuring the projections on the screen and counting them. It is not possible to find some good and some bad fields on a single slide. If some of the fields are bad they have either gained or lost some of the particles to others and therefore have destroyed the value of these other "good" fields.

A single measurement is made of each particle counted. The measure taken, for reasons stated elsewhere in this book, is that diameter which would be the controlling diameter if the classification were by sieve instead of observation. It is easier to observe which dimension this is than to describe it. Note that it is not the long diameter, since a long, narrow particle would pass a sieve opening much smaller than its long diameter. But a moment's observation will always show which dimension is the controlling one.

A convenient method of recording the count, which after the first few hundred or so recordings will serve as a rough check on whether or not the analysis is progressing well, is to head columns with the size of each classification. This will give five to ten headings across the top of the page. As a particle is measured, make a single stroke under the proper heading. Next to this put a stroke for the second particle of that size; on the fifth recording of this size, cross the four previous marks. On the sixth, make a stroke under the first, on the seventh, one next to this and so on, crossing the four strokes with every fifth. Since there will be more marks in

one column than in another, after a certain number have been counted a definite trend will be discernible by noting how far down the paper each column has been filled. At any time when such a trend seems well marked, draw a light pencil mark from the center of the last mark in the column to the center of every other column at the point the last mark has been made. This, of course, actually defines a rough distribution curve. After approximately every 100 counts a similar curve should be drawn and compared with those above it. The curves should be roughly parallel and should smooth out as the count proceeds. If they differ sharply it is a certain sign that the fields counted do not represent the powder, or that some other error of technique is present.

The work is reported in three bar graphs. It should be noted that the graphs of weight and surface differ markedly from the graph representing the number of particles. The focus of this experiment is on number, but in general the per cent distribution is expressed in weight. A sieve analysis or sedimentation test always reports in terms of weight, and it is important that the student learn to make this conversion.

Different applications require a different emphasis. The pigment industry might be interested in the surface covered by each cubic centimeter of powder, whereas makers of gas masks would be more interested in the total surface presented by one cubic centimeter. In practice it is usually necessary to know something about the use to which figures are to be put before an intelligent report of even so simple a thing as a microscopic count can be made.

## Experiment 5. The Effect of Pressure on Bronze Product Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8- or 10-in. 100-mesh sieve  
Lubricant†  
Die assembly  
Press  
Sintering boxes  
Charcoal  
A furnace operating at 1600° F.  
A ¼-in. (8 or 10-in.) coarse sieve  
Set of stamping dies, a light hammer and steel plate.  
Rockwell Hardness Tester  
Break tester  
Two micrometers, 0 to 1 in., and 0 to 2 in.‡  
A bulk balance with weights 50 to 0.05 grams.  
A pan balance with weights 500 to 0.1 grams.  
500 grams copper powder, 100% through 100 mesh,  
“Standard,” 50% through 325 mesh  
50 grams tin powder, 100% through 100 mesh,  
“Standard,” 75% through 325 mesh  
10 grams graphite (Acheson or Dixon finest flake),  
“Standard”

*Procedure.\** Weigh out the proper quantity of the copper, tin and graphite to make three sets of cores. The cores weigh approximately 20 grams each. In all experiments in which cores are made,

\* For further details see p. 120. General instructions for making metallic cores.

† Sufficient Sterotex is added to acetone to make a thin cream. The bottle is closed with a cork through which the wire of a test-tube brush protrudes. The brush itself remains in the suspension. Although it is not always necessary to lubricate the die, it is advisable to have this equipment on hand for all experiments in which a die is employed. To lubricate the die it is merely necessary to pass the wet test-tube brush through the cavity. From time to time care should be taken to remove the excess Sterotex which may build up at both openings in the die.

‡ Micrometers scaled in centimeters may be employed advantageously, as this facilitates the calculation of specific gravity; but if these are used the area of the die must still be measured in square inches, since standard gauge equipment records pressure in pounds per square inch.

always prepare not less than 7 cores under precisely the same conditions, in other words, 7 cores to the set. Actual determinations are made on but 5 of these, but it is desirable to have at least 2 spares in case some of the products give irregular results. Weigh the copper on the pan balance to 0.1 gram, the tin and graphite on bulk balance to .05 gram.

Use 90 parts copper, 10 parts tin; add 2 per cent graphite.

Mix this material by means of the spatula on the oilcloth. Screen the mix through the 100-mesh sieve, discarding the few particles which remain on the sieve. Remix the powder. Load the die roughly with spatula, and with the spatula remove excess. Insert the plunger. Transfer to the press. Press to the required pressure, immediately release and eject the green core with care. Mark one end of the core to identify it as belonging to the particular set; for instance, 5 of the cores pressed at 40,000 lbs. per sq. in. may each be marked with a number 4, two or more spare cores made at the same pressure may be marked with  $4\times$  or with 40. These cores pressed at 40,000 are a standard of reference used in the reports on several experiments. The text refers to these as "Standard" cores.

Calibrate and weigh the 5 test cores. The average diameter may be taken and the length of the individual cores added together so that the 5 cores may be considered mathematically a single cylinder. They are weighed together to one decimal place. Transfer the seven cores to a thick bed of charcoal in the sinter box. Fill the box to overflowing with charcoal. Press the lid on tightly. Place the box and contents in the furnace, which should have been operating for some time at 1600°. Note the time. The temperature of the furnace must be steady at 1500° within 20 minutes. Continue the heating for a total of 35 minutes. The heat cycle is recorded as a 15-minute sintering period. The first 20 minutes are necessary to equalize the temperature within the sinter box with that of the furnace. At the end of the sinter period remove the box and allow it to cool to room temperature. (It is advisable to schedule the operations so that the end of the sinter period will be reached approximately near the end of the laboratory period and the hot boxes allowed to cool until the next laboratory period. No artificial means of cooling should be employed, as this will introduce a variation into the heating and cooling cycle which in turn will affect the physical characteristics of the products.)

Under no circumstances should the lid of the sinter box be removed while the cores are warmer than room temperature, as oxidation will take place if the cores are exposed to the air before they are cold. When they have cooled, invert the sinter box on the  $\frac{1}{4}$ -in. sieve. The charcoal which will pass through the sieve is combined with fresh charcoal for subsequent use. Calibrate 5 cores and weigh as before. From these data, per cent change in length, diameter, volume and specific gravity may be calculated by correlating them with the same data obtained before sintering. Remove any mold fin or flash from the side of the cores. Place in the hardness chuck and determine the hardness of the 5 cores with the Rockwell tester. Next they should be broken in the break tester. Report the following properties: specific gravity, per cent change in specific gravity, per cent change in length, per cent change in diameter, per cent change in volume, average hardness at the hard end, average hardness at the soft end, average hardness in the center, hardness range (this consists of two figures, the highest and the lowest hardness determined and is a key to the consistency of the test). Also report the break strength.

For this experiment three sets of cores should be made, one at 10,000, one at 40,000 (Standard cores) and one at 50,000 lbs. per sq. in. The data should be graphically presented in a curve depicting the physical characteristics over the range of pressures employed. The three points are sufficient to determine a curve.

*Instructions to Squads.*

All squads will make one set of cores at 40,000 lbs. per sq. in. (Standard cores). In addition:

Squad B will make one set at 15,000 and one set at 55,000 lbs. per sq. in.

Squad C at 20,000 and 60,000 lbs. per sq. in.

Squad D at 30,000 and 45,000 lbs. per sq. in.

When all squads have reported this experiment the data will be correlated during the lecture period. After these data are discussed in class, a final curve may be drawn, based on the nine pressures studied by the entire class.

*Discussion.* A set of "conventional curves" depicting the effect of pressure on the physical characteristics of powder metallurgy bronze products is attached. Such curves will be attached to many

of the experiments, in fact, whenever the writer feels there has been enough work published or known to him to justify the implications the graph suggests.

The conventional curve is not the result of averaging the results of published findings, private sources, and student reports, though it is thought that it may come close to presenting a picture of what such average figures would give. However, the work has been reported in such varied units, and the products studied under such varied conditions, particularly of the heating cycle, that it would be impossible first to convert them to units that could be averaged, and second to correct the units with any factor that would compensate for the varied conditions of preparation. For instance, it has been reported that "strength" has been measured as a compression ratio, tensile strength, fiber strength, and finally the author's privately devised "break test." The tests have been made on cores made by students in this course, on bushings, and on specially shaped products. These have been made from mixtures of reduced copper, from electrical copper, and from powders of many varying particle size distributions and slightly varying chemical composition. If there be any system of reducing such figures to common terms so they may be averaged, the author is unaware of it.

But this work, particularly a great deal of it which has never been published, is of too great value to the industry to ignore. Though it is extremely unfortunate that no standard methods have been adopted by the industry, the figures have merit within their own framework. The conventional curve attempts to fuse the diverse findings into a composite form.\*

The first graph, No. 1, shows the relation between pressure of forming and density of finished product. It should be considered in conjunction with the density of the green compact, and the student is advised to superimpose the graph of the density of these over the graph shown. Note particularly that the increase in pressure does not yield a direct increase in density. There is a marked

\* The author is indebted to a number of commercial laboratories who, without disclosing so much information as might be considered to have private value, have made graphs and analytical data available. This, in conjunction with a few publications, notably that of R. P. Koehring, ("Sintering Methods and Atmospheres for Production Purposes," M.I.T. Powder Metallurgy Conference papers, pp. 86-89. Edited by John Wulff, 1941) and more particularly with the results of the research program of the several classes conducted by the writer under the auspices of the United States Government at the College of the City of New York, has made possible the production of these conventional graphs.



tendency for the curve to flatten out. Goetzel,<sup>21</sup> working on copper powder alone (not bronze), found a number of powders which produced sintered products of a lower density pressed at 50,000 and even 90,000 than at 20,000 lbs. per sq. in. This graph, depicting the effect of pressure on the density of sintered bronze products, shows that raising the pressure from 10,000 to 40,000 lbs. per sq. in. increased the density about 26 per cent; raising the pressure 50 per cent to 60,000 caused an increase of but 6.36 per cent. Raising the pressure from 20,000 to 30,000 lbs. per sq. in. increased the density 10.8 per cent, but raising it from 50,000 to 60,000 lbs. per sq. in. increased the density only 1.5 per cent. If then, high-density products are sought, other means than pressure must be employed. This is one of the important conclusions to be drawn from this graph.

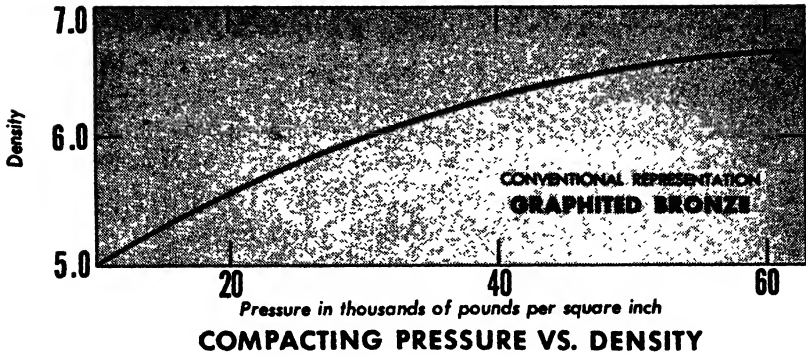
Graph 2 depicts the relation between elongation and pressure. It illustrates the fact that the higher the pressure the greater the growth of the product. If a producer is making units slightly oversize, he cannot make them smaller by increasing the pressure; on the contrary a smaller unit will be obtained if the green compact is pressed more lightly. Naturally as the heavier-pressed unit is smaller than the lighter-pressed one as it leaves the press, a compromise pressure is required. But it is important to know that growth increases with pressure, and this must be allowed for in designing a die or planning the production cycle. The term "elongation" is used here to define the increase in that dimension which is parallel to the action of the piston. The increase in the cross-section is similar, but of a lower order of magnitude. Data obtained during this experiment and subsequent ones will familiarize the student with this phenomenon—an important one for the consideration of the producer. Very often it happens that specifications for a part offer more liberal tolerance in one dimension than in another; or it may be easier to add a finishing operation to one dimension than to another to bring it within the limit of tolerance. It should be noted that the change in dimension at right angles to the source of pressure is, as regards pressure and indeed all factors controlling size, decidedly less than in the direction pressure is applied. This is an important factor in the design of dies.

<sup>21</sup> C. G. Goetzel, "The Influence of Processing Methods on . . . . Copper Powders," Columbia University, 1939, Plate 38.

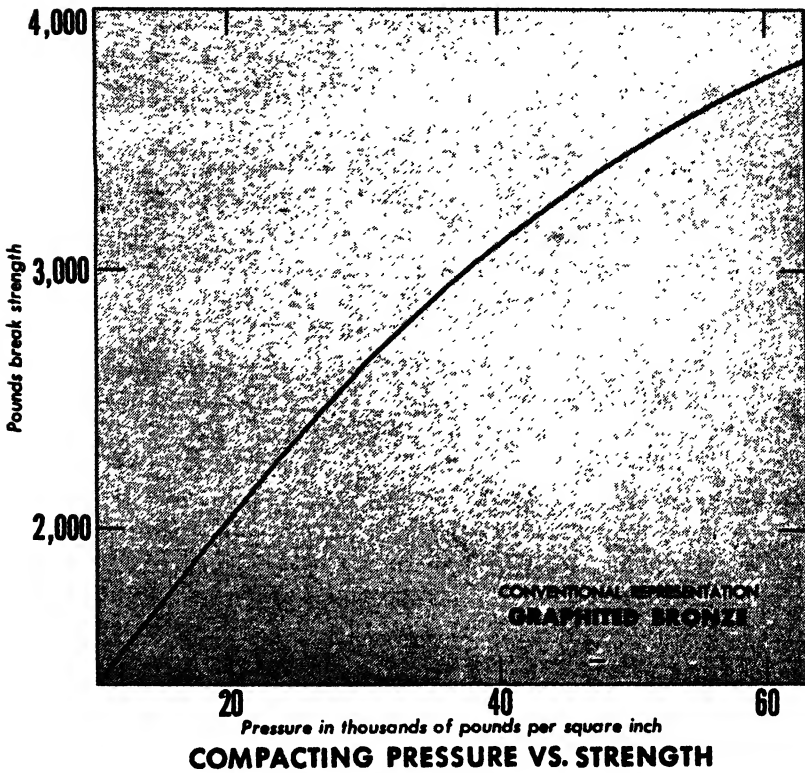
Graph No. 3 shows the relation between break strength and pressure. The break strength bears a very close relation to compression strength and fiber load, and apparently is a guide to tensile strength. In compositing this curve the author slightly modified the data found by the break tests in his classes, to conform more closely to data available in terms of compression strength and fiber tests, in order that the graph would represent the relation between the pressure and "strength" of the product. However, the degree of change from a curve based on break test alone is not marked, perhaps no more than would be required to smooth any curve made from limited data. The significance of this curve is to be found in its steepness as compared with the curves in Graphs 1 and 2. It is shown here that an increase in pressure continues to lend strength to the product even in the upper ranges. Taken in conjunction with the other graphs, they indicate that if stronger products are required, they may be made by modifying the die to compensate for greater growth, and employing greater pressure. There will be but little loss in porosity (the density curve has begun to flatten out), but a marked increase in strength.

Graph No. 4 demonstrates the relation of pressure to hardness. It is shown in *K* units, which are based on the B scale of a Rockwell Tester, equipped with a  $\frac{1}{4}$ -in. ball and 60-kilo weight. Note that in the low range the pressure has a marked influence on the hardness of the product, but that in the upper pressure range, much less is gained by using a greater pressure. Increasing the pressure from 45,000 lbs. per sq. in. by more than 30 per cent increases the hardness less than 10 per cent, and nearly all this is achieved by the addition of the first 5,000 lbs. But below 45,000 lbs. the curve is rather steep, and the addition of each 5,000 lbs. results in a marked increase in hardness.

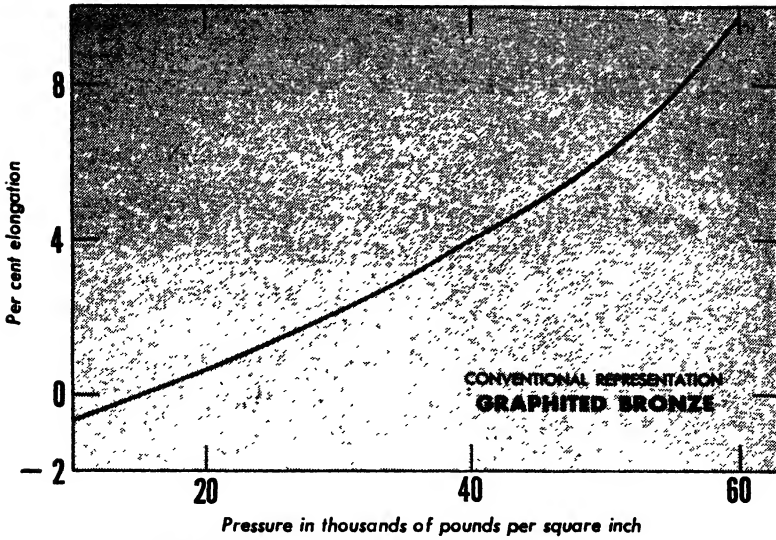
Graph No. 1



Graph No. 3

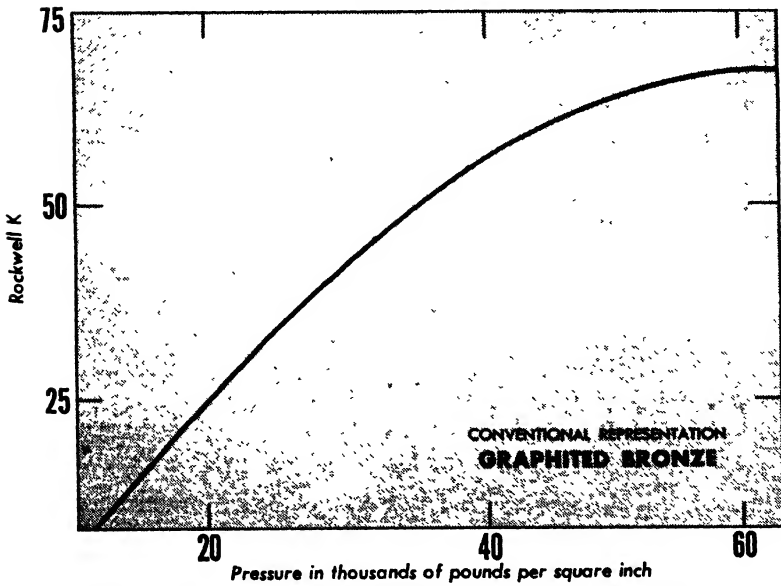


Graph No. 2



COMPACTING PRESSURE VS. ELONGATION

Graph No. 4



COMPACTING PRESSURE VS. HARDNESS

## Experiment 6. The Effect of Time of Sintering upon Bronze Product Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8 or 10-in. 100-mesh sieve  
Lubricant\*  
Die assembly  
Press  
Sintering boxes  
Charcoal  
A furnace operating at 1600° F.  
A ¼-in. (8 or 10-in.) coarse sieve  
Set of stamping dies, light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance with weights 50 to 0.05 grams  
A pan balance with weights 500 to 0.1 grams  
300 grams copper powder, "Standard"†  
30 grams tin powder, "Standard"  
6 grams graphite, "Standard"

*Procedure.* The procedure for producing the green cores follows exactly that used in Experiment 5, but it is necessary to mix sufficient powder for only two sets of cores. Both sets will be pressed at 40,000 lbs. per sq. in. No variation enters into this experiment except the heating cycle. One set of cores should be sintered for 60 minutes and one set for 80 minutes. Note that to this period of sintering must be added in each case the 20 minutes required to bring the cores to 1500° C. So the actual periods of heating are 80 and 100 minutes respectively.

### *Instructions to Squads.*

Squad B will sinter one group of cores 40 and one 50 minutes.

\* See footnote page 144.

† Standard powders are defined under Experiment 5, page 144.

Squad C, 20 and 30 minutes.

Squad D. 5 and 10 minutes.

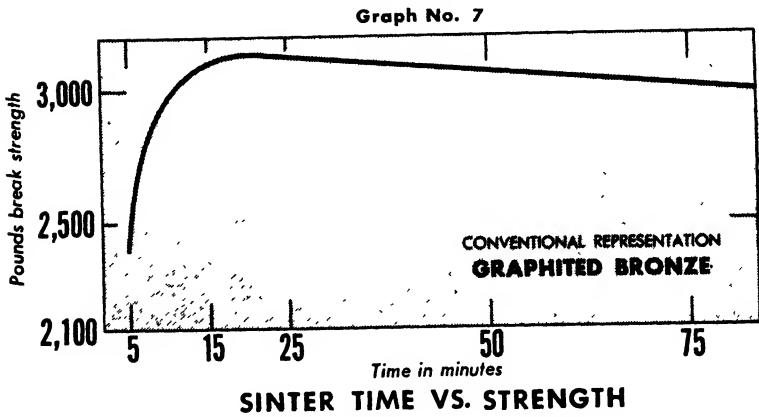
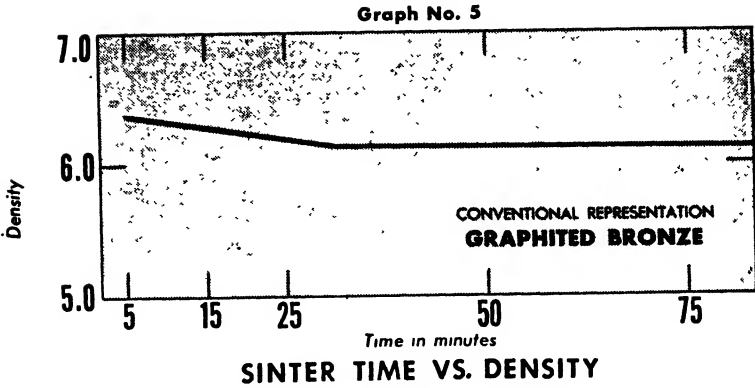
Results should be reported as for Experiment 5.

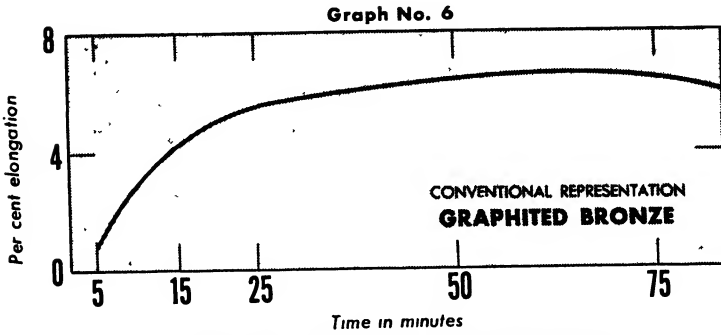
To handle this work conveniently, it is well to prepare both sets of cores, and place them in their respective properly marked boxes. With the furnace registering 1600° F., place the box of cores which is to be heated longest in the furnace first. Do this as quickly as possible and endeavor to avoid a material drop in temperature. Wait the interval between the two time periods called for, and then place the second box of cores in the furnace. For instance, Squad A will make its first loading and, after 20 minutes, its second loading. Squad D will load its second batch of cores 5 minutes after its first. By this system both boxes are removed from the furnace at the same time, and there is no drop of temperature during the critical period of heating, as would be the case if the two sets were loaded at once and one removed from the heat while the other was continuing its period of treatment.

Note that again three points are available to determine the curve in the graphic presentation. Although in this experiment only two sets of cores have been made, Experiment 5 has supplied the necessary data for a 15-minute sintering period. For instance, Squad A will report on the two sets it has made in this experiment in conjunction with the 40,000 lb. per sq. in. cores made in Experiment 5; as another example, Squad D will report on sintering period for 5, 10, and 15 minutes, also taking the data obtained in Experiment 5 for its point of reference. As in the previous experiment, when all squads have reported, the results will be correlated during the lecture. A graphic presentation can then be made covering the effect of sintering for periods from 5 to 80 minutes.

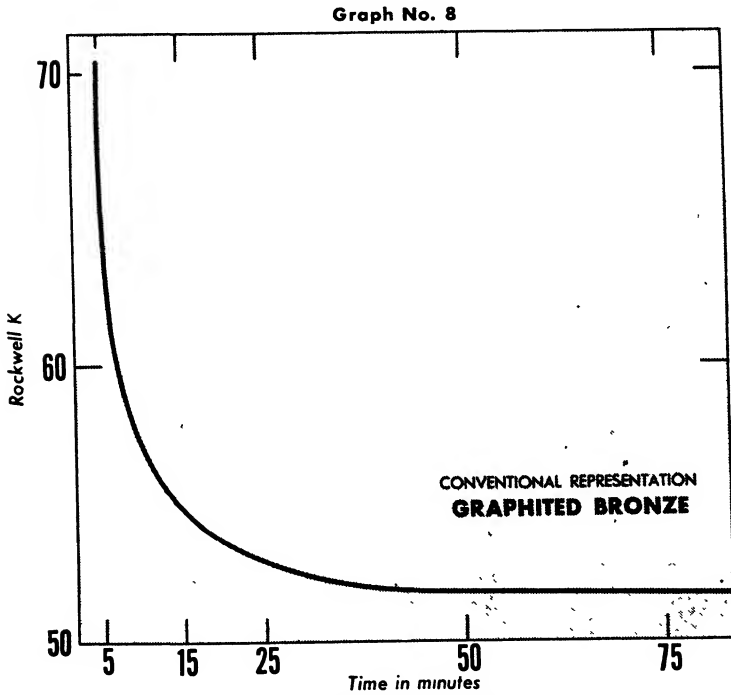
*Discussion.* A set of conventional graphs is given on pages 154 and 155. Inspection will show that the first few minutes of heating are critical. The density and the elongation show no appreciable change after 30 minutes, and the strength and hardness level off sharply before 20 minutes of heat treatment. Koehring<sup>32</sup> reports a slight drop from 5 to 20 minutes in the strength of such bronzes as this, and the strength levels as heating continues for 30 minutes.

<sup>32</sup> Koehring, "Powder Metallurgy 1940 M. I. T. Conference Papers", p. 89.





SINTER TIME VS. ELONGATION



SINTER TIME VS. HARDNESS



Now consider the conventional graphs. From Graphs Nos. 5 and 6, it would appear that some control of density and growth may be maintained by varying the time of heat treatment between 5 and 20 minutes; but if any attempt is made to reduce growth by heating for very short periods, this will reduce the strength of the product materially. (Study Graph No. 7.) Hardness (see Graph No. 8) will be improved noticeably for every minute's reduction in heating time, but a product heated less than 15 minutes is noticeably weak. Any period of heating that exceeds 30 minutes has only a very slight effect on density, elongation, strength, and hardness. This is very fortunate for the commercial process. It makes possible a rapid and continuous cycle of operations without exacting too close a control of sintering time.

The student should study these graphs with care to see how they relate to his laboratory work. He should note that if he heats longer than the specified time he will get higher expansion figures and lower densities than he should, and his hardness figures will be less marked, but somewhat low.\* Short heating will distort the results very sharply. *Never* heat too briefly. Try to heat the proper length of time.

\* Note to the instructor. If the class standardizes on 30-minute heat periods instead of 15, fewer discordant figures will be reported, since a slight departure from a 30-minute heat will have negligible results on physical characteristics. But this extends the total period of heating from 35 to 50 minutes, and unless three furnaces or a longer laboratory period are available, may seriously handicap the program. A little care by the students will make it possible to get good reports with the shorter heat, thus facilitating economy of time and equipment.

## Experiment 7. The Effect of Temperature of Sintering on Bronze Product Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8" or 10", 100 mesh sieve  
Lubricant\*  
The die assembly  
The press  
Sintering boxes  
Charcoal  
A furnace operating at various temperatures  
A  $\frac{1}{4}$ -in. (8 or 10-in.) coarse sieve  
Set of stamping dies, a light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance, with weights 50 to 0.05 grams  
A pan balance, with weights 500 to 0.1 grams  
300 grams copper powder, "Standard"†  
30 grams tin powder, "Standard"  
6 grams graphite, "Standard"

*Procedure.* Proceed as in Experiments 5 and 6, producing two sets of green cores at 40,000 lbs. per sq. in. The single variation from the exact procedure of Experiment 5 is that the furnace temperature will vary in each case from the temperature employed in that experiment. The sintering temperatures should be 1800° F. and 1700° F., respectively. Note that to insure that the sinter box reaches the proper temperature within 20 minutes, the furnace should have been running for some time 100° higher than the sinter temperature desired.

\* See footnote, page 144.

† Standard powders are defined under Experiment 5, page 144.

*Instructions to Squads.*

Squad B will sinter at 1600 and 1550° F.

Squad C will sinter at 1450 and 1400° F.

Squad D will sinter at 1300 and 1200° F.

In order to make a graphic report the data on Standard cores from Experiment 5 made at 40,000 lbs. per sq. in. will be used to give the third point on the curves required from each squad, namely a sinter temperature of 1500° C. The cores sintered at the high temperature may collapse or become so distorted that it is impossible to determine their growth or specific gravity. It frequently happens however, that though exact measurements are difficult, since a uniform diameter is lacking, the distortion is slight and a close approximation can be made. It has been observed that cores which actually exuded more than 10 per cent of the copper retained their shape so perfectly that the mold-flash or fin was still intact.<sup>33</sup> Care should be taken in removing the sinter boxes from the high-temperature oven. If they are not jarred too sharply, the cores may be retrieved in good shape. But at the temperature of deformation any external force, even a sharp shaking, may cause them to collapse. As in the case of other experiments, when all the reports have been made they will be correlated during the lecture period, and the effect of temperature on product characteristics may be graphically presented over a range of temperature from 1200 to 1800° F.

*Discussion.* The effect of the temperature at which products are sintered is much more critical than the effect of pressure of compacting, or the length of the sintering period. Here we have no flat curves extending over a wide range of variables. A change of as little as 5 per cent in the temperature employed results in a product of quite different characteristics. This provides a warning to the student to maintain the desired temperature within very narrow limits, and to the commercial producer to employ the best automatic temperature controls available, to check them frequently and make certain that their lag is very small. When the producer is making parts which permit very little tolerance, he may be justified in supplementing automatic controls with an operator whose sole duty is to keep the temperature of the sinter furnace at the proper

<sup>33</sup> W. J. Baëza, *Metals and Alloys*, 15, No. 2, 263-264, (1942).

point. A relatively small deviation will produce an entire batch of units wide of the specifications.

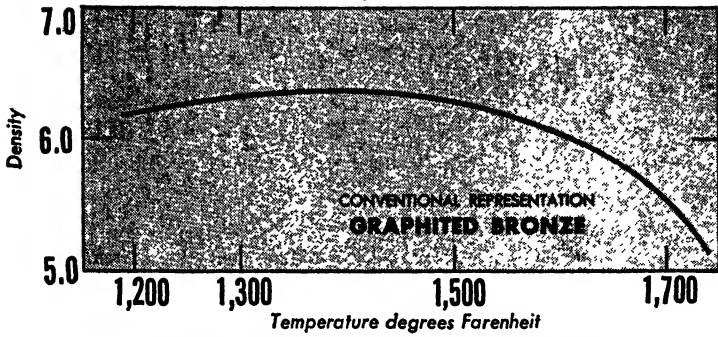
The effect of temperature on density (Graph No. 9) is less than on other characteristics, particularly temperatures below that desired. Between 1200 and 1500° F. there is but a slight increase in density, but temperatures above 1500° F. cause a very sharp drop in density. As the temperature approaches 1700° F. the product tends to distort, and at 1800° F. such distortion may become so great as to justify describing it as a collapse. As distortion increases, an accurate determination of density by the method employed becomes more difficult. A great many tests must be made and the results averaged before we can approximate figures which represent the density of these products heated at a high temperature; but though the experimental error is greater in the high-temperature range, it is not so great as to leave any doubt that these high temperatures give products of increasingly lower density. The conventional graph shows that 1500° F. is a very critical temperature as it relates to density.

Graph No. 10\* depicts the effect of temperature on elongation. It shows a very steep slope from 1300 to 1600° F., though not quite so steep between 1300 and 1450° F. At both end ranges the curve is fairly flat. The percentage growth remains nearly constant between 1200 and 1300° F., increase in elongation is about  $1\frac{1}{2}$  per cent, and between 1600 and 1800° F. the variation is less than  $\frac{1}{2}$  per cent in the range of 1700° F. But notice that at the range of standard operation, 1500° F., a slight variation has a marked effect: the elongation 50° F. above standard is greater than 5 per cent. Here again the critical effect of temperature is emphasized.

Turning now to Graph No. 11, the effect of temperature on strength is shown. In many instances the producer is more interested in this figure than in any other, for he has other means of

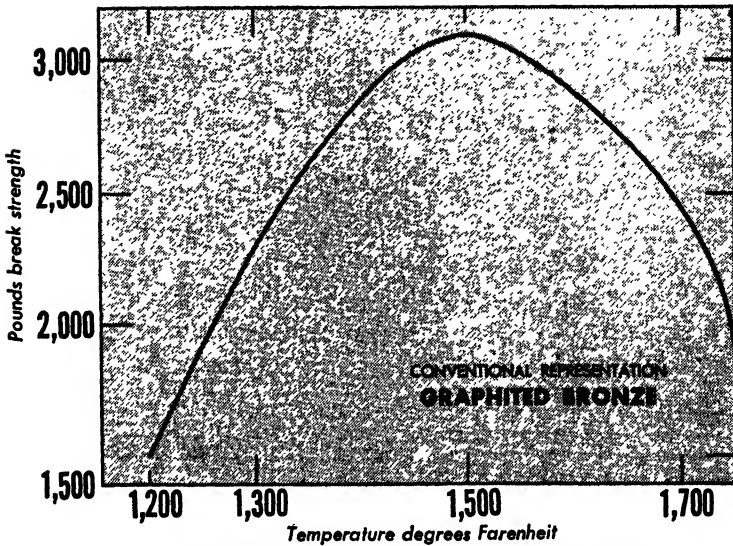
\* In making this graph reference has been almost exclusively to the data obtained by students and the author. The only published figures available which might properly be used are those of Koehring, but these cover a much smaller range from 1350 to 1550° F. His graph shows that 1475° F. is a very critical figure, since it is at this point, rather than at 1300° F., as shown in the attached graph, that his curve abruptly breaks from a flat line to a sharp slope. Inclusion of these data to form the conventional curve is very tempting, since it would result in a curve giving even greater significance to the 1500° point as a critical temperature. However, private sources show a much flatter curve than that of Koehring or the author, and the curve based exclusively on student data is chosen as the conventional curve, which appears to be a good compromise. Based on several hundred students' figures, it may be accepted as reasonably accurate.

Graph No. 9



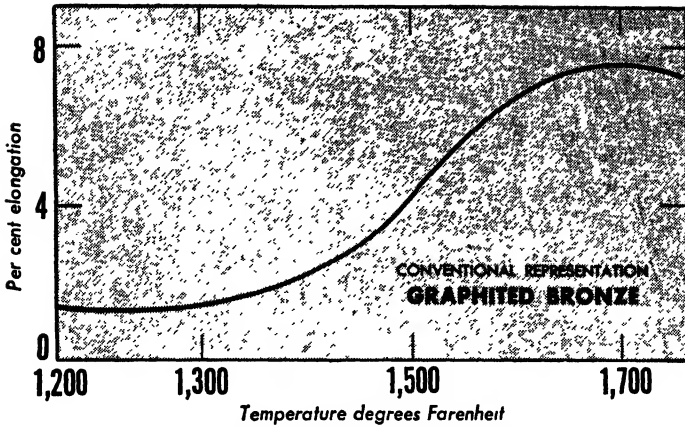
SINTER TEMPERATURE VS. DENSITY

Graph No. 11



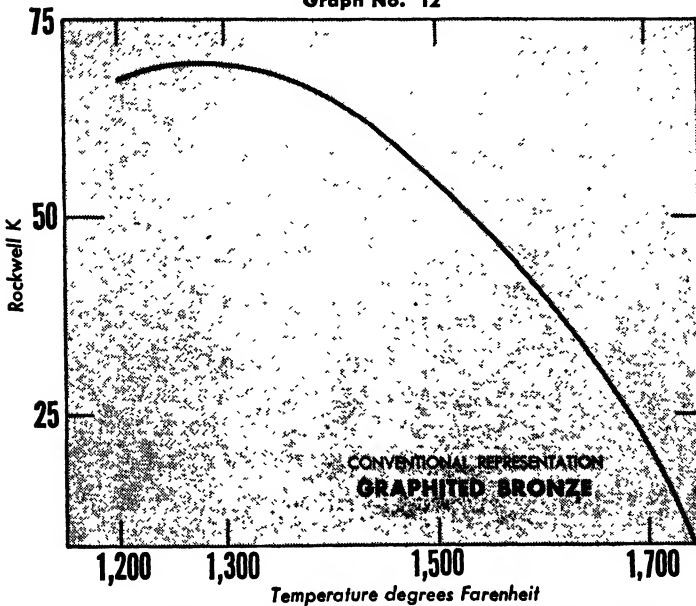
SINTER TEMPERATURE VS. STRENGTH

Graph No. 10



SINTER TEMPERATURE VS. ELONGATION

Graph No. 12



SINTER TEMPERATURE VS. HARDNESS

correcting or controlling growth and density, and great hardness is not expected of porous products, such as graphited bronzes; but mechanical strength to do the job—often considerable strength—is required. Strength may be greatly increased by subsequent repressing, but only at the cost of other characteristics. It is the producer's aim to make the strongest product possible under simple operating conditions. The curve illustrates how important sintering at the optimum temperature is. The slopes of this curve are sharp. A rapid increase in break-strength is obtained with every increase in temperature up to 1500° F. As the temperature increases beyond this point, the break strength drops sharply. To secure maximum strength the temperature must be closely controlled. For the mixture employed in these experiments, the critical temperature is clearly indicated to be 1500° F., but this may vary as much as 100° F. when other powders are employed. The important thing is to remember that there is a critical temperature for heating any mixture, and that heating below or above this will produce a weaker product. It is evident that when a producer has standardized on his raw material and percentage mixture, he should make careful study to determine the correct sintering temperature for his particular conditions. Unfortunately he cannot assume that 1500° F. is approximately right, for a comparatively slight variation from the optimum temperature will give products inferior in respect to strength.

In the final graph of this experiment, No. 12, we may observe that hardness decreases rapidly as temperature increases. Though there is a slight increase up to 1300° F. there is a drop of about 16 per cent as the temperature rises from 1350 to 1500° F. A further increase of 150 to 1650° F. causes a drop of over 36 per cent. Since however, to secure strong products it is necessary to operate closer to 1500 than to 1300° F., it is evident that hardness cannot be controlled by temperature. It is important to note that temperature must be closely controlled to secure concordant hardness figures, for a slight change here will be as marked as the effect on growth, density and strength.

## Experiment 8. Effect of Tin Variation on Bronze Product Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8- or 10-in., 100-mesh sieve  
Lubricant\*  
Die assembly  
Press  
Sintering boxes  
Charcoal  
A furnace operating at 1600° F.  
A ¼-in. (8- or 10-in.) coarse sieve  
Set of stamping dies, a light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance with weights 50 to 0.05 grams  
A pan balance with weights 500 to 0.1 grams  
300 grams copper powder, "Standard"†  
60 grams tin powder, "Standard"  
6 grams graphite, "Standard"

*Procedure.* Follow that of Experiment 5 and make two sets of cores, but in this case the tin content of each set varies. One set should be made with 2 per cent tin and another with 8 per cent tin, to each of which add 2 per cent graphite.

### *Instructions to Squads.*

Squad B will use 4 per cent and 12 per cent tin respectively.

Squad C, 6 per cent and 16 per cent tin;

Squad D, 14 per cent and 18 per cent tin.

In all cases add 2 per cent graphite.

For the third point required for a graphic presentation, the data from the tests on the standard cores of Experiment 5 are

\* See footnote, page 144.

† Standard powders are defined under Experiment 5, page 144.

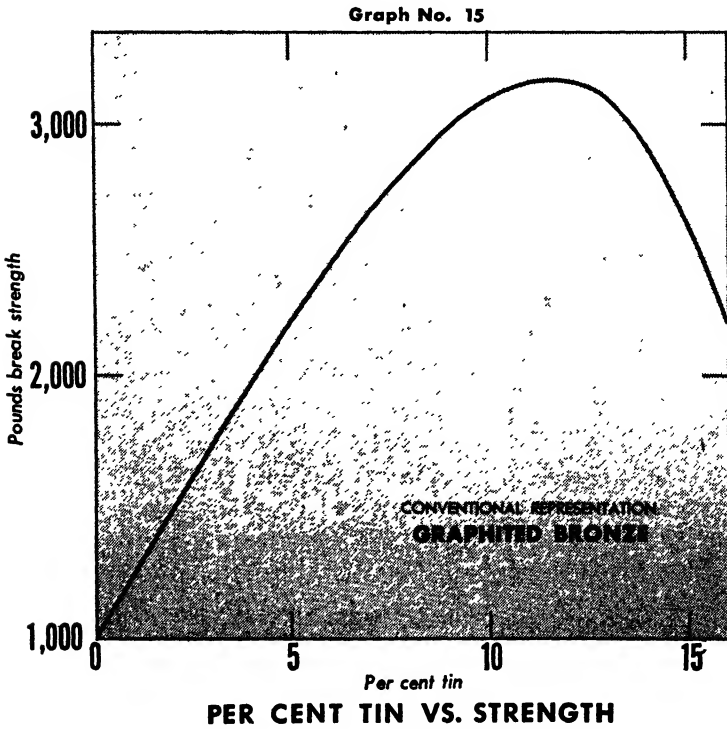
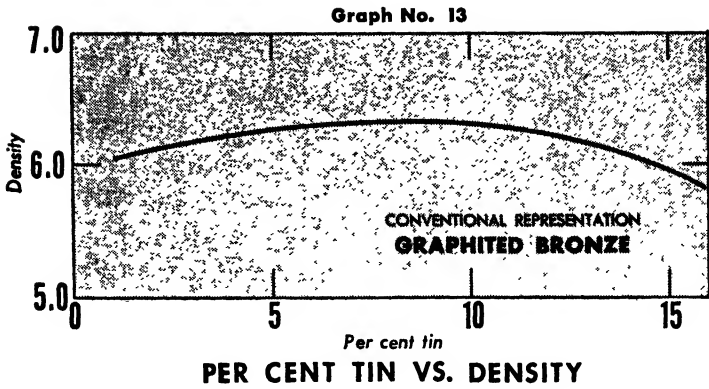


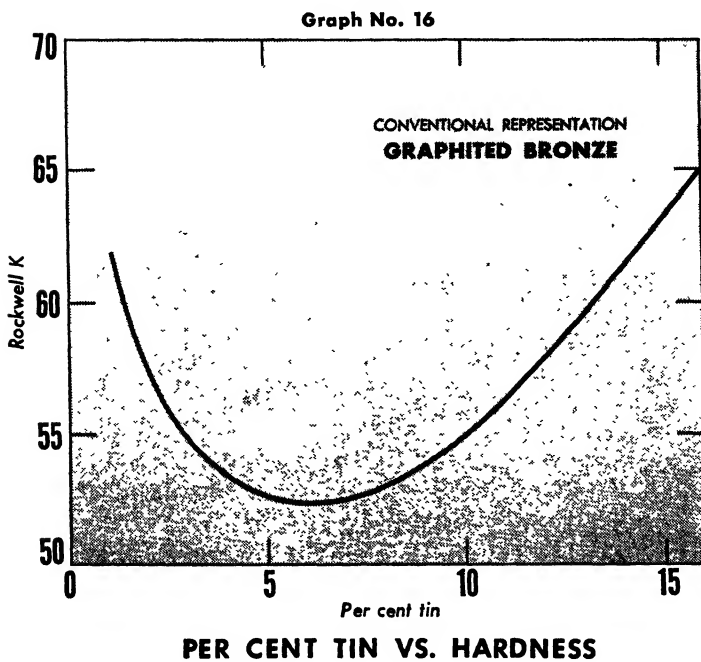
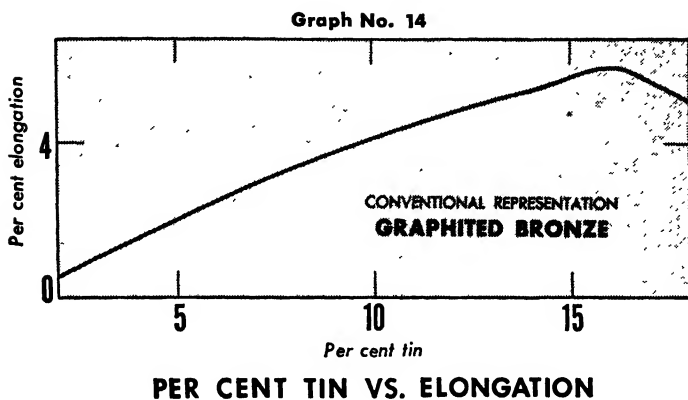
incorporated into the curve. When the reports of all squads have been made, the results will be correlated during the lecture. A graphic presentation can be made then covering the effect of varying the tin content of a graphited bronze from 2 to 18 per cent.

*Discussion.* Graph No. 13 indicates the effect of varying the tin content upon the density of the product. Examination will show that changing the percentage of tin has but a slight influence on the density; the entire range of variation causes but a few points difference in the density. The manufacturer may dismiss considering tin in relation to density. On the other hand, the percentage of tin has a marked effect on the growth. Graph No. 14 shows the relation between elongation and tin content. Note that there is a negligible growth, or even a slight shrinkage in the lower range, but that growth increases in an almost straight line of sharp slope to an apex of almost 6 per cent increase in elongation when about 15 per cent of tin is employed; thereafter there is a decrease in the effect of adding more tin. An unusual phenomenon must be occurring here, for we expect in general that an elongation curve will approximately mirror a density curve. (See, for example, the density and elongation curves depicting the effect of graphite, Graph Nos. 17 and 18.) Koehring,<sup>32</sup> who shows the effect of changing tin over the short range from 8 to 12 per cent, depicts a curve of a slightly lower order and of increasing steepness; the curve of one commercial company, however, is of a higher order, closely paralleling the conventional curve from its beginning at 5 to 10 per cent, and then becoming much steeper. Both this and the conventional curves show that while growth continues as the tin content increases above 12 per cent, it again falls off so that it is the same at both 12 and 18 per cent tin. In other words, the product will increase by the same amount of growth whether the tin content is 12 or 18 per cent. But observe that strict control of tin content is as necessary in this as in lower ranges. A slight variation from the desired proportion of tin will cause a marked variation in growth, whether this proportion be 4 or 18 per cent. It is not easy to account for the complete lack of relation between the effect of tin on density and its effect on growth. A clue lies in the suggestion that true alloys are formed, but an investigation might yield some interesting data.

Turning now to a consideration of Graph No. 15, we may examine the effect of varying the tin content on the strength of the product. This shows that the increase in strength follows a sharp slope to just below 11 per cent and that the addition of more tin causes a sharp drop in strength. Here is another suggestion that a true alloy is formed at about 10 per cent (gun metal is 90 per cent copper, 10 per cent tin; some work of the author<sup>33</sup> discloses an alpha-bronze produced at this point), and that above and below 10 per cent there is free tin or free copper in the product. Neither of these is as strong as the bronze, and both result in a weaker product. Note that the compound,  $\text{Cu}_{16}\text{Sn}$ , corresponding to 11 per cent tin, is well known. Some curves which have been brought to the attention of the writer are even sharper than the conventional, so that it would appear that many commercial powders must be very accurately compounded to give products of maximum strength. The conventional curve allows some latitude; there is little difference in strength between a compound of 10 per cent tin and one of 12. The strength appears to be maximum with the tin proportioned as in the molecule  $\text{Cu}_{16}\text{Sn}$ .

Graph No. 16 shows the relation between percentage of tin and hardness. The addition of small proportions of tin reduces hardness. As the amount exceeds 5 per cent, the hardness increases, and the slope of the curve becomes sharp above 12 per cent. Though copper is harder than tin, bronze manufacturers are familiar with the fact that the addition of 5 per cent of tin to copper nearly doubles the hardness. Increase in hardness continues until 20 per cent has been added. Thereafter, the addition of more tin has no appreciable effect until 35 per cent is present, when any further increase in the proportion of tin makes the bronze much softer. It would appear that the manufacturer need not be too concerned with his tin-copper proportions as they affect hardness; a deficiency of tin in the lower ranges will not make a serious difference, and an excess will improve hardness. Careless use of tin will increase costs, and will seriously affect strength and growth, but will not severely influence density or hardness.





## Experiment 9. Effect of Graphite Variation on Bronze Product Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8- or 10-in., 100-mesh sieve  
Lubricant\*  
Die assembly  
Press  
Sintering boxes  
Charcoal  
A furnace operating at 1600° F.  
A ¼-in. (8- or 10-in.) coarse sieve  
Set of stamping dies, a light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance with weights 50 to 0.05 grams  
A pan balance with weights 500 to 0.1 grams  
300 grams copper powder, "Standard"†  
30 grams tin powder, "Standard"  
45 grams graphite, "Standard"

*Procedure.* Proceed as in Experiment 5, the only variation from this exact procedure being the quantity of graphite employed in the mix. It will be necessary invariably to lubricate the die when bronzes with less than 1.9 per cent of graphite are molded. Be sure not to employ excessive Sterotex, as during compression some of it may be introduced into the body of the product and thus increase the carbon content, vitiating the result. One set of cores should be made with *no* graphite and one set with six parts of graphite.

### *Instructions to Squads.*

Squad B will use 1.0 and 3.0 parts of graphite;

\* See footnote, page 144.

† Standard powders are defined under Experiment 5, page 144.

Squad C will use 1.5 and 4.0 parts of graphite;

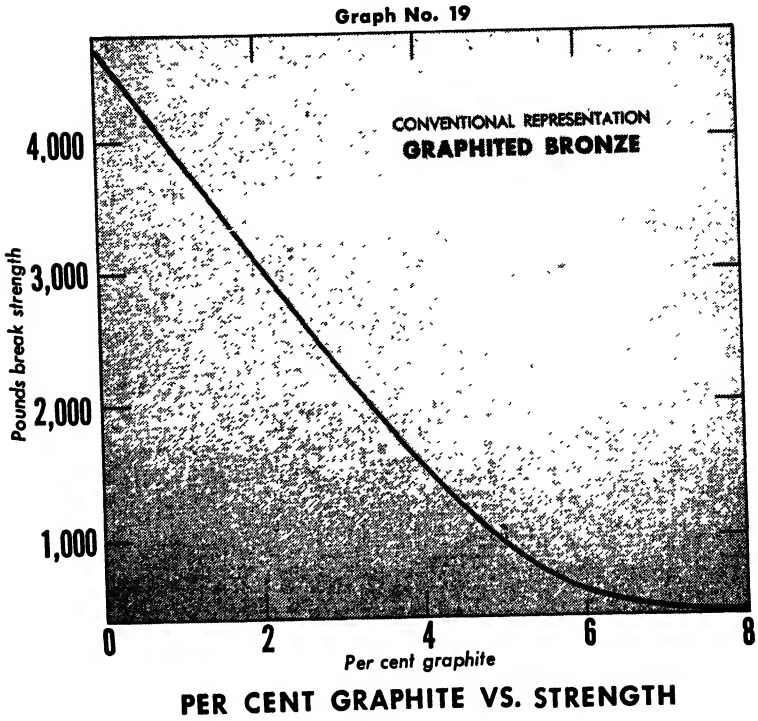
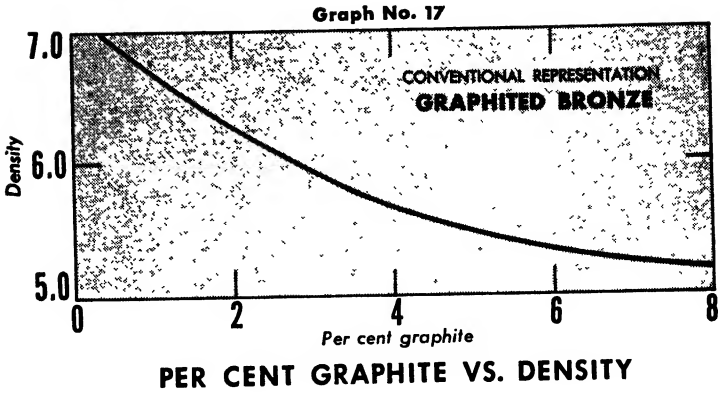
Squad D will use 2.5 and 8.0 parts of graphite.

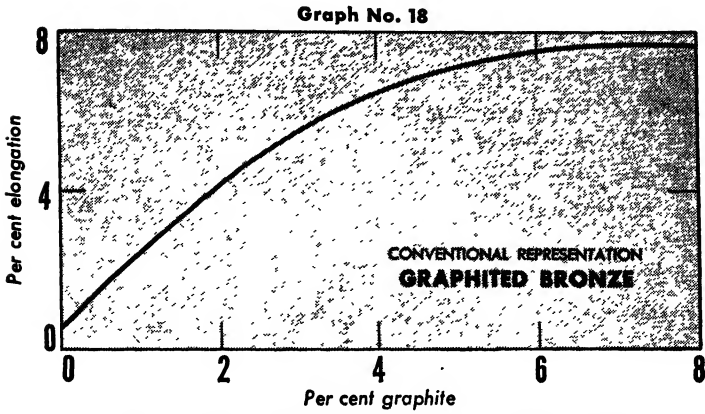
Graphic presentations may be made by borrowing the data from Experiment 5, on the Standard core, thus giving the third point at the two parts point of graphite necessary to depict a curve. When the results of this experiment have been reported by all squads, the reports will be correlated during the lecture. A graphic presentation can then be made showing the effect of graphite variation over the range from 0 to 8 parts of graphite.

*Discussion.* The graphs which depict the effect of graphite content upon bronze products show that even small differences have a marked effect upon physical characteristics. Essentially all the curves may be considered straight lines, although they actually have some curvature, which is not enough to affect the general impression. Graph No. 17 shows the effect of graphite on density. The density decreases rapidly as graphite is increased. Graph 18 shows its effect on growth. The elongation curve is a mirror of the density curve; as the graphite increases the growth increases.

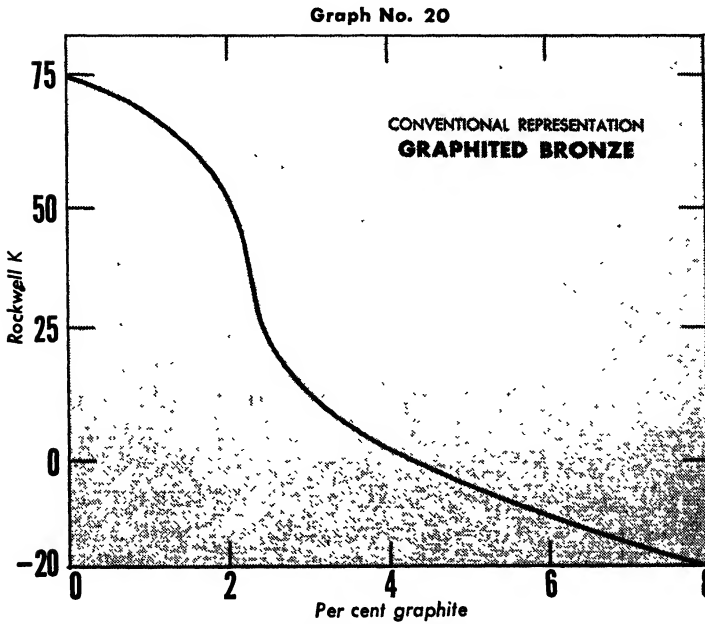
Graph No. 19 shows that the effect of graphite on strength closely parallels the density curve, and that as the graphite increases, the break-strength drops sharply.

Graph No. 20, which depicts hardness plotted against graphite, is more irregular than the other curves of this set, but still shows that hardness decreases with each increase in graphite. The effect is less marked in the low range, beginning at zero, but becomes sharp between 2 and 3 per cent, and from there on, as the percentage of graphite increases, the hardness continues to decrease rapidly. It should be noted that the actual figures for hardness as the graphite exceeds 4 per cent fall in that range of the Rockwell tester which is least accurate. Several of the figures are negative, and are best considered as an indication that the product tested is very soft, rather than that it has a definite hardness of a stated negative numerical value. Projection of the curve into this region is supported by student reports, but must be considered with such reservations as are suggested by the limitations of the test employed.





**PER CENT GRAPHITE VS. ELONGATION**



**PER CENT GRAPHITE VS. HARDNESS**



## Experiment 10. Effect of Particle Size Variation on Bronze Product Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8- or 10-in., 100-mesh sieve  
Lubricant\*  
Die assembly  
Press  
Sintering boxes  
Charcoal  
A furnace operating at 1600° F.  
A ¼-in. (8- or 10-in.) coarse sieve  
Set of stamping dies, a light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance with weights 50 to 0.05 grams  
A pan balance with weights 500 to 0.1 grams  
500 grams copper powder, 100% through 100 mesh,  
("Standard" copper), 50% through 325 mesh  
500 grams copper powder, 100% through 325 mesh,  
(Fine copper)  
500 grams copper powder, 100% through 100 mesh,  
(Coarse copper), 50% through 200 mesh  
50 grams tin, "Standard"†  
10 grams graphite, "Standard"

*Procedure.* The procedure follows precisely that of Experiment 5 in which 90 parts of copper, 10 parts of tin and 2 parts of graphite make up the composition of the mixture. But the copper powder in this case is to be varied in particle size distribution. In order to accomplish this variation, instead of using the Standard copper

\* See footnote, page 144.

† Standard powders are defined under Experiment 5, page 144.

powder, for one set of cores, substitute 100 per cent FINE powder for the STANDARD. For the second set of cores, to 80 per cent of STANDARD powder add 20 per cent of COARSE powder. Mix thoroughly and employ this new powder in the proper proportions for the bronze mix.

*Instructions to Squads.*

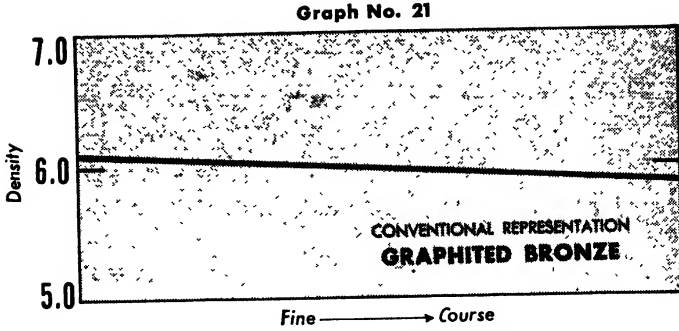
Squad B will make a mix of 20 per cent STANDARD and 80 per cent FINE for one set of cores and for the other 50 per cent STANDARD and 50 per cent COARSE.

Squad C will make a mix of 50 per cent STANDARD and 50 per cent FINE for one set of cores and for the other 20 per cent STANDARD and 80 per cent COARSE.

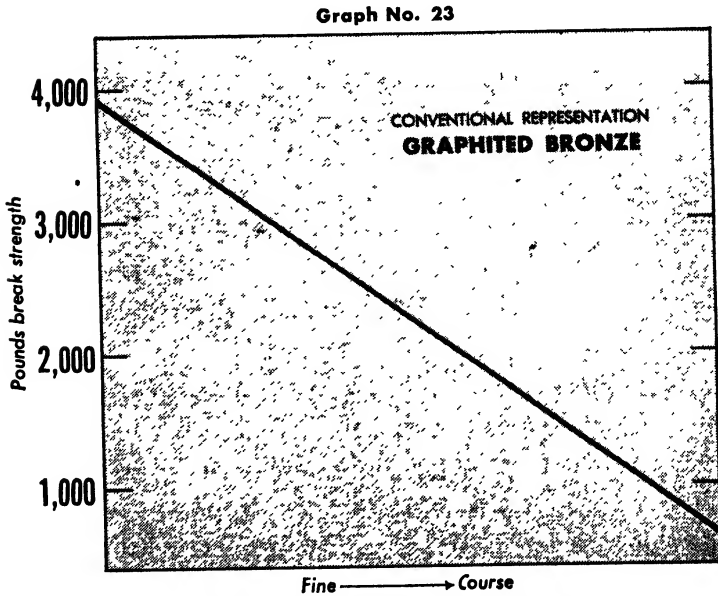
Squad D will make a mix of 80 per cent STANDARD and 20 per cent FINE for one set of cores and for the other will use 100 per cent COARSE.

A study of these figures will show that we have covered the range of size distribution from an extremely fine to an extremely coarse powder. Each squad will be able to make a rough curve showing the effect of increasing the percentage of fine and coarse powder in a copper mix, and for their third point required for graphic presentation they will be able to use the standard data from Experiment 5. When reports of all squads have been made the data will be correlated during the lecture. A graphic presentation can then be made covering the effect of variation in particle size on physical characteristics.

*Discussion.* The curve drawn from the data obtained from this experiment must be rather rough as the data is incomplete. Any attempt to set the intervals on the  $x$  axis can only be a guess, since no common unit of fineness has been worked out, and no particle size distribution of each test mixture is available. The nature of the curve, however, is such that, with the conventional graphs as a guide, corrections may be made on the  $x$  axis close enough to justify an attempt to profit by the experience derived from the work. A short cut to more accurate work, which is not too burdensome to be performed if laboratory time is available, is to determine the percentage of each copper mix which will pass the 325-mesh screen. The test employing only coarse copper will show the least, and that employing only fine powder will show 100 per cent through 325.

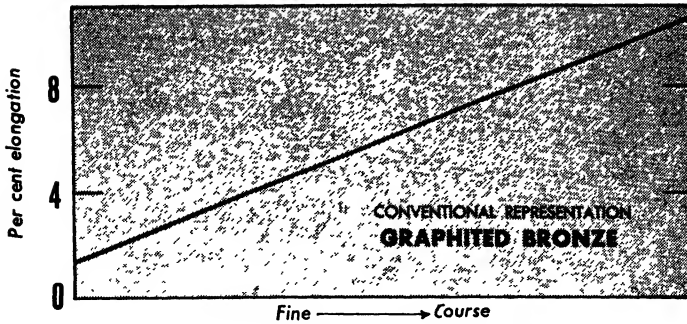


**PARTICLE SIZE VS. DENSITY**



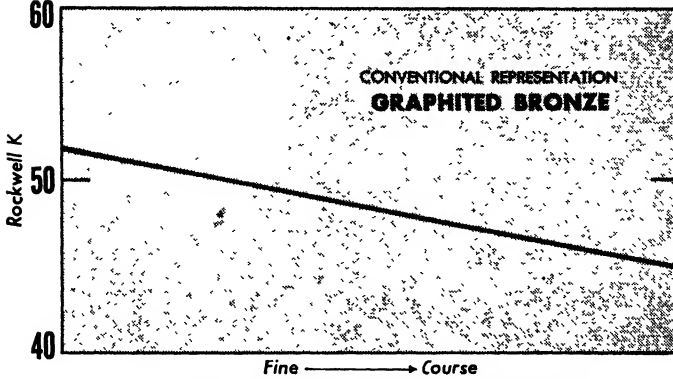
**PARTICLE SIZE VS. STRENGTH**

Graph No. 22



**PARTICLE SIZE VS. ELONGATION**

Graph No. 24



**PARTICLE SIZE VS. HARDNESS**

All the other mixes will show varying amounts of the very small particles, and the quantity may be used as a rough measure of fineness, giving a unit division for the  $x$  axis that will make it possible to plot physical characteristics against this measure with close approximation.

Graph No. 21, which shows the effect of particle size on density, indicates that the effect is very slight. Within the limits of useful powders the density decreases slightly as the material becomes coarser. The effect of size on growth is much more marked. The slope of the curve in Graph No. 22, depicting the effect of size on elongation, is much steeper. Growth increases rapidly as coarseness increases.

Conversely the strength decreases. Graph No. 23 indicates the effect of size on break point. The slope of the straight line is steep. Very fine powder gives much stronger products than coarse powder. The same effect is observed on hardness, but not in so great a degree. The slope of the straight line curve of Graph No. 24, which plots size against Rockwell K, is not very steep. It should be noticed that another factor is illustrated in these figures mixing the standard with other powders sharply changes the distribution of particle size. The effect is particularly evident in the density curve and the hardness curve; and at no point do these curves pass through a point as high as would be found if only the Standard powder had been used.

## Experiment 11. The Effect of Volatile Ingredients on Bronze Product Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8- or 10-in., 100-mesh sieve  
Lubricant\*  
250-cc. cylinder  
25-cc. cylinder  
Die assembly  
Press  
Sintering boxes  
Charcoal  
A furnace operating at 1600° F.  
A  $\frac{1}{4}$ -in. (8- or 10-in.) coarse sieve  
Set of stamping dies, a light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance with weights 50 to 0.05 grams  
A pan balance with weights 500 to 0.1 grams  
700 grams copper powder, "Standard"†  
70 grams tin powder, "Standard"  
15 grams graphite, "Standard"  
5-in. mortar and pestle  
Glass plate  
25 grams salicylic acid  
250 cc. 10% solution of camphor in acetone  
200 cc. acetone

*Procedure.* Each squad will make four sets of cores. In two sets a quantity of camphor is added and in the other sets the same quantity of salicylic acid. To add the camphor, the necessary amount of camphor solution is calculated; and after the copper, tin and graphite have been roughly mixed and screened, it is heaped on

\* See footnote p. 144.

† Standard powders are defined under Experiment 5, page 144.

a glass plate. Separate the heap in the center with the large spatula so as to leave a ring of the powder-mix around a clear space in the center of the glass plate. Pour the solution onto the center of the plate in successive small quantities, each time incorporating enough powder to make a thin paste; and proceed until all the camphor has been added. The container used to measure the camphor solution should be washed with successive small quantities of acetone and these washes in turn incorporated into the powder mix. Sufficient acetone should be employed to produce a fairly fluid paste, which should be worked on the glass plate much as an unguent is worked by a pharmacist. The working should continue until most of the acetone has evaporated; the powder should then be permitted to dry for perhaps half an hour, and then worked until it has reached a stage of dryness so complete that it is practically indistinguishable from a mix which has never been wet with acetone.

If the experiment cannot be completed in the same laboratory period in which it is begun, unpressed powder or green cores should be kept in a tightly sealed bottle to prevent the loss of camphor between laboratory periods.

The salicylic acid for the second set of cores should be weighed on the bulk balance to one decimal place and transferred to the mortar. It should be ground until all of it will pass through a 100-mesh sieve. After screening it may all be returned to the mortar. Small quantities of the roughly mixed and screened copper mix should be added successively and *lightly* ground with the salicylic acid. Use as little pressure after the copper mix is added as possible to avoid metallizing the copper. Each portion should be thoroughly mixed by grinding before the next portion is added. Continue adding copper mix and grinding until the whole has been incorporated in a uniform mixture with the salicylic acid. Thereafter the procedure exactly follows that required in Experiment 5 to produce cores under a pressure of 40,000 lbs. per sq. in.

One set of cores should be made with 0.5 per cent camphor, and another with 0.5 per cent salicylic acid. Make another set with 2.5 per cent camphor and the fourth with 2.5 per cent salicylic acid.

*Instructions to Squads.*

Squad B employs 0.3 per cent camphor and 0.3 per cent salicylic in one pair; in its other two sets it uses 2 per cent camphor in one, and 2 per cent salicylic in the other.

Squad C employs 0.8 per cent camphor and 0.8 per cent salicylic in one pair; and in the other two sets it uses 1.25 per cent camphor in one, and 1.25 per cent salicylic in the other.

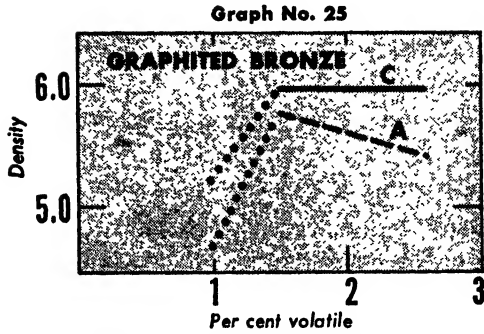
Squad D uses 1.0 per cent camphor and 1.0 per cent salicylic in one pair; in the other two sets it uses 1.5 per cent camphor in one, and 1.5 per cent salicylic in the other.

The data obtained in this experiment are insufficient to depict graphically. The student should note the difference in the physical qualities of the cores made with the same quantity of different types of volatile ingredients.

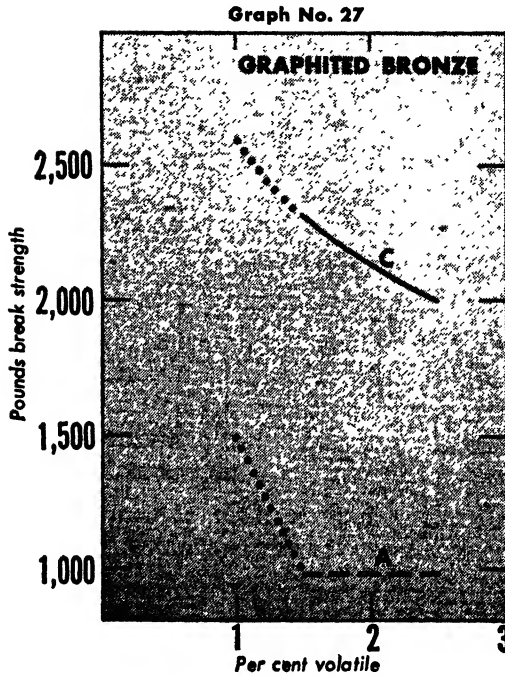
When all reports have been made, sufficient data will be available to correlate during the lecture, and a graphic presentation will be made of the effect of varying the quantity of volatile ingredient incorporated into a graphited bronze powder mix and to contrast the effect of different volatiles.

*Discussion.* Graph Nos. 25 to 28 show the curves for each volatile in the same graph, for their chief value lies in depicting the quite different effect the two volatiles have on the physical character of the product into which they have been incorporated. The student should realize that there are a tremendous number of other ingredients which might be employed to effect porosity, and the two shown illustrate that no generalization for all volatiles can be drawn from the data covering any particular one. Unfortunately, the range covered by the experiments on which these graphs are based is a narrow one, and apparently begins above the critical amount which might affect physical qualities, by slight change in proportions. From  $1\frac{1}{2}$  to 3 per cent, the curves are either flat straight lines, or straight lines of quite small slope. The experiments outlined above concentrate more attention on the range below  $1\frac{1}{2}$  per cent. The writer has insufficient data to permit drawing the curve through this range, but a few experiments have been reported to him in which 1 per cent or slightly less volatile was employed. These data are insufficient to permit any positive conclusions, but the dotted lines on the graph show the suggested extension of the better established points on the curves.

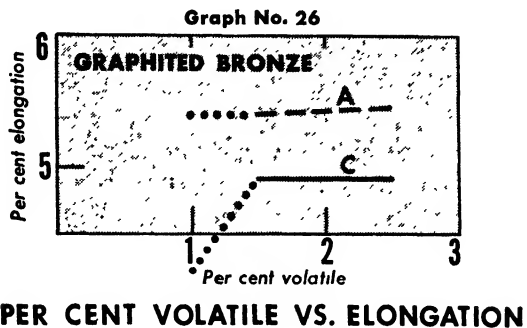




PER CENT VOLATILE VS. DENSITY

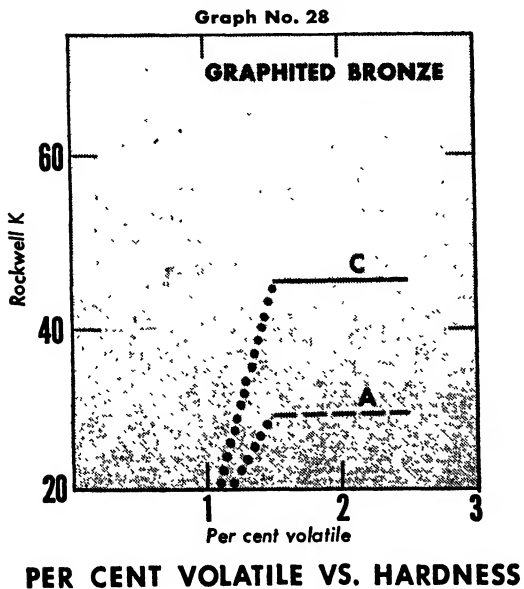


PER CENT VOLATILE VS. STRENGTH



LEGEND

- C ————— Camphor
- A - - - - - Salicylic Acid
- Indicated



Graph No. 25 illustrates the effect of the volatile on density. Note particularly that the camphor has reduced the density less than the same quantity of acid. Observe also that additional acid causes a slight decrease in density, but additional camphor has no further effect. Graph No. 26 shows the effect upon growth of adding volatiles. The curves showing per cent elongation plotted against quantities of volatile are nearly straight lines in both cases. Here, as might be expected by inference from the density curves, the camphor causes less elongation than the salicylic acid. Graph No. 27 shows the effect of volatile on the strength of the product. Here the significance of selecting the proper volatile is emphasized. Though in both cases the strength is reduced, the effect of salicylic acid is drastic. It has reduced the break to less than one-third of a similar product which contained no salicylic acid. Contrasting with the camphor, quantities greater than 1.5 per cent do not further decrease the strength. The curve is a flat straight line over the range covered. There is a slope to the straight line curve depicting the effect of adding camphor, and though the effect of adding even a small quantity is marked, it is in a different category from the effect of adding a similar quantity of acid. The hardness curves, shown in Graph No. 28 are again straight lines without slope throughout the range of volatile added. As in the case of break strength, the camphor has had less effect on the original hardness than the salicylic acid. The acid reduces the hardness to a point where the usefulness of the product might be seriously impaired. Note too that it appears that smaller quantities have even a more marked effect, but all the dotted lines on these graphs depict but lightly held tentative conclusions.

## Experiment 12. The Effect of Zinc Variation on Graphited Brass Product Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8- or 10-in., 100-mesh sieve  
Lubricant\*  
Die assembly  
Press  
Sintering boxes  
Charcoal  
A furnace operating at 1600° F.  
A  $\frac{1}{4}$ -in. (8- or 10-in.) coarse sieve  
Set of stamping dies, a light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance with weights 50 to 0.05 grams  
A pan balance with weights 500 to 0.1 grams  
500 grams copper powder, "Standard"†  
350 grams zinc powder (100% through 100-mesh),  
"Standard"  
10 grams graphite, "Standard"

*Procedure.* The procedure exactly follows that employed for the production of standard bronze cores at 40,000 lbs. per sq. in., except that zinc powder is used in place of tin.

Make three sets of cores, one set with 10 parts of zinc, one with 30 parts of zinc and one with 35 parts of zinc.

### *Instructions to Squads.*

All squads will make one set containing 30 parts of zinc. In addition,

Squad B will make one set with 15 parts and one with 40 parts zinc.

\* See footnote page 144.

† Standard powders are defined under Experiment 5, page 144.

Squad C will make one set with 20 parts and one with 33 parts zinc.

Squad D will make one set with 25 parts and one with 37 parts zinc.

Determine the change in density, volume before and after sintering, and the strength and hardness of the finished product.

When reports of all squads have been made, the data will be correlated during the lecture. A graphic presentation can then be made covering the effect of variation in zinc content on the characteristics of graphited brass products.

*Discussion.* Note that Graphs No. 25 to 40 are NOT conventional graphs. You will recall that we have defined a conventional graph as a graphic presentation of the over-all relationship between a single variable in the cycle of powder metallurgy production, and a physical characteristic of the product. It is a conventional rather than an exact graph because it does not represent the figures obtained by one operator able to control rigidly all factors, including that under examination, and able to report his results in universal terms; but it attempts to portray the results of many different operators, who, investigating the effect of one variable, rigidly controlled other factors, but made no attempt to see that these other factors were controlled by conditions common to all investigators. Furthermore, the results of the several independent investigations were not reported in common units. The conventional graph therefore is a symbol, a convention, a formal figure, representing an over-all effect, the dominant theme, implicit and explicit from many sources in many terms.

It might be said that there is greater accuracy in the report of a single, well-controlled experiment, carefully confirmed, than in the conventional graph; and for the particular experiment that is certainly true. For most physico-chemical reports it is all that should be admissible evidence. But these conventional curves are not offered as evidence that a certain phenomenon occurs in an exact way. They are offered as a representation of the predominating effect, an attempt to formulate a generalization, where there is sufficient evidence to justify drawing a tentative generalization. It is like saying that a photograph of a battlefield is a more accurate picture of warfare than the painting of a battlefield by an artist.

The photograph is certainly more accurate of that battlefield at that moment, but if the artist is worth his salt, his picture will be a better indication of what goes on in mortal combat.

But just as the artist who has seen but one battlefield and heard nothing of others cannot paint a picture of battlefields, but must content himself with portraying only that one battle, probably less accurately than the photographer, so the scientist can draw no generalization, can offer no formal or conventional curve, based on a limited observation.

The conventional curves are tentative generalizations. Subsequent work may prove that important modifications are required, but it is unlikely that they will not continue to represent the over-all relationships they depict. They are not accurate in the sense that an exact point on the curve represents the result of any one experiment; but in the sense that it represents a series of relationships it is more dependable than an exact curve based on one set of experiments.

Graphs No. 29 to 32 are based on a set of experiments which have been duplicated many times. They present the results of those particular experiments, nothing more. They MAY have the same value as the conventional graphs, but the student is warned not to generalize from them with the same assurance he may feel in generalizing from the conventional graphs.

Graph No. 29 shows the effect of increasing quantities of zinc on the density of the resulting brass. It shows that there is a wide range of effect, a sharp reduction of density in a straight line slope as the brass content increases from 10 to 30 per cent. Thereafter the curve flattens abruptly. Density is but slightly affected as the zinc content increases from 30 to 40 per cent. Contrast this with the effect of varying the tin content in a bronze (Graph No. 13), where the slope of the curve is slight, and the addition of small quantities INCREASE the density, and further quantities decrease it markedly.

Graph No. 30 depicts the effect of increasing zinc content on the growth. Over the entire range from 10 to 40 per cent the per cent elongation increases abruptly, though the greatest effect is in the range from 10 per cent, where a slight shrinkage occurs, to 30 per cent where elongation exceeds 10 per cent. At 40 per cent the elongation has increased nearly 2 per cent more. The effect of small quantities

is not too dissimilar to the effect of increasing the tin content of a bronze (See Graph No. 14). Slight additions act similarly, but note: as the tin exceeds 15 per cent, elongation does not further increase, but decreases.

Graph No. 31 shows the effect of increasing quantities of zinc on the strength of brass. The slope of the curve in the low ranges is very abrupt; beginning at about 25 per cent it flattens abruptly and there is practically no further decrease in strength. We say practically because, whether a product has a break strength of a few hundred pounds or 50, it has not strength enough for most practical applications. Quite a different curve this, from the conventional curve for bronze (Graph 15), which shows that the proper proportion of tin produces a product of maximum strength, less or more than this amount resulting in a weaker bronze.

Graph No. 32 depicts the effect of increasing quantities of zinc on the hardness of brass. The curve is not unlike that shown in Graph No. 31, but at no point is it so abrupt. As the quantity of zinc is increased, the hardness of the product decreases, the effect being fairly proportional until 25 per cent has been added; thereafter there is little further loss, the curve flattening out. This curve apparently bears no relationship to that depicting the effect of tin on the hardness of bronze. Compare with Graph No. 16.

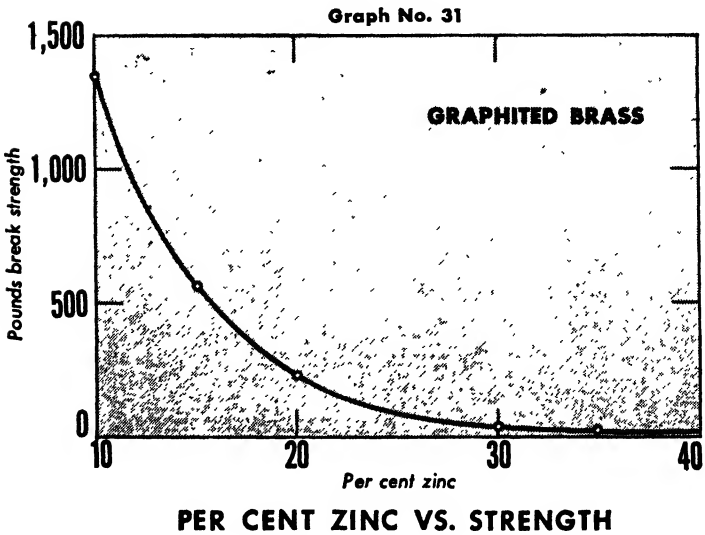
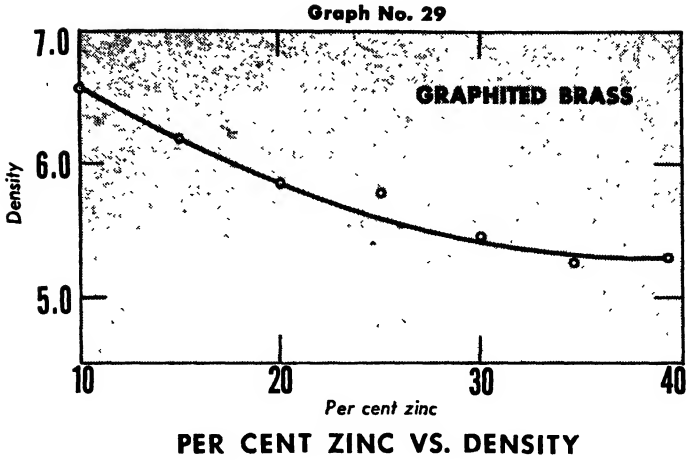
These curves suggest that no true alloy between zinc and copper is formed by the powder metallurgy technique. They seem to show the effect of adding a weaker, softer, more expandable material to a stronger, merely to produce a mixture with properties representing the modifications affected by such a mixture. This is quite different from what occurs when tin and copper are used. Here it is evident that an alloy with unique properties is formed, and the addition of the tin makes more of the alloy formation possible; then further addition of tin modifies these properties by diluting them with its own characteristics.

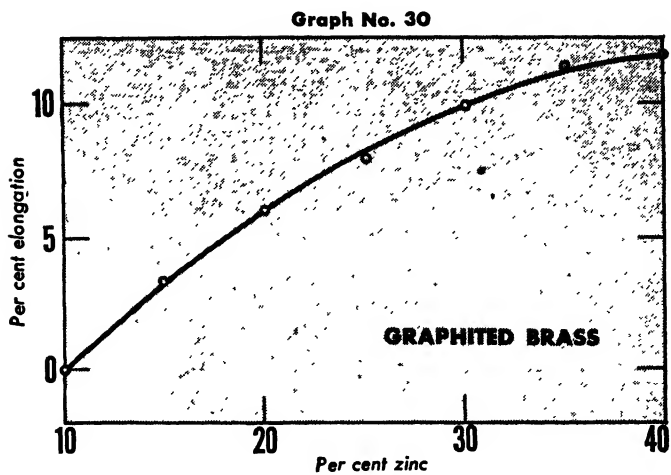
The literature has long referred to zinc-copper compounds as brass alloys: to mention the best known, there are red brass, yellow brass, and Muntz metal. But there is no evidence in the smooth curves presented to indicate that any alloy is formed by the technique employed, at ratios corresponding to these alloys. The fact that it is possible by powder metallurgy to produce a single uniform product of two or more materials without alloying them has advantages

that are frequently sought, as has been pointed out elsewhere where reference is made to contact points, welding rods and other products which are required to retain a special characteristic of both materials employed, which are often lost in an alloy. There is no general rule, however, that metals which will or will not alloy through fusion will or will not alloy through the more modern technique.

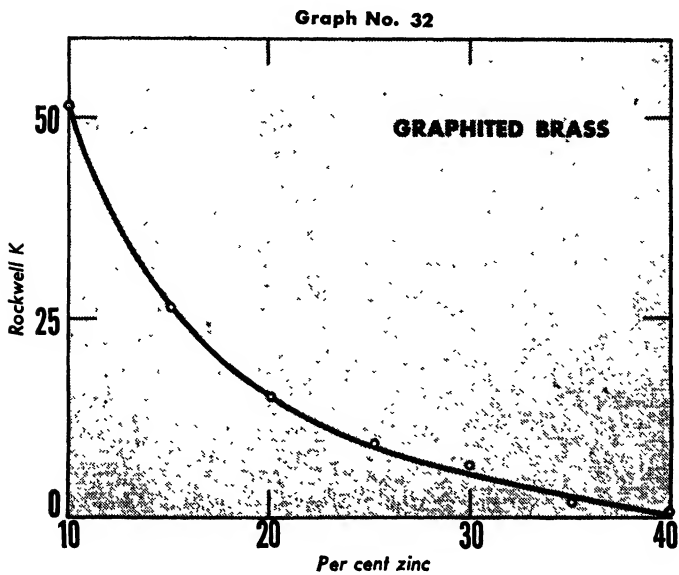
Sometimes alloying may be promoted or prevented by addition agents.







**PER CENT ZINC VS. ELONGATION**



**PER CENT ZINC VS. HARDNESS**

### **Experiment 13. The Effect of Pressure on Aluminum Alloy Characteristics**

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8- or 10-in., 100-mesh sieve  
Lubricant\*  
Die assembly  
Press  
Sinter-box top  
A furnace operating at 900° F.  
Cold-water bath  
Towel  
Set of stamping dies, light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance with weights 50 to 0.05 grams  
A pan balance, with weights 500 to 0.1 grams  
5-in. desiccator  
Three 50-cc. beakers  
Oven regulated at 110° C.  
200 grams aluminum powder  
15 grams zinc powder

*Procedure.* The cores will weigh approximately 9 grams each. Make a sufficient mixture of aluminum with zinc for three sets of cores. Use 93 parts of aluminum with 7 parts of zinc for each 100 grams of mixture. The zinc should be weighed on the bulk balance to within a tenth of a gram.

The mixing should proceed as for the bronze composition. Then press the cores at the indicated pressure in a well lubricated die. Make the usual physical measurements of the green cores but do not transfer to a carbon-packed sinter box. The cores may be laid loosely in the cover of a sinter box and placed directly in the furnace,

\* See footnote, page 144.

which should operate for 30 minutes, the last 15 of which should be at a constant temperature of 900°. As soon as the time of heating has elapsed, withdraw the cores and quench them instantly in cold water. Dry roughly with a towel and place each set of cores in a separate beaker. Dry the cores to constant weight in the drying oven set at 110° C. After removal from the oven do not permit the cores to cool in the room, but cool them in a desiccator. Determine all the physical properties that are customarily determined for bronze. See Experiment 5.

Make one set of cores at 30,000 lbs. per sq. in., another at 50,000 lbs. and another at 55,000 lbs.

#### *Instructions to Squads.*

All squads will press one set of cores at 50,000 lbs. per sq. in. In addition Squad B will make one set at 35,000 and one at 60,000.

Squad C will make one set at 40,000 and one at 65,000.

Squad D will make one set at 45,000 and one at 70,000.

There are sufficient data to present graphically the effect of pressure over a narrow range on aluminum alloy products. When all reports have been made, the data will be correlated during the lecture and will be sufficient to present a graphic presentation of the effect of pressure from 30,000 to 70,000 lbs. per sq. in. on the physical characteristics of an aluminum alloy product.

*Discussion.* If the student has time to recondition his die, and can get along without it for the time necessary to do so, he may press a core made of aluminum powder only, in a well-lubricated die at 50,000 lbs. per sq. in. If he succeeds, he should try making several more. The point of this extra experiment will be brought home forcibly. Even the best made, well-lubricated die will not readily discharge an aluminum compact. Frequently it becomes necessary to drill it out with a drill press, and then much of it will adhere to the walls and require considerable labor to remove. The addition of very small percentages of zinc is an aid in ejecting aluminum compacts, and compacts containing as much as the 7 per cent called for in this experiment offer no difficulty.

Notice that the curves in Graphs No. 33 to 36 are not conventional. Enough data have come to the attention of the writer to convince him that they are typical, but so far not enough to offer

the confirmation which should come from several unrelated sources employing different grades of powder and different alloy mixtures. A conventional curve states a relationship with no modification. For instance: "As we increase the pressure the density will . . . ." At any sinter temperature? With any size particles? Electrolytic or hydrogen-reduced? To each question the answer should be, "Yes". (As scientists we answer "Yes" with a quite well trained and carefully acquired skepticism. We accept few absolutes. There may be a size distribution, which acts differently; there may be a sinter temperature which will produce a different relationship between pressure and density. The chance of encountering such exceptions is very small. And in all sincerity we answer "Yes." No fellow-scientist quarrels with that "Yes," for he also understands it is a conventional one, just as the curves discussed.)

If to any of the questions we may ask, we can only say we have no answer, then we must not designate the curve a conventional one. In this case the data do not rest on a broad enough base to warrant their right to stand as a generalization, but are confirmed by enough sources to stand as typical.

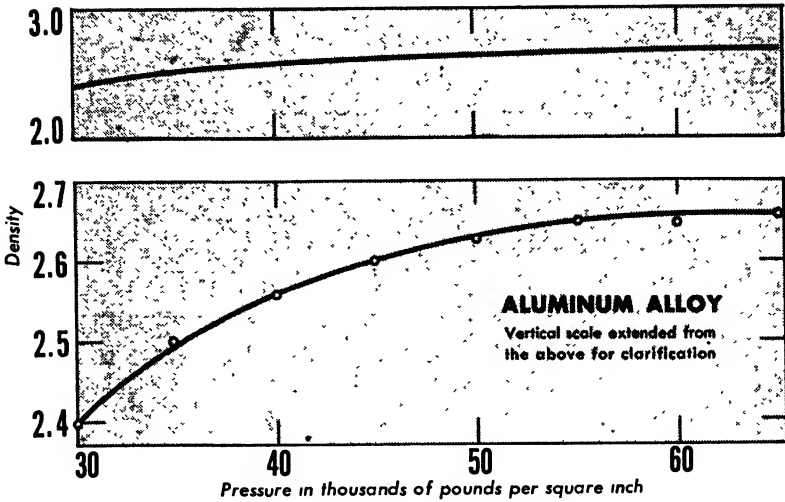
Graph No. 33 indicates the relation between pressure and density. It is drawn on two scales, so the student may observe the form of the curve which is obscured on the smaller scale. How closely does this pressure-density curve for aluminum alloy compare with that for graphited bronze? Since we have here not only another pair of metals quite different in kind, but also in the one instance the presence of graphite, the product is so different, from a metallurgical point of view, that it should afford us no surprise to find little similarity. However, we find that Graph No. 33 closely parallels that of Graph No. 1. The curve drawn to smaller scale is drawn to the same scale as Graph No. 1, and shows that though the two curves are of quite different order, they are both nearly straight lines, indicating that the density increases with pressure more markedly in the lower pressure ranges than in the upper, where increase in pressure has less effect.

Graph No. 34 depicts the relation between pressure and elongation. This too has been drawn to two scales so that the shape of the curve may be studied in the larger, and its order of magnitude oriented by comparing the smaller scale curve with Graph No. 2. This also shows a close agreement between the effect of increasing

pressure on growth, whether on a graphited bronze or on an aluminum alloy; but where the effect in the upper ranges is still marked on bronze, the effect on aluminum is slight as the pressure increases beyond 50,000 lbs. per sq. in. Of course the total effect at all ranges varies very little for the aluminum.

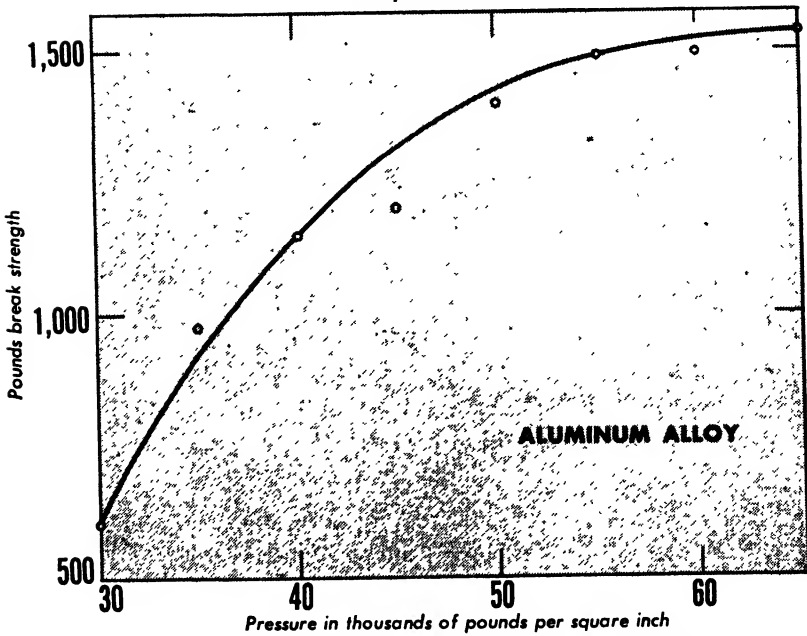
Graph No. 35 depicts the effect of pressure on the strength of this aluminum alloy. As the pressure increases in the low range the strength increases sharply, and though the effect is less in the upper ranges it is evident that increasing the pressure increases the strength throughout all the test range of pressures. Notice, though, that the effect is less marked than the effect of pressure on bronze. Compare this with Graph No. 3. The slope of the curve is much smaller. Graph No. 36 shows the effect of pressure on hardness, namely, that increasing pressure has a marked effect on the hardness: as the pressure increases the hardness increases. The slope of the curve is sharp, much sharper than the slope of the curve in Graph No. 4, with which it should be compared. Even where both curves have a tendency to flatten in the upper pressure ranges, there is much less flattening than in the case of graphited bronze.

Graph No. 33

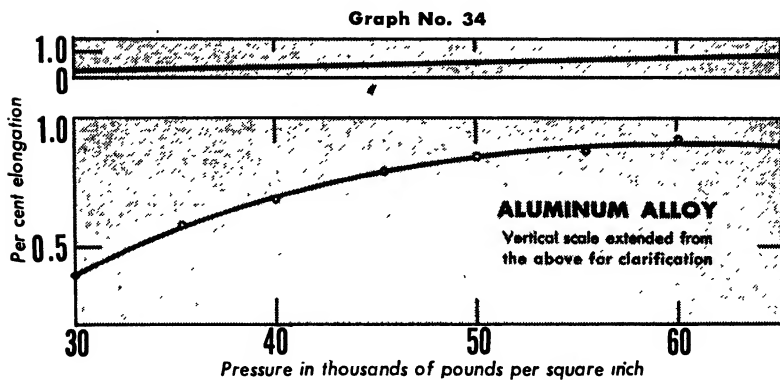


COMPACTING PRESSURE VS. DENSITY

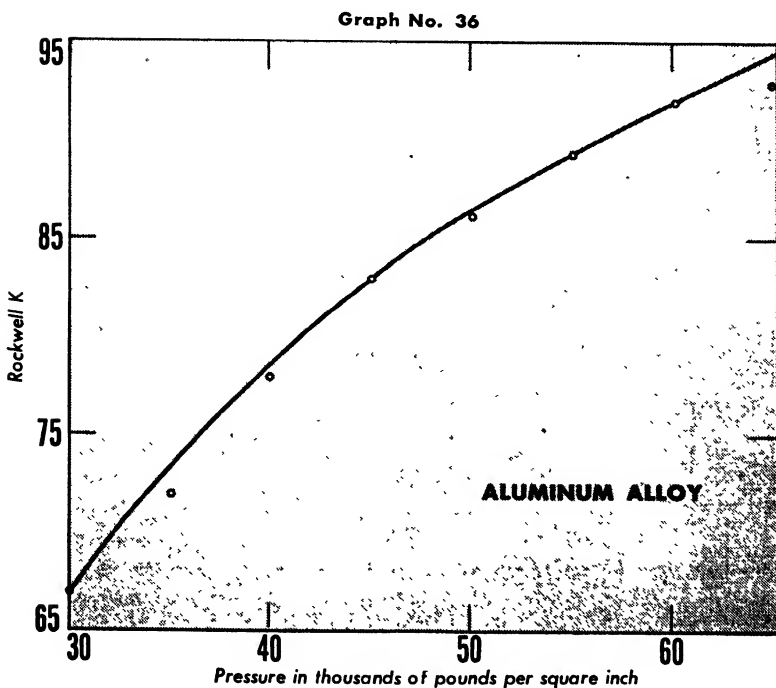
Graph No. 35



COMPACTING PRESSURE VS. STRENGTH



COMPACTING PRESSURE VS. ELONGATION



COMPACTING PRESSURE VS. HARDNESS



## Experiment 14. The Effect of Pressure on Iron Product Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8- or 10-in., 100-mesh sieve  
Lubricant\*  
Die assembly  
Press  
Sintering boxes  
Charcoal  
A furnace operating at 2000° F.  
A  $\frac{1}{4}$ -in. (8- or 10-in.) coarse sieve  
Set of stamping dies, a light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance with weights 50 to 0.05 grams  
A pan balance with weights 500 to 0.1 grams  
Sterotex powder  
550 grams hydrogen-reduced iron powder

*Procedure.* In general the procedure parallels that for the production of bronze cores, but in place of making a mixture of several powders add 0.5 per cent of Sterotex to the iron powder. The Sterotex should be weighed on the bulk balance and screened through a 100-mesh sieve before it is incorporated, and care should be taken to see that it is well mixed with the iron powder. The sinter temperature is 1900° F. The cores will weigh approximately 25 grams.

Make 3 sets of cores, one at 60,000 lbs., one at 45,000 lbs., the other at 70,000 lbs.

### *Instructions to Squads.*

All squads will make one set of cores at 60,000 lbs. per sq. in.

Squad B will make one set at 50,000 and one at 75,000 lbs. per sq. in.

\* See footnote, page 144.

Squad C will make one set at 55,000 and one at 80,000 lbs. per sq. in.

Squad D will make one set at 65,000 and one at 85,000 lbs. per sq. in.

Since this is the first introduction of the iron powder to the student, each squad should run a flow test and a specific gravity test on the iron powder.

From the three pressure points determined, a graph should be drawn showing the effect of pressure on the physical properties of iron products. When all squads have reported this experiment the data will be correlated during the lecture, and a graphic presentation will be made showing the effect of varying the pressure of formation from 45,000 to 85,000 lbs. on the physical characteristics on iron products.

*Discussion.* Care should be taken to work the Sterotex well into the iron. It is best to lay the iron over a wide space in a thin layer, and sift the Sterotex through the sieve so that it settles over it in a thin white coating. It should then be carefully worked in with the spatula and any balling smoothed out. The mix may then be screened again. The Sterotex is added as a lubricant, not a volatile or carbon additive, but it will have some minor effect as such, which cannot be avoided. However, it is important that such effects should not be localized as they will be if agglomerates are fed into the die.

Graphs No. 37 to 40 are not conventional. They are based on experiments performed by about 100 students whose work checked among themselves with reasonable deviation; but since all these men worked with but one grade of iron, the results may not be typical, but correct only for the particular material tested. It is unfortunate, since iron is at present as important to the industry as copper, that the student can be given no generalization. But with so little data available from the industry it would be rash to attempt drawing any broad conclusions.

Graph No. 37 depicts the effect of increasing pressure on density. The density increases slightly with each increase in pressure up to about 70,000 lbs. per sq. in. Thereafter, further increase adds little to the density and there is actually a loss as the pressure reaches 80,000 lbs. The curve is not unlike that of Graphs No.

1 and 33, with which it should be compared. The curve is of considerably greater order than that of Graph No. 33, but less than that of Graph No. 1, and of a lesser slope than either. These other graphs do not extend to the same pressure range, so we cannot tell if they would show that density begins to decrease as the pressure exceeds 70,000 lbs., as is the case with iron.

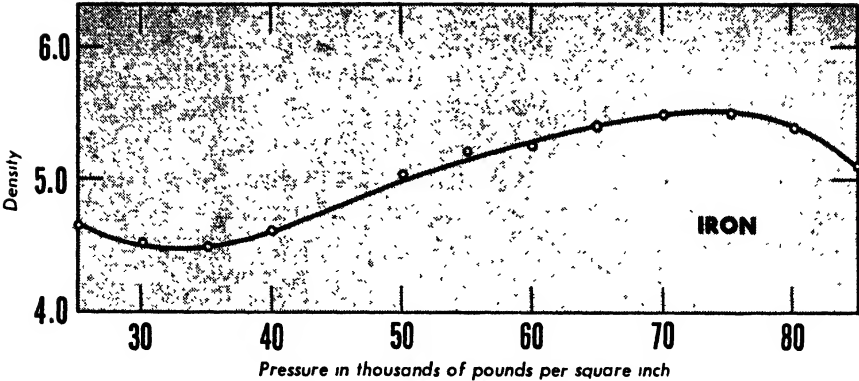
Graph No. 38 gives the effect of increasing pressures on the growth of iron products. But in this case we have no growth, but a contraction, or if you like, a negative elongation. To make it easier to compare with other graphs, the curve is plotted as negative elongation. This is an almost straight-line curve between pressures of 30,000 to 75,000 lbs., at which point it is indicated that increasing pressure no longer causes increasing contraction, but less. The curve is shown twice, once on a large scale so that its form may be observed, and once on the scale used in Graph No. 2, so that the size of the effect may be better oriented. A comparison with Graphs No. 2 and 34 will show that this curve is quite different from these, for as pressure increases the percentage change increases in all cases, but in this case in the opposite direction.

Graph No. 39 shows the effect of increasing pressure on the strength of iron products: as pressure increases there is an increase in strength, up to a point, but thereafter there is no further increase—in fact, a tendency to produce a weaker product is clearly shown. The slope of this curve is much less sharp than that shown in Graphs No. 3 and 35 with which it should be compared, and the curve tends to flatten much sooner than that for aluminum, which in turn tends to flatten sooner than that depicting the effect of pressure on the strength of bronze.

Graph No. 40 shows the effect of increasing pressure on the hardness of iron products. The curve is an elongated S with flattened ends. Slightly increasing the pressure below 40,000 lbs., there is little or no effect on the hardness, but any increase above 40,000 lbs. has a very drastic effect: the hardness increases sharply, up to about 70,000 lbs. At higher pressures little hardness is gained, and at the highest pressure used hardness actually decreased. Comparing this curve with those of Graphs No. 4 and 36, we see that only the middle portion of the curve resembles that of aluminum or bronze, although the upper region does not depart far from the upper range of Graph No. 4.

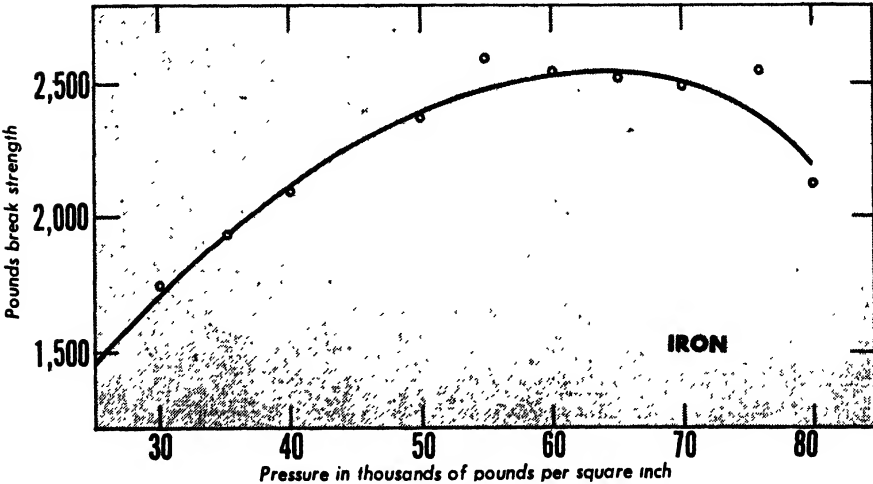
We should consider whether any general conclusions may be drawn by comparing the three sets of curves which indicate the relation of pressure to the physical characteristics of three different powder metal products. We may say that, speaking very broadly the effect is in general similar. As pressure increases density, break, and hardness increase. As to growth, the differences in effect are more prominent than the similarities. More precisely, it is evident that each product reacts in its own distinctive manner, and that a thorough knowledge of the relation between pressure and any one product permits us to predict how pressure will affect the physical properties of other powder metallurgy products only in a tentative, most general way.

Graph No. 37



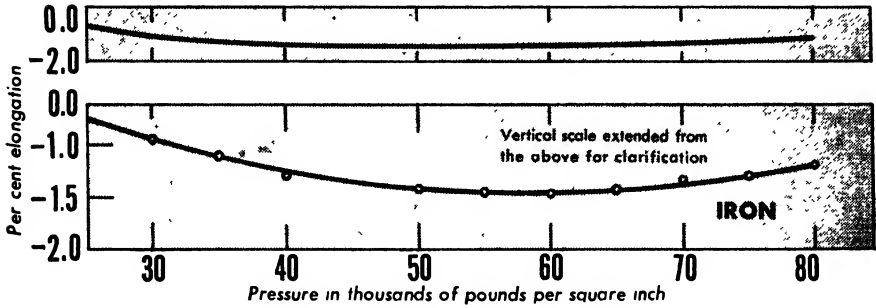
COMPACTING PRESSURE VS. DENSITY

Graph No. 39



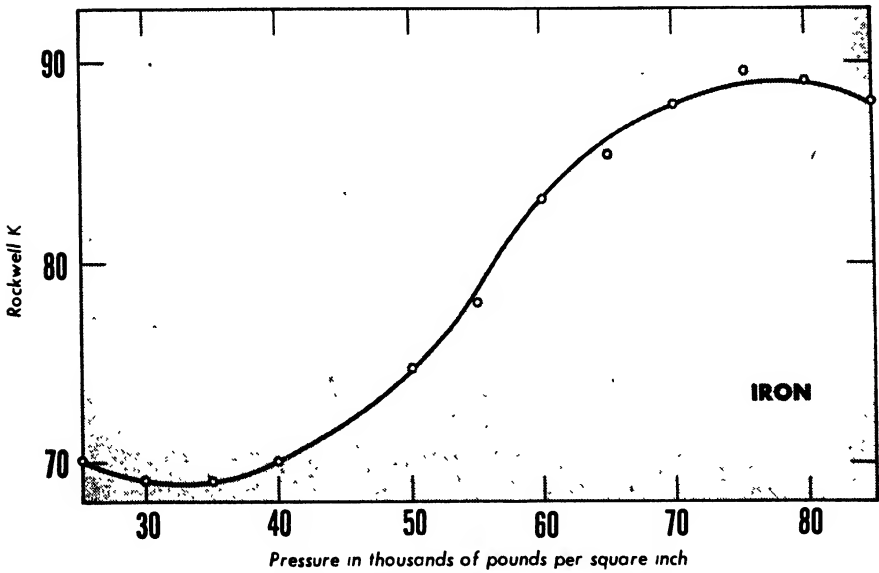
COMPACTING PRESSURE VS. STRENGTH

Graph No. 38



COMPACTING PRESSURE VS. ELONGATION

Graph No. 40



COMPACTING PRESSURE VS. HARDNESS

## Experiment 15. The Effect of Lead on Iron Characteristics

*Equipment.* A large sheet of oilcloth  
A large spatula  
An 8- or 10-in., 100-mesh sieve  
Lubricant\*  
Die assembly  
Press  
Sintering boxes  
Charcoal  
A furnace operating at 1800° F.  
A  $\frac{1}{4}$ -in. (8- or 10-in.) coarse sieve  
Set of stamping dies, a light hammer and steel plate  
Rockwell Hardness Tester  
Break tester  
Two micrometers, one 0 to 1 in., the other 0 to 2 in.  
A bulk balance with weights 50 to 0.05 grams  
A pan balance with weights 500 to 0.1 grams  
Sterotex powder  
550 grams hydrogen-reduced iron  
80 grams lead powder

*Procedure.* Proceed exactly as in Experiment 14, but to the iron and Sterotex add lead powder. The sintering is conducted at 1700° F. Be sure to lubricate the die well. All cores will be produced at 60,000 lbs. per sq. in. Make one set with 2 parts of lead, one with 5 parts and one with 8 parts.

### *Instructions to Squads.*

All squads will make one set of cores with 5 parts of lead.

In addition, Squad B will make one set with 4 and one with 12 parts.

Squad C will make one set with 6 and one with 14 parts.

Squad D will make one with 7 and one with 10 parts.

When all reports have been made, sufficient data will be available to correlate during the lecture and a graphic presentation will be

\* See footnote, page 144.

prepared showing the effect on iron characteristics of the addition of 2 to 14 parts of lead.

*Discussion.* The data available for this experiment are rather sketchy and several points which would have to be used to draw curves have been checked by so few operators, or in some cases not checked at all, that it is thought inadvisable at this time to present any graphs of the fragmentary results. They suggest strongly, however, that a true alloy is formed between lead and iron with about 6 per cent of lead. It would appear that both break strength and hardness seem to reach a peak at about this point. The addition of lead increases density and causes a contraction. These characteristics increase until the lead content reaches about 10 per cent, but further addition causes a decrease.





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