SOME REAL INFORMATION ON POWDER METALLURGY.

By R. L. Hill.

Some months ago I was asked to give a paper on this subject to the Victorian Institute of Metals, and afterwards heard that I had not given sufficient information on the elementary beginnings of the process. So this is the rarest of all things, an article on powder metallurgy practice that really tells the engineer and metallurgist who design the die and supervise the mixing and pressing operations, how these things should be done.

The working of metal powders into solids is not a new art. Early Egyptians are believed to have discovered a method of manufacturing useful implements from iron ore. They knew nothing of the art of casting, and some researchers think that they hammered bits of spongey iron into lumps of workable metal which were beaten into shape and heat treated. Tests of the metal contained in these ancient tools and vessels have indicated that it was never melted.

In this age, powder metallurgy was advanced by men like Sir Henry Bessemer, who was the first producer of metal powders on a large scale, and for many years has been used in the Tungsten electric light globe filament. In England, about the 1920’s, Messrs. Sutcliffe, Speakman and Co. were the first to attempt powder metallurgy on a large scale. They are the makers of the Emperor Press, which is considered the world’s most powerful and efficient briquetting press. Many years ago, they were asked to undertake briquetting iron ore fines which were too fine a powder to feed to the furnaces. They then devised a method of briquetting these fines and feeding them to a kiln where they were sintered. The briquettes were then rugged enough to stand the rough handling they received in furnace loading. About the same time, Japan purchased a plant to briquette their Manchurian iron ore fines; and, in 1935, this plant was duplicated to give them a plant capable of compressing and sintering approximately 100 tons of iron ore fines per hour. In 1915, tungsten carbide was invented in Germany, and was taken over and developed by the Krupp interest in 1916. The same year, General Electric researchers produced a porous sintered bearing material of copper, tin, zinc and lead, but this was too costly for commercial use. The Morane Products section of General Motors, and the Bound Brook Bearing Co. started manufacturing self-lubricating bronze bearings in 1922. America used 4,000 tons.
One could easily devote the full evening to the evolution of Powder Metallurgy, but my purpose to-night is to very briefly outline the work I have personally done.

My firm entered this field of manufacture early in 1940, as in our business as Carbon Brush Manufacturers it was essential that we should be assured of ample stocks of copper carbon which, at that time, was not made in Australia. The main item in all my experiments was copper powder and where to obtain it.

After searching the market of Australia, and finding no suitable powder, I decided to make my own. This I did by having a lead-lined tank made, approximately 18 inches square and 2 feet high. I filled this with a sulphuric acid and copper sulphate solution, placed two porcelain baskets full of scrap copper into it, then connected it to my copper plating generator set and plated on to a square foot of copper sheet using an excessive current. The result was a heavy deposit of soft copper that was scraped off every half-hour into a porcelain tray. This powder was then washed and neutralised with ammonia, and after a further washing was dried. At this stage much trouble was experienced in obtaining a powder free from oxide, but this was overcome by my designing and building a rotary furnace which is still in operation to-day. A length of steel pipe 3 ft. long and 8 inches inside diameter was mounted on four small wheels made of oilless bronze; this was covered with a muffle made of 4 inch fibrolite. To rotate this, a ½ h.p. motor was mounted on top to give 10 revolutions per minute. The ends were sealed with ½ in. plate steel having a gas tap at each end. For heating I used a piece of ¾ in. gas pipe 3 ft. long with a series of jets in it. From this temperatures up to 500° centigrade are obtained. Drying and deoxidising are done by loading the furnace with copper powder, sealing the ends, filling the furnace with town gas to exclude all air, and then trickling hydrogen through for 4 hours at 400° to 500° centigrade. By this method I was able to produce oxide free copper powder.

Soon after this stage was reached, it became apparent that I could not produce all the copper powder I needed, so after more trouble, we approached Messrs. Electrolytic Refining and Smelting Co., who agreed to make copper powder for me.

The first material that I attempted to make, after getting the copper powder right, was copper carbons. In 1938 my father visited England and Germany, and in 1939 I visited America to try and gain some information on copper carbon. Neither of
us could get any real information. We were both shown a certain amount but when, in my case, I asked what were in another room where it was obvious that copper carbons were being manufactured, I was told that they were moulding bakelite combs, and that would not interest me anyway.

This left us to our own resources and, with the help of Mr. Ritchie, and Messrs. McPhersons’ Laboratories, we were able to work out some formulas by having different makes of copper carbons analysed. In this way we soon built up a complete range of carbons. It all sounds very easy now, but in the early stages in the manufacture of copper carbons I experienced considerable trouble with copper powders that appeared to be oxide free, but actually had an oxide content of from 2% to 7%. This oxidised copper when pressed produced slabs with high porosity high electrical resistance, and was of a very crumbly nature, with a dark chocolate colour instead of red copper. After much experimenting and testing I found that a copper powder with an oxide content above 1.5% was useless for manufacturing copper carbon slabs.

The method used for determining the oxide content of copper was to measure the electrical resistance of the powder. The apparatus consisted of a series of small ebonite boxes with two pure silver pieces on the inside. The inside measurements were 1 in. x 1 in. x 1 in. Before filling, the powder was sieved through a 300 mesh sieve, and the powder retained on a 350 mesh sieve was weighed and placed between the silver pieces. An electric current of 5 amps was passed through the powder, and the voltage drop measured on a low-reading milli-volt meter.

More trouble was experienced in producing copper carbon grades where the copper and graphite content was approximately 50/50 per cent., not having enough copper in it to sinter or enough graphite to bind. This problem was solved by increasing the moulding pressure and the moulding time. To-day we have the Tramway and City Council rotary converters fitted with our copper carbon brushes, and they are giving a performance equal to the best imported copper carbon.

The starters and generators in all Ford Army trucks, cars and Bren Carriers are fitted with copper carbon brushes. Other users of copper carbon include such places as Broken Hill Pty. Co. Ltd., all Commonwealth Munition Works and United States Air Corps.

Fifteen grades of copper carbon are now being locally manufactured.

This work naturally led to a survey of other fields, and a very comprehensive study of the manufacture of self-lubricating or
oilless bearings was made. Large quantities of these had been imported from England and United States of America, but supplies were short, and the demand here increased through the shortage of ball bearings.

The Process.—Copper powder used by all of the manufacturers of oilless bronze and similar materials is made by three different processes.

The first process is to melt copper and pour it through a small orifice and drop it on a rotating cone. At this stage some makers used compressed air and some a reducing gas. This method produces a spherical shaped grain.

By taking this process a stage further we have the second process. The spherical powder is made into copper oxide and reduced in a reducing atmosphere furnace to a very fine powder. The Americans can produce this copper powder, grade and pack it for twenty to twenty-five cents F.A.S. New York.

The third method is electrical decomposition. Powder made from this method is dentritic in shape. Experience has proved that these irregular shaped particles are to be preferred where high density and strength are desired in the compressed compact. This is the type of powder being made here, and we have to pay 5/1 per lb. into our store. All of the powders that we use are passed through a 200 mesh screen before mixing with the other ingredients.

The following is a typical screen analysis of the copper powder used:

<table>
<thead>
<tr>
<th>Tyler Screen</th>
<th>% Weight Retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>+ 200</td>
<td>3.6</td>
</tr>
<tr>
<td>+ 270</td>
<td>4.6</td>
</tr>
<tr>
<td>+ 325</td>
<td>7.6</td>
</tr>
<tr>
<td>+ 400</td>
<td>8.0</td>
</tr>
<tr>
<td>- 400</td>
<td>76.2</td>
</tr>
</tbody>
</table>

Our formula is the same as is used by the English, Americans and Germans, and consists of 88.5% copper, 10% tin and 1.5% graphite. We use Ceylon natural graphite and the Acheson synthetic graphite, both graphites being air floated. Atomised tin powder is used, and this is passed through a 300 mesh screen. The following is a typical screen analysis of all the tin powders that are used:

<table>
<thead>
<tr>
<th>Tyler Screen</th>
<th>% Weight Retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>+ 200</td>
<td>Trace</td>
</tr>
<tr>
<td>+ 270</td>
<td>2.5</td>
</tr>
<tr>
<td>+ 325</td>
<td>13.0</td>
</tr>
<tr>
<td>+ 400</td>
<td>20.0</td>
</tr>
<tr>
<td>- 400</td>
<td>64.5</td>
</tr>
</tbody>
</table>
These ingredients are carefully weighed on a sensitive pair of Avery scales to the above formula. They are then placed into a steel drum 1 ft. long by 8 in. diameter, then three or four cast steel rods, 10 ins. long by 1½ in. diameter, are loosely put into the drum and the drum is sealed and placed on rollers rotating at approximately 10 revolutions per minute. By this method the powder is intimately mixed for one hour. After an hour of mixing the drum is taken from the rollers and the mixed powder is placed in large glass jars and sealed. This is to stop oxidation of the copper powder. From the glass jars it is weighed for the different sized jobs in batches of about twelve at the time. It is here that care must be taken to see that the mixture is not exposed to the air for any great length of time, as the dendritic copper oxidizes so rapidly.

Die Design.—Now the mixture is ready for the die, so a description of die design manufacture and maintenance is necessary. The main problem of the pressing operation lies in the fact that when the powder is loosely filled into the die cavity it is necessary to fill the die on the average with about three times as much material by volume as the desired shape. It is this very factor which is responsible for some of the limitations of the powder metallurgy process.

Metal powders do not act like liquids or plastic materials, so, when filling a die, one must visualise that this powder will not flow around corners or fill out intricate shapes.

The die is longer than dies used in moulding plastics. This long die length increases the difficulty of evacuating air from the die. To overcome this, the dies are made with a .10 in. clearance between the plunger and the side of the die; that is a total clearance of .20. In some compacts this clearance is not sufficient, and we have to apply pressure to the compact, then release it, and then apply the full pressure.

All compacts are pressed hot between electrically heated patterns that give a die temperature of 560° to 600° Fahrenheit. Several English, American and German metallurgists have pressed and sintered compacts in dies with pressures up to 100 tons and temperatures up to 900° centigrade. This method is not a commercial proposition, as the die life, even at the low temperatures at which we work, namely, 560° to 600° Fahrenheit, is very limited, due to the falls of the dies bulging out at the point of pressure.

Mould friction is a large factor that we have had to overcome. The method we use is to smear the inside of the die and plunger with a light lubricating oil. Overseas practice is to mix sterine or similar waxes with the powders. By using our simple method
we have increased die life fourfold, and we do not increase the porosity in any way.

At this point, a description of our presses is necessary. We have two 8 in. Ram Hydraulic presses working to 5,000 lbs. per sq. inch hand pumped. Platterns are 12 in. x 9 in. and have 12 in. daylights between them, with a 10 in. ram stroke. There are two 6 in. Ram Hydraulic presses working to 5,000 lbs. per sq. inch. One is operated by a single stroke electric pump, and the other is hand pumped. Platterns are 8 in. x 8 in. and have 24 in. daylights between them with an 8 in. stroke. We also have a 6 in. Ram Hydraulic press with no platterns. This I shall refer to later.

Now that you have a picture of the presses in your mind, I shall continue with the rest of the process. We have the die lubricated and filled with powder. Now we apply pressure to the die, using water main pressure first. This evacuates the air in the majority of cases. After this, the main pressure is applied and held for a few seconds, released, and then the compact is ejected. From here it is stacked until there is a sufficient quantity to charge the sintering furnace. This furnace is gas fired, having a muffle 4 ft. long by 2 ft. square. The compacts are packed into cast-iron boxes filled with calcined charcoal dust and sealed with fire clay.

Temperatures range from between 700° and 900° centigrade, and are obtained by an overnight firing of approximately 16 hours. During this period between 12,000 and 13,000 cubic feet of gas are consumed. Cooling down takes a day, unpacking about two hours—making approximately a two-day cycle. From furnacing, the compacts are machined; contrary to overseas sales talk, this material can be machined.

Demand for this material is so great that we have not yet had time to set up our factory to machine the thousands of bearings which we have orders for, so we have to farm out the majority of the machining.

One factory grinds bearings internally and externally. Another factory turns outside diameter with tungsten carbide tipped tools and ream the inside diameter. Still another factory turns the outside and inside diameters with tungsten carbide tipped tools.

The 6 in. diameter hydraulic press, already mentioned, is used for depressing and can work to .0005 of an inch. Microscopic examination of bearings machined by all these methods failed to reveal any burring over of grains or filling of pores. Further tests of oil absorption on bearings machined by these different methods failed to reveal any difference.
The next process is the oiling. This is done by placing the compacts in a bath of boiling oil for fifteen minutes, the oils ranging from thin spindle oils, anti-freeze oils to high melting point greases, and every job is oil treated according to its application. Now a series of experiments is being carried out for the Department of Aircraft Production, who wanted an alloy to the British specification:

- Tensile strength, 35,000 lbs. per sq. inch.
- 33 in. porosity by oil absorption
- A specific gravity of 6.5 to 7.5, and
- Rochwell H. Hardness of 45/60 = Brinell 37/39

All the following tests have been carried out by Messrs. Dance and Wane, of the Commonwealth Scientific Industrial Research Laboratories.

As a starting point a number of samples of unsintered rods, 3½ in. x 1 in. diameter, and discs, 3½ in. diameter by ½ in. thick, were pressed from our standard formula. These samples were heat treated for one hour at 800° centigrade under a reducing atmosphere and subjected to tensile hardness, specific gravity and oil absorption tests. Microscopic examination of the sintered bronzes were also performed. Test results and microscopic examinations of these first sample bronzes made it apparent that it would be necessary to modify certain characteristics of the raw materials used in order to produce a bronze material with the requisite physical and mechanical properties.

The factors which were modified included grain size distribution of all the powders used, and the cleanliness of the copper powder. The screen sizing of these first samples was everything that passed through 180 mesh sieve. Tensile strengths were between 9,000 and 12,000 pounds per square inch. The next test was with a copper with controlled screen sizing.

Particles of copper powder greater than 200 mesh caused bridging during compacting, with the consequent development of an excessive porosity in the sintered bronze. All copper powder now used is passed through a 200 mesh sieve. Another batch of samples was pressed to the same size as the originals with compacting pressures of 36,000 and 72,000 pounds per square inch, and gave them the same heat treatment. Microscopic examinations of these specimens disclosed that the porosity was still uneven and excessive. It was found that this excessive porosity could be reduced by controlling the screen analysis of the tin powder used. All tin powder is now passed through a 300 mesh screen before use.
Microscopic examination of samples prepared using the screened copper and tin revealed that the primary pores were of a more even size than previously noted, and that the porosity was more evenly distributed. The compacting pressure was 72,000 lbs. per square inch with Brinell Hardnesses of 34.3 to 41.8, tensile strength 13,100 lbs. per square inch, oil absorption 22.4.

Most micro structures of the sintered bronze material so far examined had shown, in addition to the primary porosity, a large number of small pores occurring at the grain boundaries. This is called a secondary porosity.

As far as can be seen this second porosity is closely related to the cleanliness of the copper powder used.

Compacts made using copper powder which had been cleansed in the deoxidising furnace for four hours at 450-500° centigrade with hydrogen trickling through, showed an absence of secondary porosity. Results of tests on these compacts are:

- Compacting pressure—72,000 pounds per sq. inch.
- Brinell Hardness—
  - Longitudinal 40.3
  - Transverse 42.1
- Tensile—19,220 pounds per square inch.
- Specific gravity—6.97.
- Oil Absorption, 19% by volume.

In another test we used E.R.S. special copper with 300 mesh tin, and pressed pieces 3 1/2 in. long and 1 in. diameter, at 72,000 pounds per square inch. We also pressed some flat discs 3 1/2 in. diameter by 1/2 in. thick at 21,000 lbs. per square inch.

After the usual heat treatment the test results were:

1 in. diameter bar:—
- Brinell Hardness—
  - Longitudinal, 36.1.
  - Transverse, 34.4.
- Tensile—16,600 lbs. per sq. inch.
- Specific gravity—6.76.
- Oil Absorption—21.5.

The 3 1/2 in. diameter disc pressed at less than one-third the pressure of the 1 in. bar gives:
- Brinell Hardness—
  - Longitudinal, 37.6
  - Transverse, 36.3
- Tensile—20,200 lbs. per sq. inch.
- Specific gravity—7.02
- Oil Absorption—22.2.
In these series of tests, the highest compacting pressure used was 108,000 pounds per square inch. This gave a tensile of 13,100 pounds, and a Brinell Hardness of 37.3. The best tensile strength test was a disc pressed at 15,000 pounds per square inch, tensile strength 22,400 pounds per square inch with a 19.7% porosity.

Some New Developments.—As I have stated before, one of the principal difficulties in powder metallurgy comes from the fact that the metal powders do not flow readily. This restricts the variety of shapes which can be made successfully by powder metallurgy, and, on occasion, requires complicated mould design to make compacts of a uniform density. A second problem in powder metallurgy resides in the fact that a relatively high compression ratio must be effected in order to obtain compacts having a density equivalent to that of forged and cast material. Thus some metal powders in the unconsolidated condition occupy three or four times as much space as they do after compression. This makes for long plunger strokes in the dies and sometimes requires larger apparatus than would otherwise be necessary.

Evacuation of the die and of the powders prior to introduction into the die, is of aid in overcoming the foregoing difficulties. Moreover evacuation of metal powder during or prior to the compression is an aid in avoiding the formation of lamination or planes of weakness in the compact. A considerable amount of experimental work has been done in Europe and the United States of America. Only one concern, however, has adopted the practice commercially in the United States, and only a few concerns in Europe. Generally speaking, the finer the metal powder the greater the difficulties in getting it to flow under ordinary atmospheric conditions, and the greater is the increase in flow rate of the metal powder and of the compactness which the powder will obtain when evacuated.

Metal powders can be compacted to a greater extent in vacuum when at least a portion of the gas which they entrap under atmospheric conditions has been removed, even though no great compressing force is applied. The greatest saving in compressive force is due to the elimination of the cushioning effect of the gas in the mould in which the compression takes place.

It might be thought that the only saving in compressive force obtainable by removing entrapped gas from powders subjected to compression would be at least about 15 lbs. per square inch, since this is the maximum force exerted by the gas under atmospheric conditions. But, a much greater saving in compressive force results. For example, the density obtainable by com-
pressing, under a force of 25 tons per square inch, metal powders containing the air which these powders entrap under normal atmospheric conditions, is also obtainable with a force of only 5 tons per square inch in an environment having an absolute pressure of 3 millimeters of mercury. This represents a saving of approximately 80 per cent. in compressive force.

We are designing a hydraulic press with three ram chambers, automatic weighing, die loading and ejection. This press will have a capacity of 112 tons, and the maximum tablet size will be 3½ in. diameter by 2½ in. high, pressing at the rate of one a minute. To this press we contemplate trying this vacuum loading of the die.

In our recent research we have been working with iron, iron copper, and tungsten copper compacts, and the results so far have been very promising. As you engineers set us problems by demanding larger and more complicated shapes, so we set you problems by demanding better and more powerful presses, and tougher and longer-lasting dies.