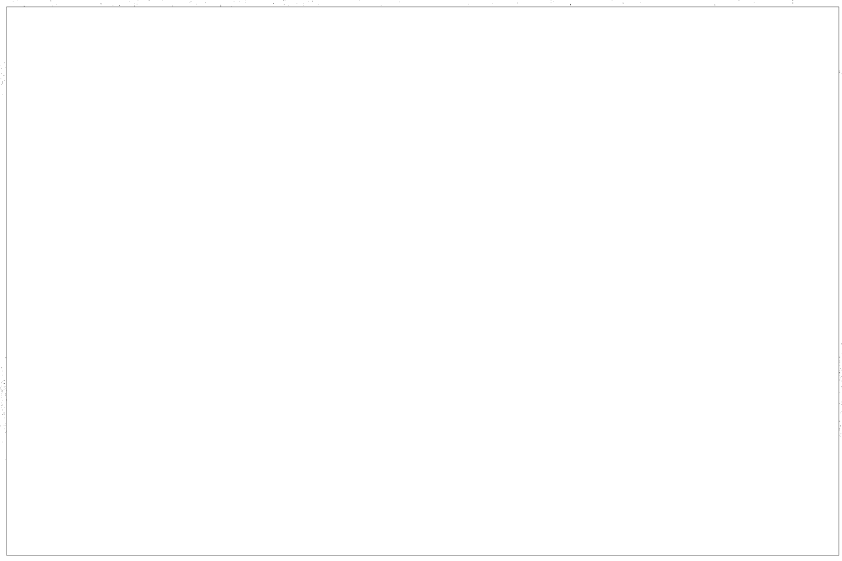
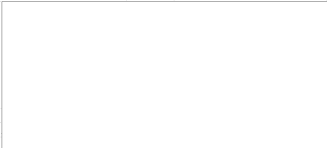


CHAPTER IV

TECHNOLOGY OF THE PRODUCTION OF METAL-CERAMIC GOODS





Chapter IV

TECHNOLOGY OF THE PRODUCTION OF METAL/COCERAMIC GOODS

Metalloceramics (powder metallurgy) is the branch of industry engaged in the production of metal goods from metal powders.

Cold-pressing of properly prepared powder mixtures, with subsequent heat-treatment of the compressed powders by baking them at a temperature below the melting point of the basic component of the alloy is the most widespread method used in the metalloceramic industry. Subsidiary technological methods may be provided for such as cold or hot treatment of the intermediate products after the baking, the elimination of one of the basic operations (pressing or baking), or their combination into one operation, such as hot-pressing, etc.

The basic advantages made possible by metalloceramic technology, the factors determining its fields of application, and the properties of the final products were analyzed in Volume 4, Chapter IV.

METAL POWDERS

Basic Methods for the Preparation of Metal Powders

Table 1 presents a brief summary of the various methods for the preparation of powders. The most important of these are: (1) the mechanical crushing and grinding of solid metals; (2) the pulverization of liquid metals; and (3) the reduction of oxides. In a number of cases, the electrolytic method is used although it is

too expensive.

Other methods for making metal powders have special applications (the obtaining of magnetic alloys from carbonyl iron and carbonyl nickel), or only a potential significance, such as the obtaining of powders by the electric erosion of the metal.

[Table I -- next page]

Mechanical reduction in size of solid metals is accomplished through: (a) the machining of metals by cutting; (b) the grinding of metals in conventional ball mills, hammer mills, crusher-roller mills, and the like; (c) the reduction in size in vorticity mills.

Machining by metal cutting for the obtaining of metal chip for the immediate manufacture of articles is not commercially feasible. Only the utilization of metal chip as a waste product becoming available in the regular flow of the metal cutting industry, for the subsequent reduction in size accomplished in ball-, hammer-, or vorticity mills.

The fine steel and low-silicon semi-steel (up to 1.3 percent Si) chip, having a particle size of about 1 millimeter (obtained from drilling, milling, and sometimes from finish-turning operations) may be used directly for manufacturing purposes without preliminary reduction in size.

The reduction in size in conventional ball mills, hammer mills, crusher-roller mills, etc. can be used in the case of brittle metals only (ferro-alloys, some special alloys, white iron, and

Table I

BASIC METHODS FOR THE PREPARATION OF METAL POWDERS

Initial Material	Technology	Product	Form of Particles	Size of Particles in Microns	Fields of Application
[1]	[2]	[3]	[4]	[5]	[6]
A. Metals and alloys in a solid state	<p><u>Mechanical reduction in size</u></p> <p>1. Machining by cutting</p> <p>The waste from metal cutting (the chip) is used for subsequent pulverization into powder in ball mills in cyclonic mills, and the like. Fine chip may be used directly without preliminary pulverization.</p> <p>2. Reducing in ball mills, crusher-^{er} roll mills, etc.</p> <p>(a) grinding of brittle metals and alloys.</p>	<p>Iron, steel, low-silicon cast iron, brass, bronze, etc.</p> <p>Manganese, chromium, antimony, bismuth, ferroalloys, white iron.</p>	<p>Acicular, curled, etc.</p> <p>Irregular polyhedrons</p>	<p>1000 - 10,000</p> <p>10 - 100</p>	<p>Initial material for further pulverization, also as porous bearings.</p> <p>Steel parts, metaloceramic cast iron for chemical equipment</p>

[1]	[2]	[3]	[4]	[5]	[6]
	(b) reduction in size of weak particles of ductile metals (friable electrolytic sediments, reduced iron sponge).	iron	In relation to the form of the particles being reduced in size	10 - 100	Induction coil cores, porous bearings and the like
	(c). special alloys, to which brittleness was imparted by addition of admixtures and special treatment	Permalloy (Fe-Ni)	Irregular polyhedrons	10 - 100	Magnetic materials
	(d) coarse grinding of ductile materials with weakened bond between the grains (chip)	Steel, brass, and the like	Irregular leaflets, curls, etc.	100-3,000	Porous bearings, steel parts, etc.
3.	Vorticity-mill grinding and reducing in size of ductile brittle [sic] metals and alloys	Iron, iron-nickel, copper, silver, aluminum, silver-tin, and others	Disk-like with bent over edges	20 - 400	Porous bearings, cores, commutator brushes, etc.

[1]	[2]	[3]	[4]	[5]	[6]
	<u>Physico-chemical reduction in size.</u>				
	1-Chemical reduction in size Intercrystalline corrosion of stainless steel 18 - 8	Stainless steel	Irregular polyhedrons	10 - 100	Stainless steel parts
	2- Electro-erosion	Various metals and alloys	--	--	In its research stage
B. Metals and alloys (predomi- nantly fusible) in liquid state	<u>Granulation and pulverization</u>	Lead, iron, copper, silver	Spherical	100-500	Prous bearings, commutator brushes, filters, magnetic materials, contacts, etc.
	1. Pouring into water				
	2. Intermixing of the alloys during solidification	Alloys of alumi- num, cadmium, tin and zinc.	Irregular granulated	< 250	Same as above
	3. Pulverization with compressed air and steam	Aluminum, copper, iron	Irregular drops	20-400	Same as above

[1]	[2]	[3]	[4]	[5]	[6]
	4. Pulverization with simultaneous mechanical action	Iron, copper, nickel, aluminum, bronze, brass, etc.	Irregular coarse drops with complex profile	20 - 400	Same as above
C. Metal Vapors	<u>Condensation of Metal Vapors</u>	Zinc	Spherical	0.1 - 10	Chemical industry, brass parts, hard alloys, and the like
C. Metal Compounds	<u>Reduction</u> L. Reduction from solid compounds (oxides, ores, organic salts) with hydrogen, carbon, and their compounds	Tungsten, molybdenum, iron, nickel, copper, cobalt	Sponge-like	0.1 - 30	Electric ^{light} bulb industry, refractory furnace windings, contacts, hard alloys, porous bearings, magnetic materials, etc.
	2. Reduction from liquid salt solutions	Gold, silver, platinum, copper, tin, nickel, cobalt	Various (acidular, tablet-like, polyhedrons)	0.1 - 10	Porous bearings, contacts, hard alloys, etc.
	3. Reduction from fused salts with alkaline and alkali-earth metals	Tantalum, niobium, titanium, thorium	Various	0.1 - 10	Draw plates, vacuum apparatus, chemical industry, and the like.

[1]

[2]

[3]

[4]

[5]

[6]

cerium, vanadium,
uranium

Thermal dissociation
Disintegration of Carbonyls

Nickel, iron

Spherical

0.1 - 5

Magnetic materials, vacuum
apparata, pure alloys,
and the like

Electrolysis

1. Water Solutions

Iron, copper,
lead, tin

Dendritic

0.1 - 30

Porous bearings, commu-
tator brushes, contacts,
magnetic materials, etc.

2. Fused Media

Tantalum, ni-
obium, titanium,
thorium, cerium,
vanadium, uranium

Dendritic

0.1 - 10

Draw plates, vacuum
apparata, chemical ind-
ustry, etc.

[1]	[2]	[3]	[4]	[5]	[6]
E. Metal and non- metal powder	<u>Preparation of powders from metal compounds and alloys by diffusion and chemical reactions - with heating</u>	Refractory carbides, Various borides, nitrides, silicides, brass, etc.	Various	1 - 100	Hard alloys, refractory compounds, non-ferrous alloy products, etc.

the like). In the case of ductile metals this method results not in the breaking up into fine particles, but in the lamination of the latter. It is therefore used only when: (a) brittle and low-strength conglomerates of ductile metal particles are to be reduced in size, as the final operation in other powder-manufacturing methods, such as pulverization of reduced iron sponge and electrolytic iron precipitates; (b) ductile metals with intergranular bond weakend are to be reduced in size; (c) ductile metals are artificially made brittle (as, for instance, the pulverization of permalloy, an alloy of iron and nickel, is made easier by adding small amounts of sulfur).

The deficiency in the pulverization of metal with the aid of the above described equipment consists in that the powders sometimes become polluted with the attrition products of the steel balls and the mill linings.

Pulverization in vorticity mills. A vorticity mill (Figure 1) consists of shell (1) (lined with attrition-stable manganese steel), in which rotate, at high speed (3,000 Revolutions per minute) and in mutually opposite directions, two propellers (2) cast from manganese steel. The material to be pulverized is charged into bin (4) in the form of coarse lumps (cut wire, metal chip, etc.), to be carried about by the vorticity currents. By colliding with each other at great speeds, the coarse pieces are broken up into particles from 0.02 to 0.4 millimeters in size. The mill is equipped with a device (3) for the air-sorting of powders. The shell of the mill is equipped with a water-jacket, with running water circulating through it to prevent the forming powders from

Figure 1. Diagram of vorticity mill as designed by Podzhus.

overheating. Special provision is made for pulverization in an atmosphere of a protective gas.

In vorticity mills of more advanced design, the air current is fed from a centrifugal blower into the shell of the mill, where it is broken up by beaters mounted on the same shaft with the blower and shell.

The attrition bodies in a vorticity mill are the metal lumps themselves, which are thrown about and collide in the air vortex. This method has the following advantages: (1) the powders are not polluted by the attrition substance from the attrition bodies (steel balls); and (2) the reduction in size taking place during the collision of the light-weight fragments proceeds not by way of lamination, which would smoothen the rough surfaces, but predominantly at the expense of surface deformation, which increases the roughness of the particles. Thus, the quality of the powders, which improves with the roughness of the initial particles, is much higher than what is possible to obtain through pulverization in ball mills.

The microstructure of the iron powders, obtained in vorticity mills grinding is shown in Figure 2. The particles have the characteristic form of a plate with bent-over edges.

Figure 2. Iron powder obtained by vorticity mill grinding. Magnified 25 times.

Figure 4. Iron powder obtained by centrifugal pulverization by the DPG method.

Figure 6. Iron powder obtained by the reduction of oxides.

Figure 7. Copper powder, obtained by electrolysis, Magnified 500 times.

Table 2 gives a typical screen analysis of the iron powders derived by grinding in cyclone mills.

TABLE 2

Typical Screen Analysis of Iron Powders Derived in
Cyclone Mill Grinding

<u>Size of Particles in Millimeters</u>	<u>Weight of the size fraction in percent</u>
0.3	4
0.15 - 0.3	44
0.1 - 0.15	12
0.075 - 0.1	14
0.06 - 0.075	21
0.05 - 0.06	2
0.05	3

To improve compressibility and remove the cold-hardening effect, the powders are annealed in a protective atmosphere (annealing time for iron powders is 1 - 2 hours at 800 degrees Centigrade).

The output of a vorticity mill, driven by a 20 - kilowatt motor, is about 7 - 10 kilograms per hour (depending on the granular size). It takes 2.5 - 3 kilowatt-hours of power to produce one kilogram of powder. One man can operate and maintain several mills simultaneously.

The basic advantages of vorticity grinding are as follows: (a) the utilization of waste products from the metal-cutting industry (chips); (b) little power is required; (c) simplicity of process permits the employment of unskilled personnel; (d) good compressibility of the powders (after annealing) and amenability to baking; (e) powders are free from polluting admixtures; (f) it is possible to obtain powders from alloys.

The method of vorticity grinding is of particular advantage in the manufacture of iron powders for porous bearings, steel parts, and the like.

Reduction in size of a metal by pulverizing it in its liquid state is used only in the case of easily-fusible metals, such as tin, lead, zinc, aluminum, copper and their alloys. The various procedures in this case reduce themselves to the granulation of the liquid metal when poured into water, pulverization of the liquid metal when poured over a rapidly rotating disk, pulverization of the liquid metal by a stream of compressed air or steam, or to various combinations of the above.

The DPG method -- centrifugal pulverization, combining the pulverization of the liquid metal with mechanical action upon the individual particles (see Figure 3), is of particular interest. A thin stream of fused metal flows from a jet, surrounded by a water jacket, into which water is fed under pressure. The water stream carries the liquid metal onto a rotating disk designed with special fittings for the further breakup of the metal particles.

fused metal

water under
pressure

Figure 3. Diagram of centrifugal pulverization as per the DPG Method

The shape of the particles of the iron powder, obtained by this method, is shown in Figure 4 on the inserted page. The particles have a rough profile, and run in size from 20 to 400 microns. A typical screen analysis of such a powder is given in Table 3 below.

For easily-fusible metals and their alloys (tin, lead, aluminum), reduction in size in their liquid state is the most widespread, practically the only method in use.

Reduction of Oxides. In the preparation of tungsten and molybdenum powders, the reduction-of-oxide method is used exclusively.

TABLE 3

Typical Screen Analysis of Iron Powder Manufactured by the DPGMethod

<u>Size of particles (in Millimeters)</u>	<u>Weight of fraction, in percent</u>
0.3	4
0.15 - 0.3	28
0.1 - 0.15	5
0.075 - 0.1	5
0.06 - 0.075	39
0.05 - 0.06	10
0.05	9

For the manufacture of electric bulbs, tungsten and molybdenum are reduced with hydrogen in tubular furnaces at a temperature of 800 degrees Centigrade (Figure 5).

cooler

Figure 5. Tubular electrical resistance furnace for the reduction of tungsten.

The initial material is charged into metal combustion tubes inverted into the tubular furnace. The combustion tubes are passed through the furnace heating zones with various temperatures, and, after the reduction is completed, they are moved along into a running water cooler. The combustion tubes are charged into the tubular

furnace at predetermined time intervals. The usual operating installation consists of several furnaces. The hydrogen, flowing out of the furnace, passes through a water-absorption unit, from there into the next furnace, and so on, until it reaches the last furnace, after passing through which it is burned up.

No particularly pure grade of tungsten is required for the making of hard alloys, and in that case, the tungsten is reduced from the oxide with soot in Tamman furnaces. (See Figure 24).

Cobalt and nickel, employed in the preparation of hard alloys, are sometimes obtained by the reduction of dispersion oxides.

A more difficult assignment is the economical reduction of iron powders. It is rational to use only cheap initial material, such as the separated rolling mill cinder from low-carbon steels, which can be reduced only at high temperatures (within the range of 1,000 - 1,100 degrees Centigrade). The reduction of the iron cinder can be done in tubular furnaces of the same type, as shown above in Figure 5, except that they are producer gas or lighting gas heated, so that the reduction takes place in an atmosphere of producer gas or of converted lighting gas. A considerable difficulty encountered in this process is the required frequent replacement of the tubes.

The iron powders from the reduction of iron cinder contain some non-reduced oxides (about one percent or more of SiO_2 and MnO).

The reduced powders have a characteristic sponge-like structure, in which small individual particles adhere into low-strength chunks (See Figure 6, microphoto, in inserted sheet).

These chunks usually disintegrate into their component particles during the operation of pressing.

Electrolysis. The electrolysis of water solutions is the method most widely applied in the fabrication of copper powders. In the electrolysis of copper, the precipitate is deposited on the cathode directly in the form of powder. The cathodic deposition of the powder-like easily removable dispersion precipitate is promoted by: the low ionic concentration of the metal in the initial solution, by the low temperature, and by the high density of the current. By controlling these conditions, and also the circulation of the electrolyte, with the acceleration of which circulation the dispersion characteristic of the powder is reduced, a product of the desirable characteristics may be obtained.

After removal from the cathode, the powder is washed, filtered, and desiccated by one of the following methods: (1) in a vacuum under a residual pressure of 50 - 100 millimeters of the mercury column; (2) with superheated steam; (3) in a reducing atmosphere (hydrogen, producer gas, lighting gas); (4) in boxes stoked with coal.

Power consumption in the fabrication of copper powder runs at the rate of 2 - 3 kilowatt-hours per kilogram.

The particles of the powders, obtained through the electrolysis of water solutions, have a characteristic dendritic microstructure (see Figure 7, inserted sheet).

The advantages of the electrolytic process are as follows:

- (a) a high degree of purity of the powders obtained;
- (b) good compressibility (sometimes only after annealing) and amenability to baking;
- (c) the facility of attaining a standard output;
- (d) suitability to large scale -- as well as to small scale -- production;
- (e) the possibility for the use of contaminated initial materials;
- (f) the possibility for the separation of valuable admixtures from the anodic slimes.

The electrolysis of fused media is used in the prefabrication of the powders of some rare metals (Ta, Nb, Ti, Th, Zr, V, U), which are obtainable only with difficulty by any other method.

The Technical Control of Powders

In the metalloceramic industry, the powders are tested for the determination of admixtures, the size and structural characteristics of the particles, and the volumetric characteristics. Sometimes, the fluidity of powders is also tested.

Admixture Content. The permissible admixture content in the powders is predetermined by the permissible content of same in the end product, with the exception of oxides, which are being reduced in the baking process. Specifications, therefore, permit a relatively high oxygen content in the powder (0.2 - 15 percent).

Size and micro-structure of particles is usually determined with the aid of a microscope. To obtain a micro-slide, a small amount of powder is poured into a small crucible and poured over with bakelite lacquer or any other transparent plastic mass. Upon the solidification of the bakelite under a heat of from 60 to 140

degrees Centigrade over a period of 12 - 48 hours, the specimen is removed from the crucible, ground and burnished. In quantitative measurements, it must be remembered that the plane of the micro-slide does not always intersect the particles along their maximum diameter. When the particles are spherical in shape

$$d = \frac{\pi}{4} D = 0.79 D,$$

where D is the greatest diameter, d is the mean diameter of the particles, as observed in the micro-slide. (8)

In the case of some fine powders, such as powders of tungsten, cobalt, iron, and the like, obtained by reduction, the microscoping measuring of dispersion becomes distorted, due to the combining of particles into agglomerations. In such cases, the powder is triturated, together with turpentine, with the aid of a glass rod, or the side of the particles of the micro-slide, compressed to a predetermined porosity of the powders, is established, since in compression the agglomerates disintegrate into individual particles.

Screen analysis is another method for the determination of the powder particle sizes. The powders are passed through a series of screens with perforations of various sizes.

Typical screen analyses of powders were given in tables 2 and 3.

The volumetric characteristic of powders may be expressed by any one of the following:

1. Volumetric weight γ (the weight of a unit of volume) in grams/cm³, it being the case that is necessary to differentiate between the poured weight of a powder and its packed weight.

2. Gravimetric volume $v = \frac{1}{\gamma} \text{ cm}^3/\text{gram}$

3. Relative density $\mathcal{D} = \frac{\gamma}{\gamma_0}$, where γ_0 is the density of the powder material. Relative density is expressed by an abstract number (a regular fraction or percent).

4. Relative volume $\beta = \frac{1}{\mathcal{D}}$ which shows by how many times the volume of a free-flowing body is greater than the volume of the powder material, and it is expressed by an irregular fraction, or in percent.

5. Porosity $P = 1 - \mathcal{D}$, or $P = 100\% - \mathcal{D}$, which characterizes the part of the volume occupied by the pores, and is usually expressed in percent.

6. Coefficient of porosity $\xi = \beta - 1$, or $\xi = \beta - 100\%$, showing the proportion of the volume of the pores to the total volume of the free-flowing material. It is expressed by an abstract number, or in percent, and it may assume any value within the range 0 to ∞ .

The weight of a unit of free-flowing powder is determined in the Scott Volumeter (Figure 8).

Figure 8. The Scott volumeter: 1-support; 2 - funnel with screen; 3 - frame; 4 - inclined glass pads; 5 - small crucible

The volumeter consists of a funnel with screen and a frame with several inclined glass tablets forming a chute, through which the powder passes on its way to the crucible, the volume and the weight of which are known. The poured weight is determined by dividing the weight of the powder in grams by the volume of the crucible in cubic centimeters

The volumetric characteristic determines the factor of the compressibility reduction in area during the pressing operation. If, for instance, the relative density of the non-compressed powder is 25 percent, and that of the compressed powder is 75 percent, it means that the powder is compressed threefold and the height of the press mold is to be 3 times the rated height of the resultant blank to come out of the mold.

The volumetric characteristic also determines the "structural" strength of the powder. The structural strength of the powder increases with the decrease in its poured weight.

The density of packing of the powder depends on many factors, which usually affect its structural strength.

The most important of these are the following.

Size of the particles. The structural strength of a powder is primarily stipulated by the engagement effect exerted by the surface rough layer of the particles. In fine powders, the relative roughness is usually greater than in coarse powders. Therefore, in most cases, the structural strength is diminished, and the poured weight reduced with the increase in size of the powder particles.

The form of the particles. The fibrous and dendritic forms provide for the greatest degree of interlacing and mutual engagement of the particles. Such powders are the strongest and the lightest in weight.

Powders with equiaxial and particularly, spherical particles, are the heaviest. The more pronounced the degree of surface roughness, the stronger and the lighter the powder is.

Distribution of particles by size. In the presence of a mixture of small and large particles, the first may be disposed between the second, thereby consolidating the packing and increasing the poured weight.

Intra-particle porosity. Internal pores increase the volume of the particles, without lowering their surface strength, as a result of which powders with intra-particle porosity have a lower poured weight than powders with compact particles.

Surface oxides. Oxides are more brittle than their basic metals. Therefore, the formation of oxides always results in lower strength and higher poured weight (regardless of the reduction in the specific gravity of the material).

Treatment of powders. Treatment in ball mills in most cases increases the poured weight of the powders, as a result of a decrease in the surface roughness of the particles of the destruction of the surface layer and the decrease in its strength.

The relative density of some of the industrial powders is given in Table 4.

Powder fluidity is measured by the amount of powder in grams running through under predetermined conditions, in one minute, through a small hole of predetermined diameter. Knowing this value is essential in automatic pressing.

Figure 9 shows a diagram of a device for the determination of fluidity by the Hardy method.

Figure 9. Device for determining fluidity by the Hardy method. 1 - support; 2 - funnel; 3 - receiver.

Table 4

The Relative Density of some Industrial Powders

Powders	Relative Density of powder in percent	
	poured	packed
Electrolytic copper	12-35	20-45
Electrolytic iron	20-30	30-50
Reduced iron oxide	10-25	20-40
Reduced tungsten oxide	10-25	20-40
Carbonyl iron	35-40	45-50
Granulated tin, copper, lead	30-50	40-65
Vorticity/mill-treated iron	25-35	30-40

This device can also be utilized for measuring the poured weight of the powders. The powder runs through a stainless steel funnel, the angle of the cone being 60 degrees. The diameter of the narrow part of the funnel is 2.5 millimeters, its length is 3 millimeters. The weight of the amount of powder, passing through the funnel in one minute, is recorded. Fluidity is diminished with increased adhesion and friction between the particles, or in other words, with the increase in the dispersion and roughness of the particles and the diminution of its poured weight.

THE PREPARATION AND MAKEUP OF THE CHARGE

Preliminary operations are necessary for the preparation of a charge of predetermined chemical composition, as well as of a pre-specified degree of dispersion and other physical characteristics. These operations consist of separating the powders into fractions, the mixing, and the preliminary treatment.

Separation into fractions. In order to separate the powders into fractions by particle size, the conventional type of equipment, employed in the chemical industry and in the ore-dressing processes is used. To segregate the relatively coarse fractions with the diameter of particles in excess of 50 microns, the powders are passed through wire (brass, bronze, copper), or silk screens. Of all the varieties, preference is to be given to the enclosed-type design, which eliminates dust dispersion.

Figure 10 shows a screen of the enclosed-type, designed by TsNIITMASH.

Figure 10. Enclosed screen designed by TsNIITMASH: 1 - charging bin; 2 - lid; 3 - cradle with screen; 4 - discharge bin; 5 - truck.

Electromagnetic screens, which require no electric motors and drives, and are operated directly from the lighting network, are distinguished by a high degree of portability, low cost, and a high output. However, the sifter tissue in electromagnetic screens rapidly becomes clogged up with powder and goes out of commission.

For the screening of charges containing gluing or lubricating substances, it is sometimes necessary to force the material through the sifter by hand or by mechanical means.

The separation of powders into fractions by screening is practical only for particle sizes down to 250-300 mesh. When particle sizes are down to 40 - 50 microns, it becomes necessary to resort to air separation.

Air separators are sometimes part of the regular equipment in the manufacture of powders (See Figure 1 -- [Vorticity mill]).

Powder blending. By blending two or several initial powders of various chemical composition or degree of dispersion, a charge of predetermined composition and physical properties is obtained. The existing methods of blending are as follows.

1 Blending in drums with eccentric rotation axis. The surmounting of friction between the particles and the intermixing of the latter are attained at the expense of the vibration of the

charge in its rotation about the eccentric axis.

2 Blending in ball mills with steel balls. The surmounting of friction in the intermixing of the particles is attained through the impact of the steel balls. At the same time, reduction in size, increase in hardness and in the poured weight of the powders takes place. The weight of the attrition balls fluctuates within the range of from 20 to 200 percent of the weight of the powder charge.

3 Wet blending in ball mills. Friction between the particles being intermixed is weakened by the effect of the liquid (water, alcohol, gasoline), and a more perfect blending, as compared to the dry method, is obtained.

Dry blending in drums is economical in the following cases:

- (a) for powders of soft metals, subject to high deformation in the ball-mill treatment;
- (b) for metal-graphite charges, since, when these are treated in ball mills, the graphite particles can completely envelop the metal particles, thereby reducing the strength of the end products;
- (c) when the necessity arises to limit the inter-reacting between the components of the mixture;
- (d) in those cases, when no particularly thorough blending is required.

When a higher degree of inter-reacting between the components is specified, as in the preparation of hard alloys, or when necessary to pulverize the particles for the obtaining of a thorough blend, the ball-mill treatment is preferable. To attain a particularly thorough blend, as required in the preparation of tungsten-titanium hard alloys, wet-blending in ball mills over a

period of 5 days (24 hours a day) is resorted to. In order to reduce this long attrition period, vibratory mills are employed, in which case the operation is cut down to less than 24 hours.

In some cases, additional components are introduced into the charge, usually in the form of oxides or from solutions (particularly in the case of ductile metals present only in small quantities in the original charge)⁽¹⁾ Sometimes it becomes necessary to blend metallic powders with organic substances, which are not available in powder form (rubber, stearic acid, glycerin, and the like). In such cases, the charge is mixed with a solution of the corresponding component in a volatile organic solvent, such as gasoline or alcohol. Upon the volatilization of the solvent, the powder particles are covered with a more or less thick film of the substance being introduced into the original charge

Preliminary treatment of Powders. In a series of cases, the powders are subject to special preliminary mechanical, or heat treatment, or a combination of both, for the purpose of modifying their physical properties (degree of dispersion, compressibility, and the like). Such treatment may be conducted in combination with the blending or the screening operation. Frequently, preliminary annealing of the powders is resorted to in order to increase their plasticity and compressibility (at the expense of the reduction of oxides and the removal of the cold-hardening effect). Annealing is sometimes employed to obtain homogeneous solid solutions -- homogenization of the mixture (the annealing of copper-zinc charges, tungsten carbide and titanium mixtures, etc).

Preliminary annealing is conducted in a protective or re-

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Preliminary annealing is conducted in a protective or re-

ducing atmosphere, at temperatures running approximately within the range of 60 - 100 percent of the baking temperature (on rare occasions, somewhat higher than the baking temperature). Such annealing is usually conducted in the baking furnaces.

BRIQUETTING THE POWDERS

By compressing the powders into briquettes semi-finished products are obtained, to dimensions corresponding to the requirements of the end products to be manufactured and taking into account the deformations, to which the briquettes are subject in subsequent operations (baking, calibrating, and the like). The strength of the briquettes must be adequate to absorb the strain of carrying and packing prior to the baking operation.

Briquetting comprises the following operations: (a) proportioning (gravimetric or volumetric) and the pouring of the charge into the press-molds; (b) pressing operation; (c) removal of briquettes from the molds.

Gravimetric proportioning of the charge is employed for non-automatic pressing, volumetric proportioning - for automatic pressing.

The pouring of the charge is to be done in a uniform manner, care being taken to maintain the horizontal level of the powder in the press mold. This attained by various methods: by hand-shaking of the press-mold (tapping it with a wooden mallet) or by its mechanical vibration, by using press molds of predetermined volume, designed precisely to accommodate a given weight of charge (this method is recommended particularly when automatic pressing is used), and finally, by the use of special levelers.

Equipment

Press molds. The press mold (Figure 11) consists of 3 main parts: (1) the tumbler (matrix), which serves for receiving the powder and for the formation of the lateral surface of the compressed mass; (2) the plunger, which is the mobile part for the formation of the top surface and for the compression of the powder; (3) the tumbler supporting base for the formation of the bottom surface of the briquettes and for preventing the powder from pouring out of the press mold. In addition to the 3 basic parts, the press mold is equipped with various auxiliary parts, which provide for the effective ejection of the briquette from the mold, for the maintenance of precision in the size of the briquette, for the uniform compression at both ends, and the like.

By the method for the removal of the briquette, the press molds are classified into two types: (1) briquette forced out of the mold by pressure (Figures 11, 13, 14, 15, 16, 17), and (2) briquette removed from mold upon the disassembling of the latter (Figure 12).

(a)

(b)

Figure 11. Simplest press mold designed with cylindrical end products in view: a - compression position; b - forcing briquette out of the mold: 1 - tumbler; 2 - plunger; 3- tumbler supporting base; 4 - compressing the powder; 5 - pressing-out base

Figure 12. Collapsible press mold: 1- press mold jaws; 2- plunger;
3 - pad; 4 - chock; 5 - bolts; 6 - compression position.

Figure 13. Press mold for bush bearings: 1 - top plunger; 2 -
tumbler; 3 - rod; 4 - bottom plunger; 5 - press table; 6-sleeve
(charging device); 7 - compression position.

(a)

(b)

Figure 14. Press molds for uni-directional compression.

Figure 15. Press mold for 2-directional compression of TsNIITMASH-
design bush bearings: 1 - tumbler; 2 - rod; 3 - ring; 4 - top
plunger; 5 - bottom plunger; 6 - pad; 7 - base support.

Figure 16. Press mold with spring for two-directional compression.

(a)

(b)

Figure 17. Briquetting for end products of complex shape: a-with one plunger; b-with two plungers (1 and 2).

In hand-operated press molds with the ejection of the product (See Figure 11, b), the tumbler base is removed, the tumbler set on a special base, and the product is ejected by pressure applied to the plunger. The amount of ejecting pressure usually fluctuates within the range of 5 to 60 percent of the briquetting pressure. (In ratio to the height and cross section of the briquette and the lubricant). In press molds mounted on presses, the ejection is done from below with the aid of special ejectors, replacing the tumbler base supports (see Figure 13). In the process of ejection, the briquette is subject to some expansion, as a result of the elastic after-effect (0.3 percent in a diametrical direction and 0.5 percent in height). In order to prevent laminary cracking, a gradual widening of the ejection hole (a taper of 0.5 - 1 degree of inclination to the axis of compression) is to be provided .

Collapsible press molds are less productive, and they are used only in low-strength briquetting (tungsten and hard alloys) or in the obtaining of briquettes of complex shape. In pressing, the collapsible press mold is set into a special chock (see Figure 12), in which it is braced with bolts. In some cases, the press mold is encased in a special jacket. In this case, the design of the internal parts facilitates the dismantling. In multi-cradle collapsible press molds several briquettings can be made simultaneously.

To attain precision in the height of the briquette, arresting devices are used. The arresting device is frequently a collection of rings or plates, against which the plunger of the press is thrust, due to which arrangement the descent of the punch will be halted when a predetermined level is reached, providing thereby for the specified height of the briquette. A collection of plates laid upon the bottom plate of the press will also arrest the downward motion of the press plunger at a predetermined level. It is also possible to eliminate the arresting device altogether, if the press mold is provided with a punch, which will be thrust against the upper end of the tumbler, when a predetermined level is reached by the press plunger.

The shaping of internal surfaces in briquetting is effected either with the aid of a punch (die), a base support, or a specially designed rod. With reference to this, several types of press mold design are differentiated.

1 - A press mold for uni-directional briquetting, in which the function of shaping the internal openings is performed by a punch (see Figure 14, a). Such types of press molds should not be used for the manufacture of bush bearings beyond the size of 30 millimeters in diameter and height.

2 - A press mold for uni-directional briquetting, in which the function of shaping the internal openings is performed by a specially designed rod (see Figure 14, b). This press mold may be used for the manufacture of cylinders and thick-walled bush bearings up to 60 millimeters in diameter and height.

3 - A press mold for two-directional briquetting with two

traveling punches -- top and bottom (see Figure 15). It is designed for manufacturing products of considerable height (150 - 200 millimeters and above), or of a high ratio of height to diameter or to wall thickness (15 - 17 times). Press molds of this type are frequently designed with a special pad, which will stop the motion of the bottom punch until such time as the top punch ^{has} attained a predetermined level. When that position is reached, the arresting pad is removed, and the compression proceeds from below, at the expense of the movement of the tumbler relative to the bottom punch. In still other designs, the premature descent of the tumbler is arrested by a special spring, which balances the weight of the latter (see Figure 16).

The advantages derived from the use of two-directional press molds are that they produce briquettes of uniform density and high quality. The disadvantages are that they run into considerable sizes, complex designs and higher cost.

The press mold sizes are determined by the following calculations. The height of the tumbler (matrix) equals the height of the briquette, multiplied by the compression factor, plus the thickness of the top and bottom rings. The compression factor is the ratio of the volumetric weight of the briquette to the volumetric weight of the powder. For most of the industrial powders, the compression factor approximately equals 3, and the height of the matrix is 3 - 3.5 times the height of the briquette. The punch (die) and other operating parts of appropriate computed height.

The clearance between the punch and the matrix is to be adequate to allow the free passage of air during the compression of the

powder. However, when clearances are excessive, the powder will penetrate into them. Kieffer and Hotop⁽¹⁶⁾ recommend clearances within the range of 0.2 to 0.5 percent of the briquette diameter. The clearances used in the USSR are smaller than the above indicated (they are in accordance with the running and free-running fits to the 2nd class of precision). Still closer clearances are used in the United States (see Figure 13).

The internal diameter of the matrix equals the external diameter of the bush bearing, with the change in sizes, which will result from baking, taken into account, plus the compression tolerance in calibration. If there is shrinkage during the baking of some powders, the internal diameters are to be increased accordingly, and vice versa. It follows that the dimensions of the press mold are to be in relation not only to the dimensions of the contemplated end product, but also in relation to the characteristics of the powder used. The calibration tolerance is usually 0.25 - 0.40 millimeters (depending on the diameter of the bush bearing).

The diameter of the rod which forms the internal surface of the bush bearing, equals the internal diameter of the bush bearing, with the due allowance made for the change in dimensions, which will occur in the baking of the briquette, plus the clearance between the calibrating rod of the punch and the internal diameter of the bush bearing. The latter usually equals 0.20 - 0.30 millimeters.

The external diameter of the matrix is computed by proceeding from the strength of the matrix material and the stresses developing in the briquetting operation. The briquetting pressure is increased with the increase in the hardness of the initial

powder. However, when clearances are excessive, the powder will penetrate into them. Kieffer and Hotop⁽¹⁶⁾ recommend clearances within the range of 0.2 to 0.5 percent of the briquette diameter. The clearances used in the USSR are smaller than the above indicated (they are in accordance with the running and free-running fits to the 2nd class of precision). Still closer clearances are used in the United States (see Figure 13).

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powders, in their densities, and in the ratio of the height of the briquette to the wall thickness of the matrix. It generally fluctuates between 1,000 and 6,000 kilograms per square centimeter. The specific lateral pressure exerted in briquetting usually amounts to 25 - 35 percent of the total specific pressure. The lateral (specific) pressure P_2 can be computed by formula

$$P_2 = \xi' P_0 = \mathcal{D} \frac{\mu}{1 - \mu} P_0 \quad (1)$$

where ξ' is the coefficient characterizing the ratio of the lateral pressure to the total pressure; \mathcal{D} is the relative compactness of the briquette; μ is the Poisson coefficient [Poisson's ratio]; P_0 is the specific pressure of briquetting.

The values of ξ' for various metal powders, in relation to their relative densities are enumerated in Table 5.

Table 5

Metal	Value of ξ' with density of powder in percent						
	100	90	80	70	60	50	40
Tungsten	0.21	0.19	0.16	0.14	0.12	0.10	0.08
Iron	0.39	0.35	0.31	0.27	0.23	0.19	0.16
Tin	0.49	0.44	0.39	0.34	0.30	0.25	0.20
Copper	0.54	0.49	0.43	0.38	0.32	0.27	0.22
Lead	0.79	0.71	0.63	0.55	0.47	0.40	0.32

The form and the dimensions of the end products are to be anticipated when designing the press molds (see Volume 4, Chapter

IV "Metallo-ceramic Materials"), with also taking into account the low fluidity of the metal powders. For instance, in the case of a press mold with one punch (Figure 16,a), the compactness of the briquette in the center and along the edges will be uniform.

The most essential parts of the press mold (the matrices) are fabricated from EKh - 12 steel, which, after hardening, has a hardness of 60 - 64 H_{RC}. Less expensive steels (U7, ShKh - 15, St.3, St. 5, and others) are used for the other parts of the press mold. In the United States, high-speed steel, hardened to 62H_{RC}, is used in the making of the matrix and rod.

The useful life of a press mold of average diameter runs within the range of 20,000 - 50,000 briquettes, after which they can be reground into press molds of a larger diameter, or upon some regrinding, nitrided. The nitriding causes some shrinkage in the internal diameter, making it possible to resume briquetting to the original dimensions. In grinding the press molds on magnetic tables, the press mold parts become magnetized. To eliminate the adhesion of iron powder to the magnetized parts, it becomes necessary to demagnetize the latter.

Presses. Hydraulic and mechanical (friction and crankshaft) presses are employed. Friction presses are less convenient and their use entails the risk of press mold breakage. Presses designed for both top and bottom pressure (for the ejection of the briquette) are preferable. Some special designs provide not only for vertical but also for lateral pressure. The briquetting of metal powders in the United States is done by hydraulic presses up to 5,000 tons in capacity and mechanical presses (crankshaft and eccentric) up to 1,500 tons in capacity.

With non-automatic proportioning and briquetting, a crew of 2 - 3 men in one 8 - hour shift, using one press, can produce from 4,000 to 8,000 articles (depending on their size and complexity of shape).

In the United States of America the presses are driven automatically (see Figure 18). The charge loaded into funnel (1), flows gravimetrically into charging sleeve (2), ending above the press mold matrix (3). Upon the filling of the press mold, the charging sleeve is retracted, ceding its place to the upper punch (4), which does the compressing. The compressing stroke at an end, the bottom punch (5) ejects the article, and the charging sleeve (2) moves ahead for the next charge, simultaneously removing the preceding finished article from the table.

Figure 18. Diagram of automatic operation of press (United States)

With automatic proportioning and press operation, the rate of production is determined by the speed of the stroke of the press. The production of simple small articles in a 25 - ton Stokes rotating automatic press proceeds at a rate of 1,000 strokes per minute. The production of large and complex products in American-made presses is sometimes slowed down to 4 strokes per minute. The rate of speed of the plunger stroke in American-made hydraulic presses is sometimes as high as 750 millimeters per minute under load and up to 6,000 millimeters per minute under idle run.

Processes Taking Place in Briquetting

The changes in the structure and properties of powders in the briquetting process are basically reduced to the following.

(1) The area of contact between individual particles is considerably increased. In a first approximation, the area of mutual contact increases approximately in direct ratio to pressure.

(2) The porosity of the powders is reduced, first of all at the expense of the largest pores, the smaller ones following in order of size.

(3) In compression, the particles undergo deformation, which originates at the areas of contact, and with further consolidation, it is propagated from the surface layers into the depth of the particles.

(4) No crystallographic orientation (texture), as is indicated by X-ray tests, is manifested.

(5) Agglomerations of particles and sometimes (particularly in the case of free-flowing powders) the particles themselves are pulverized.

(6) A toughening of the particles due to a "cold hardening" effect takes place.

(7) The strength of the briquettes increases in direct ratio to the pressure exerted (in rough approximation).

Briquetting Pressure

The pressure required for compressing the powder into a

briquette is made up of the following components: (a) pressure p_1 consumed for the consolidation of the powder, in the absence of losses to the external friction of the particles against the walls of the press mold, and with a uniform pressure and density distribution in all parts of the briquette (net pressure); (b) pressure loss p_2 to external friction of the powder particles against the walls of the press molds; (c) excessive pressure p_3 , caused by the non-uniform distribution of pressure and density in various parts of the briquette. The summary briquetting pressure $p = p_1 + p_2 + p_3$ can be called the total briquetting pressure.

In the case of briquettes of small diameter (about 10 millimeters) and small height (1 - 3 millimeters) the total pressure is practically equal to the net pressure (it exceeds it approximately by 5 - 10 percent).

The input of net pressure p_1 can be determined approximately from the following relation (which does not take into account the increase in strength during compression):

$$\log p_1 = \log \sigma_K - L(\beta - 1) \quad (2)$$

where β is the relative volume of the briquette; L is the compression modulus (a constant); σ_K is a constant, equal to the specific pressure requisite for the imparting to the briquette a density of 100 percent. σ_K is approximately equal to the Brinell hardness (more specifically, the Meyer hardness) of the metal, from which the powder originates. In plotting the above in the form of a curve with the aid of coordinates ($\beta, \log p$), the result will be the straight line (1) (see Figure 19) with the following parameters: L is the tangent of the angle of inclination to the

abscissa, and $\log \sigma_K$ is the segment intercepted by the straight line on the ordinate $\beta = 1$ (which corresponds to the density of the consolidated material).

Figure 19. Compression diagram plotted in coordinates $(\beta, \log p)$:
1-the ideal curve; 2-the actual curve.

The actual compression diagram, with the toughening of the powder taken into account, is represented by curve (2) (figure 19), with its convexity downward. A tangent to the curve drawn at point $(\log p_1, \beta_1)$ will intersect the ordinate $\beta = 1$ at point σ_K , which point corresponds to the hardness of the toughened metal in the powder at pressure p_1 and volume β_1 . At the same time, the intersection of the curve with the ordinate $\beta = 1$ corresponds to the maximum hardness of the strengthened metal (the outflow pressure as per O'Neil⁽⁶⁾). Table 6 below shows the values of σ_K and L for various powders at various degrees of consolidation. The values for σ_K oscillate between the hardness of the non-toughened metal and the outflow pressure.

The ratio between net pressure and density is expressed with greater degree of precision by a formula, which takes into account the toughening in compression:

$$\log p_1 = \log \sigma_K - m \log \beta, \quad (3)$$

where p_1 is the specific pressure of briquetting; β is the relative volume; m and σ_K are constants (σ_K is the outflow pressure).

TABLE 6

VALUES OF σ_K AND L FOR POWDERS OF VARIOUS METALS AT VARIOUS COMPRESSION DENSITIES

Powder Material and Characteristics	Low Density of Compression			High Density of Compression		
	Relative Density of briquette (in percent)	in kg/mm ²	L	Relative Density of briquette (in percent)	in kg/mm ²	L
Tin, pulverized, poured weight 3.50 grams/cm ³	56	3.2	0.45	--	--	--
Copper, electrolytic, fine-grained, poured weight 0.97 grams/cm ³	39	48	0.65	93	100	1.05
Copper, electrolytic, medium-grained, poured weight 1.42 grams/cm ³	40	36	0.68	93	88	1.39
Copper, electrolytic, coarse-grained, poured weight 2.47 grams/cm ³	46	48	1.10	93	100	1.50
Copper, granulated, poured weight 4.50 grams/cm ³	--	--	--	93	85	1.72
Iron, electrolytic, annealed, poured weight 2.70 grams/cm ³	46	65	1.60	90	78	1.96
Iron, reduced, poured weight 0.18 grams/cm ³	--	--	--	85	98	0.9
Tungsten carbide	69	1580	3.10	--	--	--

The net pressure, in bringing the powder to 100 percent density, does not depend on the structure and the pouring weight, but only on its composition, and is equal to the outflow pressure. In compressing the powder to less than its 100 percent density, the net pressure is increased with a decrease in its pouring weight and an increase in the cold-hardening of the particles.

The pressure loss to friction of the powder against the walls of the press mold (friction force) Δp for cylindrical briquettes is expressed approximately by the following formulas:

$$\Delta p = 4f \xi \frac{Dh}{D} P = 4f \xi \frac{h_k}{D} P \quad (4)$$

or

$$\frac{\Delta p}{P} = 4f \xi \frac{h_k}{D} \quad (4a)$$

where P is total pressure (compression force); $\frac{\Delta p}{P}$ is the part of the compression force lost to friction against the walls of the press mold (as a constant); ξ is the coefficient of the lateral transmission of pressure at 100 percent density of the briquette (a constant); D is the relative density of the briquette; h is the height of the briquette; $h_k = Dh$ is the reduced height of the briquette (the height of the briquette compressed to 100 percent relative density); D is the diameter of the briquette.

The specific pressure lost to friction against the walls of the press mold $p_2 = \frac{\Delta p}{P} P$, where p is the full specific pressure.

The coefficient of friction f for "dry" metal powders without special lubricating additions usually fluctuates within the range of 0.08 - 0.20⁽¹⁾. The addition of graphite and lubri-

cating substances reduces this coefficient 1.5 - 6 times. More precisely, the loss of pressure to friction is determined by the more complex formula:

$$\ln \frac{P}{P_h} = 4f \xi \frac{h_k}{D}, \quad (5)$$

where \ln is the natural logarithm; P is the compression pressure at the punch; P_h is the compression pressure at the spot of the briquette, which is at distance (reduced) h_k from the punch.

If there occur no other non-uniformities in the distribution of compression and density excepting those caused by the friction of the powder against the walls of the press form, the total specific pressure $p = p_1 + p_2$, or

$$p_1 = \left(1 - 4f \xi \frac{h_k}{D} \right) p = kp, \quad (6)$$

where $k = \text{constant}$.

Non-Uniform Distribution of Density

Loss of pressure to friction against the walls of the press mold results in a non-uniform distribution of density (and, consequently, of hardness, toughness, and other characteristics) along the height, as well as along the cross section of the briquette (figures 20 and 21).

Figure 20 shows graphically the improvement in the distribution of density with the addition of graphite and with two-directional compression.

Grams/ cm³ h_k mm

Figure 20. Variation in density along the height of the briquette grams/cm³ in relation to distance from upper punch h_k in millimeters: 1-electrolytic copper, pouring weight 1.42 grams/cm³, without graphite, uni-directional compression; 2-Same, with 4 percent graphite; 3-Same without graphite, two-directional compression.

Top

s
i
d
e

bottom

Figure 21. Distribution of hardness (as per Shore) in a briquetted copper powder cube.

Ejection Pressure

The pressure consumed for the ejection of the briquette from the press mold is equal to the loss of pressure to friction against the walls of the press form, and it can be computed by formula (4), or with more precision, by formula (5).

Expansion of Briquettes After Removal from Press Mold

In the process of compression, the external pressure p , constricting the briquette, is balanced by the internal elastic stresses, which are equal to p , but are oppositely directed, i.e., they tend to expand the briquette.

After the pressure is discontinued, the longitudinal expansion of the briquette is hindered by the friction against the walls of the press mold, and the transverse expansion -- by the walls of the press mold. Following the removal of the briquette from the press form, the internal stresses will cause its expansion along the height as well as along its diameter (elastic after-effect).

For most of the commercial powders, this expansion amounts to about 0.3 percent along the diameter, and to about 0.5 percent along the height of the briquette. In low-quality powders, the transverse expansion attains a value of 1 percent and the longitudinal expansion -- a value of 5 percent.

Toughness of Briquettes.

The toughness of the briquettes is increased approximately in direct ratio to the compression pressure. The progress of the compression-toughness curve proceeds in three stages (Figure 22).

During the first stage, under the lowest compression forces, toughness increases more rapidly than under pressure of the first degree. Typical cases of briquette disintegration during the

first stage of compression are the crumbling into a heap of shapeless powder (Figure 23, a), or the formation of longitudinal or transverse cracks (depending on the direction of application of the compression load) (Figure 23, b and c).

During the second stage, under the medium compression forces, toughness increases in direct ratio to pressure. Disintegration, which may occur during this stage, is characterized by the formation of cones (pyramids) at a 30 degree angle to the direction of compression (Figure 23, d).

During the third stage, under the highest compression forces, toughness increases at a slower rate than under the first degree of compression. Disintegration, which may occur at this stage, usually takes the form of lamination cracks after the ejection of the briquette from the press mold.

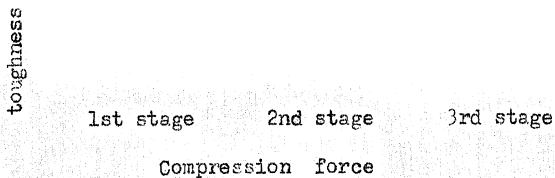


Figure 22. Ratio of briquette toughness to compression force.

(a) (b) (c) (d)

Figure 23. Various types of briquette disintegration.

The toughness of the briquettes basically runs in relation to the following factors:

(1) Compression forces being equal, the toughness of the briquettes is reduced with the increase in hardness of the metal in the initial powders. In tin briquettes, the values of resistance to compression may be in excess of 100 percent of the compression force. In iron and copper powders, it attains a value of 70 - 80 percent of the compression force, in tungsten powders, it amounts to only several percent, and in the case of powders of hard alloys it amounts to only fractions of a percent. This is the specific reason for the addition of agglutinants into hard-alloy powders before briquetting. In the case of annealed powders, the briquette toughness is higher than in the case of cold-hardened powders.

(2) Toughness is increased with the split-up and roughness of the powder particles.

(3) Toughness of briquette is decreased with the oxidation of the particle surfaces and with the addition of graphite.

(4) With the diminution in the powder pouring weight, toughness of briquette increases considerably.

(5) Pouring weight and powder composition being equal, the toughness of the briquette depends little on the size of the powder particles, and is somewhat increased with the coarseness of the initial powders.

(6) Resistance to compression is greater in a longitudinal direction. This anisotropic characteristic of toughness is particularly pronounced in powders with flat-shaped particles.

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- (6) Resistance to compression is greater in a longitudinal direction. This anisotropic characteristic of toughness is particularly pronounced in powders with flat-shaped particles.

(7) Resistance to compression is considerably greater than resistance to tension. For instance, in the case of copper powders, resistance to compression attains a value of 80 percent of the compression force, while in tension it does not exceed 1 percent of the formative pressure.

(8) The toughness of the briquettes greatly depends on the dimensions and configuration of the end product to be obtained.

The toughness of the briquettes is inadequate for their direct use in production.

Briquette rejects. The most serious cause of rejects is the formation of transverse and diagonal lamination cracks.

They are a result of the following: (a) improperly designed and defective press molds; (b) improper compression and ejection procedure, and (c) poor quality of powders.

The most frequent appearance of cracks is caused by improperly designed and defectively made press molds.

In solid press molds, design and structural defects will result in the appearance of transverse lamination cracks during ejection of the briquette. To forestall the formation of such annular lamination cracks, a tapered expansion of the egress opening of the press mold (0.5 - 1 degree angle) is provided. Irregularities in the press mold walls, due to poor fabrication or to excessive wear, will also be conducive to cracking.

Another cause of transverse cracks, with the use of solid press molds, is the application of excessive compression force. The elastic after-effect, which causes the cracks, always increases

in direct ratio to the compression force, while the toughness of the briquettes, that prevents the formation of cracks, increases at a slower ratio. As a result, in the presence of some compression value, the destructive elastic forces will overcome the toughness of the briquette, and lamination cracks will appear.

In assembled press molds, the shifting press mold wall, occurring during the compression of the ejection of the briquette, will cause the crumbling of the briquette with the formation of diagonal cracks. In most cases, such shifts occur while the compression forces are still high.

An excessively slow ejection and, particularly, a stop made during the ejection, will also cause lamination cracks.

All factors, instrumental in the reduction of briquette toughness, such as smooth flat powder, high pouring weight of powder, powder oxidation, graphite in the charge, the cold-hardening of powder particles and the like, are contributing factors in the formation of lamination cracks.

Briquettes are inspected for the absence of external defects, for proper dimensions and weights.

BAKING

The operation of baking consists in annealing the briquettes at a temperature, that is below the melting point of the basic component, for the purpose of improving the mechanical properties of the end products. Sometimes, in order to obtain certain specified properties in the end products, supplementary treatment, following the

baking, is employed, as will be described further down in the text.

Furnaces

Furnaces of the most varied design and operation are used for the baking of metaloceramic products. The selection of a furnace type is done in relation to a series of factors: the baking cycle (the protective atmosphere, temperature, and exposure in baking), the cooling cycle, the composition of the end product, its quantities, size, and shape, and the like. In its turn, the selection of the appropriate baking cycle depends a great deal on the furnaces used.

Electrical resistance furnaces with nickel-chrome or high-resistance alloy winding are suitable for baking up to 1050 - 1100 degrees Centigrade, gas and petroleum furnaces -- up to 1200 degrees Centigrade, Silit resistor electrical furnaces -- up to 1350 degrees Centigrade, electrical furnaces with molybdenum winding -- up to 1400 degrees Centigrade. The Tanman furnace (see Figure 24), used in the fabrication of hard alloys, is heated by an electric current passing through a carbon tube, producing temperatures up to 2000 degrees Centigrade. High-frequency furnaces (Figure 25) operate at a temperature of 2000 - 3000 degrees Centigrade. The highest temperatures, up to 3200 degrees Centigrade, for baking or refractor metals, are obtainable in the so-called welding cupolas (Figure 26), by passing the electric current directly through the articles being baked.

When the articles to be baked are loaded into sealed boxes, chamber or tunnel furnaces of the conventional type may be utilized. If the part of the protective atmosphere is played by the fuel

filling (as, for instance, by petroleum coke in the baking of bronze-graphite bearings), the same type of baking boxes are used as in the case-hardening process. When the protective atmosphere is supplied by circulating gas, the baking box is equipped with tubes (Figure 27) for the circulation of the protective gas.

Figure 24. The Tamman Furnace

(a)

(b)

Figure 25. Diagram of high-frequency furnace: a-electric diagram; b- furnace diagram; 1-refractory plate; 2-sealing water; e-quartz tube; 4-mica lid; 5-article to be baked; 6-support; 7-quartz tube; 8-connecting lead-in pipe for protective gas.

Hydrogen

Hydrogen

Figure 26. Weeding Cupola.

Figure 27. Baking box with tubes for the circulation of the protective gas.

Due to porosity, metaloceramic articles have an accelerated affinity to oxidation. A protective atmosphere is, therefore, necessary not only for the baking, but also for the cooling. In connection with this, the design of the most advanced furnaces includes the computation for the diminution of thermal losses and the reduction in consumption of the protective gas in cooling. Such results are attained in cupola furnaces and in tube furnaces equipped with cooler.

Cupola Furnace (Figure 28) consists of two cupolas: the internal cupola, which is hermetically sealed and equipped with tubes for the circulation of the protective gas, contains the articles to be baked, and the external winding-equipped cupola for heating. This external cupola is removed at the end of the baking process, and mounted upon another internal cupola containing the next batch of articles to be baked. Such a procedure results in the saving of electric power for the preheating of the external cupola, in the acceleration of cooling, and in the reduced consumption of the protective gas.

b a

Figure 28. Cupola furnace: 1-block for setting down the articles to be baked; 2-internal cupola; 3-external cupola with windings; 4-windings; a and b - inlet and outlet for protective gas.

Figure 27. Baking box with tubes for the circulation of the protective gas.

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b a

Figure 28. Cupola furnace: 1-block for setting down the articles to be baked; 2-internal cupola; 3-external cupola with windings; 4-windings; a and b - inlet and outlet for protective gas.

Tube furnace (Figure 29) is equipped with cooler, which is a chamber with double walls (see Figure 5), between which running water is circulating. In this chamber, articles are cooled for 30 minutes down to room temperature, in a protective atmosphere. The protective gas enters the furnace by the principle of counter-current, from the cooler to the charging terminal. The losses of heat and protective gas to cooling are reduced to a minimum.

Figure 29. Molybdenum-resistance tube furnace: 1-molybdenum resistance element; 2-molybdenum rollers; 3-clamp; 4-charging boat; 5-feeding device; 6-ingress and egress of protective gas.

Tube furnaces are equipped frequently with an automatic push rod or conveyor for the effective progress of the articles through the furnace. With the automatic progress of the articles being baked through the furnace, a maximum possible uniformly standard production is assured.

The Protective Atmosphere

A protective atmosphere in baking must accomplish the following: (a) assure the reduction of oxides; (b) prevent the formation of non-desirable production pollutions (soot, carbides, nitrides, etc.); (c) prevent the non-desirable burning-out of individual components in the baking process (for instance, of carbon in hard alloys); (d) assure the safety of the baking process.

As a protective atmosphere, various fillings are used, such as: carbon, graphite, metal chip, reducing and protective gases, hydrogen, producer gas, the incomplete combustion products of illuminating or natural gas, dissociated ammonia, a combination of protective gases with protective fillings, and the like.

In the process of baking, hydrocarbons disintegrate, with the precipitation of soot on the articles. Therefore gases with considerable content of hydrocarbons, such as illuminating gas, natural gas, methane, and propane, are to be subjected to incomplete combustion only. Figure 30 is a graphical presentation of the ratio between the composition of the partially burned natural gas and the air content in the mixture. The apparatus for the incomplete gas combustion is shown in Figure 31.

Volumetric per-
centage of gas
Ratio between air volume
and gas volume

Figure 30. Ratio of composition of incompletely burned natural gas to the air content of the mixture.

Figure 31 Apparatus for the incomplete combustion of natural or illuminating gas (designed by General Electric).

In baking metals, which do not react with carbon, but form

easily reducible oxides (for instance, copper, tin, bronze and the like), any protective atmosphere may be used: coal fillings, producer gas, incompletely burned natural gas, and the like.

When baking iron and its alloys, the possibility of the formation of chemically combined carbon, at the expense of the reaction between the metal and the protective atmosphere, is to be taken into account, by controlling the content of CO and CO₂ in the protective gas in such a way as to avoid the oxidation, as well as the carbonization of iron.

In the baking of tungsten and refractory metals, which easily form carbides, it is best to use pure hydrogen as the protective atmosphere. In the baking of hard alloys, on the contrary, it is necessary to prevent the burning out of the carbon (for example, by the use of carburizing fillings), particularly when the baking is done in molybdenum-resistance furnaces.

In the baking of metals, which form difficultly-reducible oxides (such as chromium, aluminum, etc.), it is best to avoid the content of CO₂ and H₂O in the protective gas. In such cases, the baking is usually done in the presence of sharply desiccated hydrogen, which is allowed to pass through the furnace filled with metal chips.

The baking of metals, such as tantalum, with an accelerated affinity to all gases, with the exception of inert gases, is to be effected in a vacuum.

Processes Taking Place in Baking

The processes taking place in baking are basically reduced to the following: (a) change in the dimensions of the contact surfaces between the powder particles (usually an increase); (b) change in the dimensions and porosity of the articles (both shrinkage and expansion); (c) recrystallization; (d) absorption of residual stresses; (e) reduction of oxides. All these processes are somehow linked either to quantitative changes in the contacts between particles (shrinkage, recrystallization), or to qualitative changes, such as the transition from oxidized film contact to pure metal contact, transition from a tensed to a normal contact.

The coherence of the metal powder to the briquette is due mainly to the purely mechanical engagement through the surface irregularities of the particles, as a result of which the toughness of the briquettes is rather low. In the baked-in articles, the coherence between the grains is due predominantly to electrical engagement forces, effective between the atoms lying on the boundaries of the particles, due to which effect toughness is greatly increased.

Ratio between porosity, structure, and properties of baked-in metals and the structure and properties of the initial powders.
All other conditions being equal (same chemical composition of initial powders, same porosity of briquettes, and same baking procedure), the ratios between the properties of the baked articles and the properties of the initial powders are as follows.

1. The higher the degree of dispersion of the initial powders

the greater the shrinkages (Figure 32), the density, and the mechanical properties of the baked articles (see Volume 4, Chapter IV "Metallo-ceramic Materials"). Recrystallization of fine powders usually sets in at lower temperatures. Therefore, the fabrication of articles from such powders will take lower baking temperatures.

Shrinkage

Figure 32. Shrinkage of iron at various temperatures, as per (12) Dawihl, : 1-size of particles-one micron; 2-size of particles-50 microns.

2. The lower the pouring weight of a powder (for powders of similar structure and similar pouring weights), the higher the shrinkages (Figure 33), the densities, and the mechanical properties. Other factors contributing to the obtaining of tougher briquettes, are the roughness and the splitting up of particles, the low content of oxides in the powders, and the like.

For iron, the relative volume is 1.55

Change in linear dimensions

pouring weight

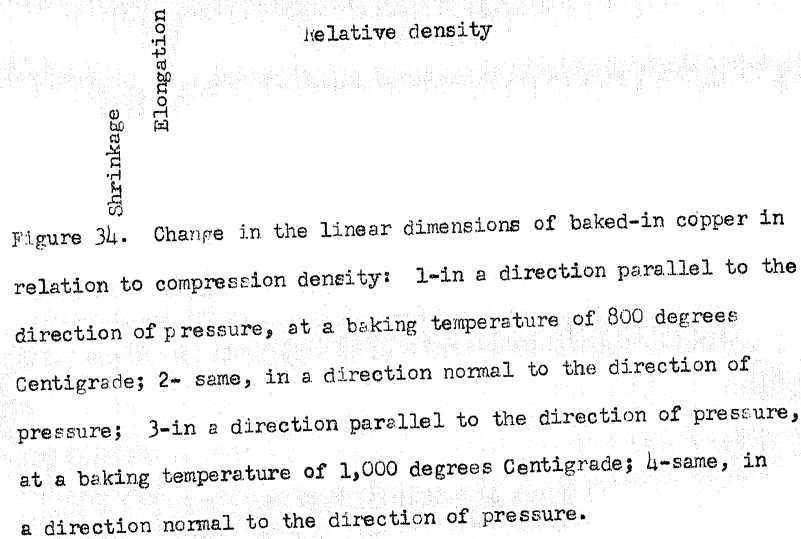
3.66 grams/cm³

Figure 33. Change in the linear dimensions of baked articles in relation to the pouring weight of iron powder: 1 - in a direction parallel to the direction of compression, at a temperature of 1,100 degrees Centigrade; 2 - in a direction normal to the direction of compression, at a temperature of 1,100 degrees Centigrade.

The effect of the density of compression (compression pressure).

The higher the density of compression, the lower the shrinkages (Figure 34), but the final value for density and mechanical properties for most of the powders increase to the very highest pressures used industrially (Figure 35). In the case of many powders, the reduction in porosity after baking is in direct ratio to the initial porosity of the briquettes.

For some powders, there is an optimum compression density (pressure), after the surpassing of which the density and the mechanical properties of the baked articles are diminished. This is usually linked with the formation of defects, such as lamination and hidden lamination, due to excessive pressure.



σ_b, σ_s Kg/mm²; %

grams/cm³

compression pressure

Figure 35. Ratio between the ultimate tensile strength σ_b , Kg/mm²,

the yield point σ_s kg/mm², the elongation δ percent, the specific weight γ grams/cm³, and the compression pressure p m/cm² (iron, baking temperature 800 degrees Centigrade, for 30 minutes in a hydrogen atmosphere), as per Eilender and Schwalbe (13).

The effect of the baking temperature. In the case of most powders, shrinkages (Figure 32), density, toughness, and deformation (elongation, reduction in cross-section), generally speaking, increase continuously with the increase in baking temperature almost up to the melting point. For some powders, however (particularly, fine-grained, showing considerable shrinkages), there is an optimum baking temperature, above which a diminution in density and mechanical properties, at times rather considerable, sets in. This is due to the rapid and non-uniform shrinkage in the briquettes made from such powders at high baking temperatures, which result in local looseness, buckling and even cracking (burning) of the articles. In the case of iron, with the α - γ transformation occurring at point A₃ and somewhat above it, there occurs a diminution in shrinkages, density, and mechanical properties (Figure 36).

Figure 36. Ratio of the mechanical properties of iron during the α - γ transformation to the baking temperature. (as per Eilender and Schwalbe (13)).

The change in hardness after baking proceeds in a different

manner from the changes in the other mechanical properties. With the increase in temperature, the value of the residual stresses, induced by compression, is reduced, the density and coherence of the powder metal are increased, resulting in increased hardness. Therefore, the change in hardness, following the change in temperature, is different for briquettes of different densities (Figure 37). Briquettes of low density, obtained under low pressures, have insignificant residual stresses, and show a high degree of shrinkage in baking. The hardness of such briquettes increases continuously with the increase in temperature. Briquettes of high density, obtained under high pressures, have considerable residual stresses and show low shrinkage in baking, therefore, the hardness of such briquettes will drop within a certain temperature interval.

Figure 37. Change in Brinell hardness in the baking of briquettes from nickel powder obtained under various pressures (as per Grube and Schlecht⁽²⁰⁾): 1-compression pressure of 800 kilograms/cm²; 2-compression pressure of 2000 kg/cm²; 3-compression pressure of 4000 kg/cm².

The apparent increase in the size of the particles for most of the powders of pure metals begins at a temperature of about ⁽¹⁷⁾ 2/3 - 3/4 of the absolute melting point of the metal. The temperature of the beginning of the increase in size does not depend on the compression pressure and is somewhat reduced with an increase in the degree of dispersion in the initial powders. For most of the pure metals, the baking temperature practically coincides

with the temperature of the apparent increase in the size of the particles, since, below it, it is hard to obtain adequate toughness, and above it there is excessive increase in the size of the grain. For metals forming difficultly reducible oxides, which inhibit the size increase of the particles (such as aluminum), the baking temperature is higher and approximates the melting point.

The general porosity of dispersion powders, showing shrinkage in baking, is reduced with the increase in temperature. During this process (particularly with the beginning of the growth in size of the particles), part of the exposed pores is transformed into closed ones, and a part of the intergranular pores -- into intracrystalline pores. However, the decrease in general porosity notwithstanding the size of individual pores may become increased in the process. In coarse powders which cause an increase in the size of the end products, porosity is increased in baking, with the pores remaining exposed.

The effect of baking time and the rate of the temperature rise. The higher the baking temperature, the more rapid the shrinkage and the attainment of the optimum mechanical properties (Figure 38)

Relative density

100 percent

high temperature

mean temperature

baking time

Figure 38. Ratio of the relative density of briquettes to baking (16).
time

Figure 39 shows graphically how the mechanical toughness attains its maximum values in a relatively short time, while the deformation indexes (elongation) take a longer time in rising with the increase in baking time. Practically, the baking time fluctuates from 15 minutes for small charges and small sizes of end products to 24 hours and above for heavy charges, large sizes, complex shapes of end products, and low sintering ability of the material

Kg/mm^2
 Ultimate Tensile Strength
 Toughness
 Elongation

Figure 39. Variation in the mechanical properties of metalloceramic iron in relation to baking time (as per Eilender and Schwalbe⁽¹³⁾).

The more rapid the rate of the temperature rise, the less uniform is the heating through of the products and the baking shrinkage, and the greater the tendency of the end products to buckling, cracking, fusing, and other types of disqualifying defects.

The Baking of Multi-component Metalloceramic Materials. The baking of metalloceramic materials consisting of two or more components may fall into the following basic categories:

1. Baking at a temperature higher than the melting point of both components, with the resulting formation of solid solutions and chemical compounds: (a) when components are not inter-reactive, as copper-graphite, and (b) when components are inter-reactive, with the formation of solid solutions or intermetallic compounds, as copper_nickel, iron-nickel,

2. Baking with the formation of a liquid phase: (a) components not inter-reactive, as tungsten-copper; (b) components inter-reactive, with the formation of small amounts of a liquid phase, as tungsten-nickel, tungsten carbide-cobalt, iron-copper.

In the combination iron-graphite, the baking may be accomplished under conditions, when the iron does not react with the graphite (1a), or when the iron does react with the graphite (1b). The baking of iron-copper alloys may be accomplished, in some cases with and, in some cases, without the formation of a liquid phase.

In the baking of materials, consisting of components which do not react to each other in their solid state (copper-graphite), the baking cycle and the properties of the resulting material are determined basically by the quantitatively predominant component (in this case, copper). The graphite merely plays the part of a mechanical contamination inhibiting the mutual contact of the copper particles. Therefore, the higher the graphite content, the lower the shrinkage, the relative density, and the mechanical toughness of the material. The introduction of graphite into the coarse-grained metal powders is particularly injurious, since in this case, the graphite may isolate completely the copper particles from contact with each other. Small amounts of graphite (up to 2 percent) introduced into fine powders, on the contrary, have a beneficial effect, due to the improvement in compression (the reduction of friction).

In the case of mutually-reactive components under baking conditions without the formation of a liquid phase, the following

(1) Shrinkage, density, and mechanical properties are of a lower value than in the baking of pure metal briquettes.

(2) The speed of the diffusion processes becomes higher with the increase in the degree of dispersion of the initial powders, with longer treatment in the ball mills, and with greater compression pressure.

In the case of mutually-reactive components with the formation of a small amount of liquid phase (such as tungsten carbide-cobalt, tungsten-nickel), a very high density (close to theoretical) may be obtained, and the mechanical properties are not below those in cast alloys. In these cases, the more easily-fusible component enters into the alloy to the amount of 5 - 25 percent (volumetric), and the baking proceeds at a temperature somewhere above the melting point of the easily-fusible metal, or of the solution of the refractory metal in the easily-fusible one. The introduction of such components results in the obtaining of tough and compact alloys at low baking temperatures, which in many cases amount to 50 percent of the melting point of the basic component.

The baking of materials made up of components, which are non-interactive in their solid as well as their liquid state, at temperatures above the melting point of the easily-fusible component (for instance, tungsten-copper) is frequently accompanied by considerable difficulties (the liquation of molten copper). In such cases, it becomes necessary to resort sometimes to special variant methods in metaloceramic technology.

Rejects in Baking

Baking is one of the final operations, which bring to the surface not only the defects caused by an improper baking procedure, but also the defects which might have been caused by the preceding operations in the preparation of the powders. The most important types of flaws are as follows:

(1) Hidden lamination -- the formation of small lamination cracks, not visible to the naked eye. They may be due to an improper pressing cycle, and they are magnified in the baking process. The preventive measures are the same as in the case of visible lamination.

(2) Buckling and shape distortion frequently observed in flat articles made from fine powders where the thickness is insignificant as compared with the length. This type of defect is promoted by: bad mixing, non-uniform density of briquettes due to the improper design of press molds or an improper pressing cycle, too rapid temperature rise, surface oxidations or burning due to the improper selection of the protective atmosphere, and an excessively high baking temperature. Preventive measures consist in removing the enumerated causes, and in some cases, by the use of powders, less inclined to buckling, also baking under pressure. The defects may also be eliminated by subsequent cold- or heat-treatment under pressure (calibrating, baking under pressure).

(3) Burning -- the cracking of articles under baking temperatures, which are excessively high for the given furnace charge.

This type of flaw is promoted by the same factors that cause buckling.

(4) The formation of shrinkage macropores, observed during the baking of articles, compressed from a mixture of powders containing two or several different components. The flaws are induced by non-uniform shrinkage, which is particularly due to the non-homogeneous mixing of the components.

Preventive measures are: more thorough mixing and a preliminary annealing of the baking furnace charge. The flaws can sometimes be eliminated by subsequent cold- or heat-treatment under pressure.

The formation of a low-density inadequately baked core, which is observed principally in the baking of powder mixtures, producing articles of high density as a result of considerable shrinkage in baking with the formation of a liquid phase. The flaws appear as a result of the more rapid heating of the periphery of the articles, when the baking temperature is attained, causing the shrinkage in the external zone to be completed before the shrinkage in the internal part of the article is begun. The premature formation of a compact external zone inhibits the shrinkage of the internal layers and the circulation of gases. The formation of differences in the chemical composition as between the external and internal zones of the article, resulting from the improper selection of the protective atmosphere of baking, is also conducive to this type of flaw. Preventive measures comprise more gradual heating and the proper selection of the preventive atmosphere.

VARIOUS METHODS IN METALLOCERAMIC TECHNOLOGY

The basic and the most widely used method in metalloceramic production is the technological process comprising the operations of mixing, pressing, and baking. The numerous other variants differ from the above basic method as follows.

(1) By specific peculiarities in the basic operations of mixing, pressing, and baking, and also by the introduction of additional intermediate processes between pressing and baking.

(2) By the elimination of the pressing operation.

(3) By the elimination of the baking operation.

(4) By combining the operations of pressing and baking into one (hot-pressing).

(5) Supplementary mechanical and heat-treatment of the baked products.

Specific peculiarities of the basic operations may be as follows:

(a) The pressing and baking of mixtures consisting in part or in full of oxides or metal compounds, which are reducible in baking. This variant method is used in some cases for improving and facilitating the mixing of the powders (as in the production of hard alloys), in other cases, for the attaining of high porosity, and in still other cases, to reduce production costs by combining the operation of obtaining the powder (reduction) and the baking operation into one.

(b) The pressing and the baking of mixtures with special additions for the obtaining of products with a high degree of porosity (above 40 percent). Such additions are either burned out or volatilized in baking (as ammonium carbonate), or washed out after baking through the pores (as soda).

(c) The machine-grinding of the semi finished product prior to baking, or directly after pressing, or after the low-temperature intermediate baking. This variant method is used when some baked materials as, for instance, hard alloys and magnetic materials containing aluminum, nickel, and cobalt (Alnico), are not machinable.

The pressing operation eliminates itself in the case of carbonyl steel. In this case, slightly rammed powders of high pouring weight are baked in sealed boxes. The ingots up to two tons in weight are amenable to subsequent forging and rolling operations.

The operation of baking eliminates itself in the process of obtaining cores for Pripin Coils (the pressing of the powders of ferromagnetic metals and alloys together with plastic masses) and porous iron for the caulking of pipes (the pressing of iron powder together with bitumen).

Supplementary mechanical or heat-treatment is used in many cases for the purpose of bringing the metalloceramic end products up to precision dimensions or to higher density and toughness. The machining by cutting of non-porous metalloceramic materials is no

different from the machining of conventional metals and alloys. For the peculiar features in the mechanical cutting of porous metalloceramic materials, see Volume 4, Chapter IV "Metalloceramic Materials."

To bring porous metalloceramic materials to precision dimensions, cold-pressing in press molds, or so-called calibration, is usually resorted to. Calibration press molds differ from compression press molds by lower height (no reserve is required for the compressing of the powder) and less complex design. Figure 40 shows a TsNIITMASH-designed calibration press mold for the diameter calibration of bush bearings. A calibration press mold of American design, on which calibration by height is also possible, is shown in Figure 41.

Figure 40. Calibration press mold designed by TsNIITMASH: 1-tumbler; 2-punch; 3-tumbler base; 4-product.

Figure 41. Calibration press mold of American design: 1-punch; 2-stopping device for face calibration; 3-tumbler; 4-ejector; 5-working table of press; 6-metalloceramic product.

The dimensions of calibration press molds are such as to take

into account the elastic after-effect (expansion up to 0.1 percent) in obtaining end products of specified dimensions. The ratios between the dimensions of the compression and calibration press molds are so selected that the parts, with the changes developed in baking taken into account, arrive for calibration with transverse dimensions, which are by 0.5 to 1 percent greater than the specified dimensions for the end products.

The amount of pressure used for calibration is usually 10 - 30 percent of the operating pressure. To reduce this amount of calibration pressure, it is rational to calibrate only after a partial impregnation of the article with oil. Calibration is best done in crankshaft presses. One calibrator can complete 5,000 articles per shift.

A subsequent treatment by pressure (cold or Hot), also a supplementary heat-treatment are used to increase the density and to improve the mechanical properties of the end products. Thus, in order to obtain compact and tough materials on an iron or copper base, a supplementary cold-pressing in press molds, sometimes followed by annealing, is resorted to. Moldings made of refractory metals (W, Mo, Ta) are hot-forged and broached. Table 7 enumerates the changes in the mechanical properties of metalloceramic iron after the various production operations. By the supplementary treatment of metalloceramic materials, it is possible to attain mechanical properties equal to those of conventional solid metals.

In the United States, the General Motors Company presses bush bearings from coarse-ground automatic steel chips at 4,500 kg/cm². The compressed briquettes are baked in tube furnaces, equipped

TABLE 7

THE MECHANICAL PROPERTIES OF METALLOCERAMIC IRON AFTER THE VARIOUS PRODUCTION OPERATIONS⁽²¹⁾

Operations	Relative density in percent	Brinell Hardness	Yield Point in Kg/mm ²	Ultimate Tensile Strength in Kg/mm ²	Elongation in percent	Reduction in cross-section area in percent
Cold-pressing at 800 Kg/cm ²	77-83	70 - 75	--	0.28 - 0.35	0	0
Cold-pressing at 6,000 to 8,000 Kg/cm ² and baking at 1000 degrees Centigrade	83 - 88	40 - 50	--	17.5 - 26	8 - 12	8 - 12
Same as above, but with supplemen- tary pressing at 6,000 - 8,000 Kg/cm ² after baking	90 - 93	65 - 70	--	26 - 29	0 - 3	0 - 3
Same as above, but with supplemen- tary annealing at 1,000 degrees Centigrade, after the supplemen- tary pressing at 6,000 - 8,000 Kg/cm ²	90 - 93	56 - 65	11 - 14	23 - 26	16 - 22	16 - 22
Hot-pressing at 810 degrees Centi- grade and a pressure of 3500 Kg/cm ²	95 - 100	60 - 100	--	35 - 40	--	--
Cold-pressing after baking at 1000 degrees Centigrade with subsequent hot-forging	100	90	21	35.5	35	60

with push-rods, at a temperature of 1100 degrees Centigrade. The blanks, prior to cooling, are press-forged in special dies equipped with central mandrels, the latter are fitted into the holes of the baked blanks, which are compressed laterally by eight radial clamps. The pressure during the hot-forging fluctuates within the range of 4,500 - 8,000 Kg/cm²; the rate of production during this operation is 550 pieces per hour. After the hot-forging, the products are water cooled, machined for the removal of lateral projections, undergo final calibration and sand-blasting for the removal of slag and barb. Figure 42 shows a drawing of the part and its external appearance after the various treatment stages.

I

II

III

8 symmetric projections

Figure 42. A drawing of the part and its appearance after the various stages of treatment: I-after baking; II-after hot-forging; III-after final treatment.

A particular type of treatment is the subsequent impregnation of the baked-in shell, made from a metal of higher refractoriness, with the molten mass of a more easily-fusible metal. This treatment is used for the obtaining of antifriction iron-lead materials, lead bronzes, tungsten-copper contact materials and the like.

In order to increase the hardness and the corrosion-

stability of porous iron materials, the baked articles are treated with superheated steam (see Volume 4, Chapter IV "Metalloceramic Materials").

HOT-PRESSING

Hot-pressing is an operation in which pressing and baking are combined into one. Strictly hot-pressing is differentiated from baking under pressure. The difference between these two operations is that hot-pressing is predominantly high-speed pressing. With high speeds of the pressing operation, the phenomena of creep are very little in evidence, also there is no complete diffusion and recrystallization. In connection with this, subsequent annealing must be resorted to in order to equalize composition and structure. The hot-pressing of nonferrous and ferrous metal powders is still non-economical, since: (1) it is accompanied by considerable wear in the new press molds and, frequently, even by the difficulty in finding suitable press mold material capable of withstanding the high temperatures; (2) it is a low productivity operation as compared to cold-pressing; (3) it does not eliminate the necessity for subsequent annealing, as well as for the preliminary cold-pressing. However, from a qualitative angle, hot-pressing offers considerable advantages. Table 8 cites comparative data ⁽¹⁸⁾ on the values of ultimate tensile strength (σ_b) of iron and copper samples, obtained by hot-pressing on the one hand, and cold-pressing ^d and baking, on the other hand. Table 9 cites data ⁽¹⁴⁾ showing the considerable reductions in pressing pressures with higher temperatures, in the case of powders with particle sizes of 150 microns.

TABLE 8

Hot-pressing or baking temperature in degrees Centi- grade	p in Kg/mm ²			
	For baked samples		For hot-pressed samples	
	Cu	Fe	Cu	Fe
610	14.2	--	26.3	19.7
715	13.2	6.6	24.1	29.3
810	10.3	11.5	23.5	39.6
920	--	14.7	--	--

TABLE 9

Powder Material	Relative bri- quette densi- ty in percent	Operating pressure in Kg/mm ² at temperatures in degrees Centigrade							
		20	500	600	700	800	900	1000	1100
Sponge									
iron	90	160	30	16	9.5	5	4	6	3
	95	240	55	30	16	8	6	9	6
	100	350	95	47	32	16	12	14	11
Electro-									
lytic iron	90	120	33	19	13	6	4	6	5
	95	200	38	16	9.5	5	2.4	--	--
	100	310	53	27	16	6	--	--	--
Reduced									
iron	90	200	43	28	17	9.5	6	6	8
	95	310	120	63	32	19	12	6	4
	100	470	110	47	32	16	--	--	--

At present, hot-pressing is used commercially only for the manufacturing from hard alloys of articles of very large size, weighing approximately up to 100 kilograms, or thin plates, disks, and the like, which become buckled in baking. The hot-pressing of hard alloys is performed in carbon molds, which are good only for a very limited number of pieces (1 - 10 pieces). By pressing in a protective atmosphere that prevents the press mold from burning out, the durability of the latter may be somewhat prolonged. The heating is effected by passing an electric current through the press mold.

Baking under pressure is done at low compression speeds. Characteristic of this process are long exposures to baking temperature and, in connection therewith, the full cycle of the processes of diffusion, recrystallization, also the phenomena of creep, in conformity with which the pressures are not high.

Baking under pressure is used for the manufacturing of metal-ceramic friction disks (see Volume 4, Chapter IV "Metal-ceramic Materials"), which otherwise would exfoliate from the lining, and for preventing the buckling or the adjustment of hard alloy cutters. Baking under pressure is best accomplished in cupola furnaces.

THE INSPECTION OF END PRODUCTS

The inspection of the end products is concerned with the absence of external flaws, the determination of the chemical composition, dimensions, weight, structure, porosity, mechanical properties, also technological tests.

The determination of the chemical composition of metallo-ceramics, their dimensions and weights follows the same procedure as in the case of metal products.

Porosity π is determined on the basis of measuring the specific gravity by formula

$$\pi = 100 \left(1 - \frac{\gamma_n}{\gamma} \right) \text{ percent,}$$

where γ_n is the specific gravity of the porous article, γ is the specific gravity of the compact metal. In articles of simple shape, specific gravity is computed by proceeding from dimensions and weight. When articles are of complex shapes, specific gravity is determined by the difference in weight of the article in water and in air. Before weighing the article in water, it is oil-impregnated to prevent the water from filling up the pores. Specific gravity is computed by formula

$$\gamma_n = \frac{G_1}{G_2 - G_3}$$

where G_1 is the weight of the article prior to impregnation, G_2 -- after impregnation, and G_3 is its weight in water. The determination of the amount of exposed and hidden pores is done on the basis of measuring the degree of oil-absorbency. Knowing the specific gravity of the oil, it is easy to determine the volume of the exposed pores. Air-permeability is indicative of the size of individual pores. The larger the individual pores, the greater the degree of air permeability. For the determination of the latter, the metaloceramic bushing is clamped at its faces into a special clamp and placed into a graduated measuring cylinder filled with water. From the direction of one face, under a predetermined small pressure (a fraction of an atmosphere), air is fed into the bushing, with the

result that, coming through the pores, it expels the water from the cylinder. Air-permeability is determined by the volume of air passing through the pores of the bushing per minute.

In determining the microstructure it is necessary to guard against the distortion and the tightening of the pores in grinding or in burnishing. For this purpose, the pores are preliminarily impregnated with bakelite lacquer, followed by heat-treatment for the bakelite hardening.

The determination of the mechanical properties of metalloceramic materials is complicated by the following peculiarities. The porosity of metalloceramic articles makes the determination and evaluation of the mechanical properties rather difficult. Their small sizes and non-homogeneous density makes the cutting out of sampling specimen rather complex. In addition, cutting out of strips invariably reduces the toughness of porous materials. It is therefore recommended to measure the hardness of the articles directly, without any cutting involved. Tests to determine the ultimate tensile strength can be made directly on the article or on its chips by the method of wedge pressure (as per Ludwig⁽⁵⁾). For the methodics of testing, see Volume 3. Testing for ultimate strength in tension and compression is usually conducted on samples pressed from the same powders in special press molds and baked under the same conditions, as the consignment of articles being tested. Impact ductility tests are made on samples without any preliminary notching.

Porosity greatly affects all the mechanical properties. Usually (see Volume 4, Chapter IV "Metalloceramic Materials"), the

reduction in porosity by 1 percent corresponds to a mean increase in the toughness indexes by 5 percent (from 2 to 15 percent). In porous metals, the ultimate compression strength is considerably (1.5 - 4 times) greater than the ultimate deflection strength, and the latter, in its turn, is 1.5 - 4 times greater than the ultimate tensile strength. Elongation in porous metals is insignificant.

E N D

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