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POWDER METALLURGY

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ABSTRACT OF THE DISCLOSURE

A fine grained metallic article produced by powder metallurgy from a metal powder such as aluminum is obtained by (*a*) coordination of the amount of lubricant in and compaction pressure of a shaped mass of the powder so as to establish interconnecting porosity, and (*b*) heating the shaped mass having interconnecting porosity up to its sintering temperature in a moisture-containing atmosphere at a rate such as to control pore oxidation and form an oxidic internal-skeleton supporting-structure so as to prevent sagging during sintering.

This application is a continuation-in-part of our earlier filed application Ser. No. 210,072, filed July 16, 1962, Ser. No. 373,445, filed June 8, 1964, and Ser. No. 398,490, filed Sept. 21, 1964, respectively, all now abandoned.

This invention relates to an improvement in aluminum powder metallurgy and more particularly to a product and a method for obtaining the same characterized by high strength isotropic properties.

Powder metallurgy is a very convenient method of obtaining intricately shaped objects with a minimum of effort and expense. Broadly speaking, the technique of powder metallurgy involves mixing the particular metallic components in powder form, inserting the mixture of metallic powders in a suitable shaped die or mold in order to impart the desired shape, followed by sintering at a temperature sufficient to cause diffusion of the powder particles to form a substantially homogeneous article. While the technique of powder metallurgy has been in use for many years, there are certain metals which can only be treated in this fashion, if at all, with extreme difficulty. Foremost among these difficulty treatable metals is aluminum. A characteristic of aluminum is its tendency to rapidly form an inert oxide coating. Such an oxide coating on aluminum particles serves to act as an insulator to prevent adequate diffusion between adjacent particles. Even in those instances where powder metallurgy techniques have been applied to aluminum, the products obtained are characterized by rough surface distortions, non-uniformity of appearance, and very frequently contain blow holes and/or blisters. For these reasons, the powder metallurgy techniques with respect to aluminum has never become a commercial reality.

From a theoretical standpoint, if completely pure elemental aluminum were employed, and the entire operation took place in the complete absence of any oxidizing substance such as, for example, water vapor, some of the problems inherent in aluminum powder metallurgy might be obviated, but such conditions are substantially impossible.

Cremer and Cordiano in Technical Publication No.

1574 of the American Institute of Mining and Metallurgical Engineers (1943) discussed some of the problems in aluminum powder metallurgy, and indicated that for any kind of successful results, the die or mold employed for shaping the powder mixture would have to be lined with a flat lining of overlapping metal flakes which are applied by suspending flake powders in carbon tetrachloride and spraying a thin film to form a substantially impenetrable layer on the die walls. Under some circumstances a fatty acid or soap was added to the carbon tetrachloride. Even with this precaution, Cremer and Cordiano still found that it was necessary to employ shaping pressures on the order of 50 t.s.i. in order to get good results. This die coating and high pressure shaping was required even if aluminum powder was not the sole constituent but was present in admixture with other alloying powders.

Other workers such as Goetzel in U.S. Patent No. 2,155,651 have found it necessary to use a completely desiccated atmosphere during sintering, which takes place at a temperature of about ⅔ of the weighted average of the melting points of the elements present in the mass, a temperature, which from the specific examples given, appears to be intended to maintain sintering below the eutectic temperature.

The as-sintered products can be fabricated to have elongations at break of at least 5%, when the product has a density equal to or greater than 95% of the theoretical density and in many instances at lower densities.

Powder metallurgy techniques applied to aluminum in the past are substantially incapable of producing regular uniformly appearing specimens. Accordingly, the products of this invention are distinguishable from conventional powder metallurgy products by their regularity, uniformity and even appearances, free from bulges, irregularities and macroscopic holes.

By means of this invention, it is possible to fabricate aluminous products by powder metallurgy techniques that will have extremely high ultimate tensile strengths, extremely high crush strengths and excellent ductility. Contrary to the methods hitherto employed in the powder metallurgy of aluminous parts, the method of this invention is capable of yielding shaped aluminous parts, containing over 90% by weight of aluminum, having crush strengths in excess of 8,000 p.s.i. and frequently in excess of 10,000 p.s.i. with a deflection at failure of greater than about 3%. Such crush-resistant products are particularly useful as bearings.

The powder meallurgy products of this invention are particularly useful for cold forging. For example, if a cylindrical blank prepared in accordance with this invention is cold forged by conventional means into a complex shape such as a gear in a closed die, the metal flows uniformly into the die cavity and all teeth are well formed so that the gear product exhibits dimensional uniformity. In the case of cast wrought aluminum compositions employed in this same die to produce a gear, the metal flow would be non-uniform and thus certain teeth of the gear would be less well formed than others. The gear in general would exhibit obvious dimensional non-uniformity.

Similar superiority of the compositions of this invention is seen in other application. For example, if a cylindrical blank produced in accordance with the invention were cold impact extruded into a thin-walled cylindrical can, the uppermost edges of the can would be observed to be substantially at right angles to the can without projecting points or waviness. In contrast, conventionally prepared cast wrought aluminum of the same chemical composition when extruded in this method, would be observed to have a considerable number of "ears," i.e., projecting points, requiring that excessive amounts of material be trimmed from the can during the finishing operation.

In carrying out the process of this invention, particulate aluminum plus a lubricant are admixed with any additional metal powders, as hereinafter described, placed in a suitable die or mold, compressed, if desired, depending upon the ultimate density and degree of porosity required, and placed in a suitable heating area such as a furnace or oven, and heated rapidly up to the sintering temperature in the presence of an inert atmosphere (inert to aluminum at the sintering temperature) having a restricted (but not absent) moisture content. This temperature is maintained for a itme sufficient to develop optimum sintered and alloyed properties in the product. At the conclusion of the sintering operation, the product is cooled rapidly, or slowly as desired. Thereafter, any one of the variety of subsequent working treatments can be performed.

The aluminum should be in particulate form. The exact shapes of the aluminum particles are relatively unimportant in that they can be in the form of powder, needles, wire clipping, scrap turnings, and the like. However, the use of generally spherical particles are generally less responsive to pressure transmission during the compaction stage. Thus, any non-spherical aluminum particulates having major dimensions in excess of about 3 microns are satisfactory. Smaller aluminum particulates often present both an explosion hazard and a die filling problem. The maximum particle size will be determined by the size of the final product, the capacity of the equipment employed and by the necessity of insuring a uniform mixture of each of the components. For example, in the case of products intended as forging blanks for hot and cold forging and extrusion applications where compacting pressures in excess of 3 p.s.i. are to be employed, the aluminum particles should not be too coarse and hence should preferably pass through a 10 mesh sieve. In some instances, it is possible to employ particles as large as ½ inch aluminum granulated ingots or shot.

Typical commercially available particulate aluminum materials that can be employed as starting materials in this invention include the following air-atomized types having the specified mesh distribution, expressed in percent by volume.

Mesh Distribution	Type A	Type B	Type C	Type D
100	11.0 (percent vol.)	39.5%	23.0%	0%.
100/140	13.4	16.5	59.4	0.
140/200	14.5	15.5	16.9	0.
200/270	11.5	8.2	0.6	0.05.
270/325	14.0	10.6	0.0	10.8.
−325	35.6	9.7	0.05	89.0.
Flow Rate Sec./50 gm	None	94 sec.	93 sec.	None.
Density gm./cc	1.115	1.165	1.042	0.902.

In addition, commercially available centrifugally spun molten aluminum particulates are available. Typical examples have the following dimensions:

	Length, in.	Diameter, in.	Apparent Density, gm./co.
(Alcoa 3/5 mesh shot)	0.0933 0.0517 0.0418 0.0532	0.0141 0.0088 0.0072 0.0104	1.176 1.152 1.148 0.996

The final powder mix employed in this invention will contain from 50% to 99% or more of the particulate aluminum depending upon the properties desired of the final product and the quantity of lubricants and other additives. It is desirable but not essential that the aluminum powder be relatively pure metal such as, for example, helium atomized aluminum. In such metals the oxide coating surrounding the aluminum particles are minimized. However, air atomized aluminum can also be employed in the practice of this invention without deleterious results. Impurities contained in the aluminum are best minimized and preferably should not be present in an amount of more than about 1%.

Where a pure aluminum product is desired, of course, no additional metals need be added to the powder mix. However, the strength properties as well as other attributes of aluminum can be enhanced, as is well known, by the use of other alloying elements. Thus, for example, a stronger product can be obtained in the case of analuminum-copper alloy containing about 5% copper than can be obtained with pure aluminum. Accordingly, it is often highly advantageous for the development of a strong final product to include a small quantity of particulate copper in the powder mix. The type of particulate copper employed is again not critical except that good results are attained when a dense product is desired if a small amount of copper in flake form is present in the powder mixture. Thus, if the compaction pressure employed, as will be explained hereinafter, is such as to compress the powder mixture to a density in excess of about 85% of its theoretical density, then the presence of copper flake serves not only as a source of copper to form the alloy but also serves as an auxiliary lubricant to ease compaction and ejection problems. For such purposes, from about 1% to 5% by weight of the total powder mixture can be copper flake. Care must be taken, however, when employing copper flake to use an amount less than that which would cause visible striation in the powder mixture. This is a phenomenon easily observed by the naked eye. Generally speaking, no striation problems arise when up to about 5% by weight of copper flake is employed. Occasionally, greater quantities of copper flake, for example, up to 10%, can be employed without serious striation problems if the particle size of the aluminum powder is quite small.

Conventional copper flake can be employed and will generally range in thickness from 8×10^{-4} inch to 5×10^{-7} inch with flake area generally ranging from 1.5×10^{-9} to 4.9×10^{-6} square inch per flake. Commercially preferred flake range in thickness from 0.5×10^{-5} to 8×10^{-5} and in area from 0.15×10^{-7} to 4.9×10^{-7} square inch.

Where alloys containing a greater amount of copper are required, or where advantage need not be taken of the beneficial properties of the copper flake, copper powder can be employed as a supplement to or in place of copper flake. Copper powder, when employed, is generally not employed in particle size greater than that which would pass through a 325 mesh sieve. The large particle-sized copper powders tend to cause the formation of macroscopic holes in the final product. Of course, if such holes are desired in the final product, then larger particle-sized copper powder can be employed.

While copper has been described as an alloying additive in the powder metallurgy mix of this invention, it can be supplemented partially or replaced completely by other alloying additives including, for example, magnesium, aluminum-magnesium alloys, silicon-aluminum alloys, zinc, brass, or any other conventional aluminum alloying additives. These aluminum additives can be in the pre-alloyed form with aluminum or other metals or can be present as the pure elemental metals. Certain additives such as silicon would only be added in the prealloyed form. When employed, they will be present in the particulate form having a particle size sufficiently small to pass through a 25 mesh sieve and preferably, to avoid the presence of macroscopic holes, through a 325 mesh sieve. The actual concentrations and proportions of the various alloying elements are preselected in accordance with the end properties of the final product. Again, the use of from about 1 to 5% by weight of the total powder mixture of flakes of the alloying additive results in facilitating compaction.

The total amount of alloying additives such as copper and magnesium, including both flake and powder employed in the powder mixture, can range from 0 when no alloying additives are present up to about 45% of the weight of the mixture. Generally, the best physical properties are obtained when the amount of alloying additives in the mixture is below about 10% and preferably within the range from about 1% to about 8% of the weight of the mixture.

The lubricant added to the powder mix should be one that is non-reactive with any of the components in the mixture and non-oxidizing. The lubricant can either be a liquid or a powder. Furthermore, the lubricant should be capable of volatilizing completely at a temperature within the range of 100° C. and the eutectic temperature of the particular mixture of metals in the powder mix. Preferably, volatilization should occur below about 400° C. This volatilization must be complete without leaving any residue. Organic non-oxidizing materials such as high molecular weight amides have been found to be most suitable for this purpose. Saturated hydrocarbons, such as paraffin oil, having sufficiently high boiling points, are also acceptable. Generally, conventional die lubricants are satisfactory. Representative commercially available lubricants are "Nopcowax" and "Stearotex." A good lubricant is one that facilitates the sliding of metal particles past one another. However, the actual chemical structure of the lubricant is immaterial as long at it possesses the properties indicated above. The lubricant serves to aid in dispersing the powders as well as to lubricate during the compressing stage.

The presence of a lubricant in the mixture, in accordance with this invention, in addition to its lubricating function, also serves another purpose in the process. Under sintering conditions, volatile impurities contained in the starting metals vaporize forming gas pockets within the interstices of the metal. The gas pressure within the metal builds up with the continuance of sintering, as the gas expands, causing the metal to take on an uneven shape or, in many instances, the gases are present in sufficient quantity to break through the surface of the metal causing bulges, holes, and miscellaneous other indentations in a random pattern, thereby detracting from the appearance of the final product and vastly diminishing the strength of the product in unpredictable fashions. When the lubricant is uniformly dispersed within the mixture, the lubricant volatilizes rapidly while the powder mixture is being heated to the sintering temperature. The lubricant, now in the gaseous form, escapes from the product, forming a series of interconnecting pores or vent ports, microscopic in dimension, throughout the metallic structure, at those places where the original lubricant was mixed in and where the gas passed through. This interconnecting pore or ventport structure remains after the lubricant escapes. As sintering proceeds, and gaseous impurities are formed during the sintering operation, gas pressure within the product is not given an opportunity to build up to cause distortion since as gas forms, it escapes through the interconnecting pores previously formed by the volatilized lubricant.

Where the product is not compressed to any appreciable extent, interconnecting porosity need not be supplied in this faction since there will be relatively large spaces at abundant intervals throughout the shaped object, which might, for example, be intended for use as a filter. Therefore, the lubricant can be omitted, if desired, in those instances where the pressure applied during shaping is less than about 3 t.s.i. For greater shaping pressure, the lubricant is a necessary component of the mixture, in an amount of at least ¼% and up to about 5% by weight.

While the invention has been described in terms of aluminum being employed as the primary metal, problems similar to those encountered in aluminum powder metallurgy are also experienced when magnesium, titanium or beryllium are the primary metals. Hence, this invention can be adopted to the use of magnesium, titanium or beryllium in place of aluminum as the primary metal. Also applicable to processing in accordance with this invention are other metallic elements whose oxides are not reducible by hydrogen at the melting point of the element, such as zinc and lead.

Particular combinations of metals are useful for particular purposes. For example, aluminum-copper alloys form particularly strong products when produced in accordance with this invention. In some instances, however, as when cooper content exceeds about 2%, the compact undergoes shrinkage and becomes distorted during sintering at the elevated temperatures employed. The addition of particulate magnesium to the powder mixture, either in the elemental form or as a master alloy with aluminum, in an appropriate amount, serves to reduce or minimize, if not completely eliminate such shrinkage and distortion. In addition, the magnesium adds other beneficial advantages including a speed up of sintering time, reduction in product discloroation, increased strength and toughness of the final product, and increased post-hardening response. When the amount of magnesium is increased beyond the amount that serves to eliminate shrinkage, an expansion effect may be obtained, which is useful for some purposes. For the magnesium to serve to reduce distortion, the powder mixture will contain at least about 2% copper and generally not more than about 8% copper, and magnesium in an amount of at least about 0.05%. Frequently, an amount of magnesium in excess of about 1% is not necessary to control shrinkage but additional amounts, up to about 10% are sometimes required. For some applications, up to 20% magnesium is desirable.

Shrinkage is also noted frequently upon sintering when the alloying additive mixed with aluminum is a zinc-copper alloy such as brass. Silicon also has the effect of counterbalancing shrinkage caused by copper and brass in aluminum systems, but is not by any means as effective in this respect as magnesium.

Some or all of the beneficial effect of magnesium on the aluminum-copper system is lost if the powder mixture contains certain additional metals such as manganese, nickel and iron. These latter metals tend to detract from the tensile strength improvement imparted by the magnesium and therefore are best omitted from the aluminum-copper-magnesium system unless some special effects are needed. In general, in the aluminum-copper-magnesium system, impurities should be minimized. Of course, when aluminum-magnesium master alloys are employed as the source of magnesium, impurities in such alloys are best avoided or in any event kept below about 1% by weight based upon the master alloy.

After the lubricant and the various metals are mixed, they should be thoroughly blended to insure a uniform dispersion. The method of blending is essentially immaterial as is the time of blending. As an example, blending may take place for a period of some 15 to 60 minutes in a double-cone blender or in other suitable devices.

The mixed powder is placed in a suitable mold or die having the particular shape desired of the sintered product. Where a bearing is to be produced, the compacting can be effected by means of a die capable of producing the desired outside diameter, into which a core rod is inserted to establish the required inside diameter. To maintain uniform densification, equal top and bottom punch movements are used to impart a pressure of from about 3 to 40 t.s.i. and thus obtain the desired density, which can range anywhere from as low as 60% or less of theoretical to over 95% of theoretical. Upon release of pressure, the shaped bearing has sufficient integrity. If the final product is desired to be porous for use in filter applications and the like, little or no compaction is necessary. The degree of porosity of the final product can be controlled by the compaction pressure that is applied. In general, if the product is to be porous at all, the compaction pressure must be below 5 t.s.i. In the method of this invention, satisfactory porous products can be obtained even in the absence of any compaction and for many purposes, no compaction at all is preferred.

The actual compaction pressure employed depends upon the density desired of the final product. A porous product is generally one, which after compaction, exhibits a density of up to 85% of the theoretical density for the particular mixture. Theoretical density is calculated by taking into consideration the density and volumetric concentration of each of the individual components present in the mixture before treatment.

The interconnecting porosity, or ventport structure, which constitutes an important feature of this invention, generally ceases to exist at a density greater than about 98% of the theoretical density. Accordingly, the compaction pressure employed in this invention should be such as to not exceed a theoretical density of 98%. For most purposes, the compaction pressure will be below 40 t.s.i. and rarely will the compaction pressure even reach 30 t.s.i. For high strength, substantially non-porous structural parts, the pressures employed are such as to produce a density in the compacted product ranging from about 85% to 98% of the theoretical density.

Thus there can be seen that for porous products, the compaction pressure employed will range from 0 up to about 5 t.s.i. while for high strength, substantially non-porous products, the compaction pressure will range from about 5 up to about 30 t.s.i.

Once the appropriate density is achieved, the compacted mixture must be heated rapidly to the sintering temperature. The rate of heating is very important. The sintering temperature itself is selected by a process of trial and error for each particular system. The particular sintering temperature adequate for any given system is a temperature above the eutectic temperature but below the point of general fusion of the metallic components at the concentration employed and preferably below the fusion temperature of the primary metal. For purposes of this specification, the term "eutectic temperature" is intended to be inclusive of what is normally referred to in metallurgy terminology as the eutectic temperature, the peritectic temperature, or if no binary or polymetal eutectic effect is observed, the fusion temperature of the lowest melting point metal present. If only one metallic element is present, the eutectic temperature is herein arbitrarily defined as a temperature 50° C. below its melting point. For many alloys, optimum conditions are achieved in the range between the solidus temperature and the liquidus temperature.

Within this range of temperatures, generally encompassing a spread of from 70° C. to 200° C., the optimum temperature is that where the interdiffusion or self-diffusion of the alloying constitutents or aluminum particulates, respectively, is rapid enough such that adequate properties are attained in a commercially acceptable time period. For each composition, the particular optimum temperature will vary to some extent within the stated limits and thus must be determined individually. This can be done conveniently by preparing several small samples for a given composition and sintering each in accordance with this invention, using temperature approximately 20 degrees apart within the applicable range and determining from the ultimate tensile strength of the several samples what would be the optimum temperature range. There would normally be about a 15° C. to 20° C. optimum range for each composition. For aluminum-magnesium and aluminum-copper systems, it is generally found that the sintering temperature exceeds about 600° C.

The range of heating to the sintering temperature must be as fast as possible short of causing blistering due to too rapid gas build-up. A rate of heating of from 20° C. to about 600° C. per minute is employed. The minimum effective heat-up rate within this range will depend upon the environment, the composition of the particular mixture and the degree of compaction. Once established for a given mixture, the rate of heating can be maintained for subsequent runs with this mixture. The faster the rate of heating, the better the strength properties of the final product. Offset against this is the tendency of the product to blister or to degrade in properties when the rate is too high. Accordingly, the optimum heat-up rate for any given powder composition must be determined by trial and error. In most cases, the minimum effective rate of heating will be at least about 40° C. per minute, preferably 50° C. per minute, and in many instances, a higher rate of heating is required. A guide for use in determining minimum heat-up rate in a given instance is discussed below.

From the scientific standpoint, it appears that the heat-up rate employed must be at least greater than the rate of oxidation under the heating conditions. However, the actual rate of oxidation is difficult to compute so that trial and error is the most satisfactory method of achieving optimum heat-up rates.

In considering heat-up rate, it is not the rate at which the furnace or other heating means reaches the sintering temperatures that is important, but instead, it is the actual temperature of the sample. This can easily be ascertained by conventional means through the insertion of a thermocouple in the sample. Preferably, the temperature conditions within the furnace and the thickness of the sample should be such that the temperature through the sample is approximately the same.

While it is desirable that the heat-up rate be rapid from room temperature to the sintering temperature, it is only essential that this rapid rate be employed starting from the volatilization temperature of the lubricant or 400° C. whichever is the lower.

A particularly important aspect of the use of magnesium is the fact that when the magnesium content of the aluminum-copper-magnesium powder mixture does not exceed about 0.3%, a slower heat-up rate can be tolerated. Thus when the magnesium content of the mixture is between 0.05% and 0.3%, the heat-up rate frequently can be as low as 5° C. per minute. In many cases better results are obtained with faster heat-up rates, in excess of 20° C. per minute. However, where economic or other considerations dictate a slower heat-up rate, these low magnesium mixtures can be fabricated into products having elongations of at least 5% at the slower rate. Once the magnesium content is in excess of about 0.3%, the heat-up rate must be at least 20° C. per minute as described above. Only in a very few instances, particularly when very fine particle size aluminum powder is employed can the magnesium content be as high as about 0.5% at the slower heat-up rate while still obtaining marginally acceptable properties. For aluminum-magnesium mixtures, in the absence of copper, the heat-up rate should be in excess of 20° C. per minute, regardless of the magnesium content.

The sintering atmosphere must be carefully controlled. The atmosphere must be inert to the components present and non-oxidizing in nature. A reducing atmosphere such as an atmosphere of hydrogen is useful as an added precaution in many instances, but is not indispenable. Such reducing atmospheres are included within the scope of the term "inert atmosphere" since they are only precautionary and not intended to actually take part in the process. Other inert atmospheres such as atmospheres of helium, argon, neon, xenon and krypton can also be used. Nitrogen-containing inert atmospheres such as dissociated ammonia can be used, but generally are not completely satisfactory unless magnesium is one of the metallic components employed.

The moisture content of the atmosphere must be carefully controlled. The atmosphere must be semi-dry but not moisture-free. It is important to have some moisture present in the sintering atmosphere since in the present invention, the presence of a limited amount of moisture appears to have an effect on causing the formation of a finer-grained-stronger product. This is a beneficial effect of the invention since moisture-free atmospheres are expensive and difficult to maintain. The moisture content of the atmosphere employed during the sintering step of this invention should be such that the atmosphere in the environment of the sintered material would have a dew point ranging from −80° F. to −10° F. By "dew point," it is meant that temperature at which the gas in the vicinity of the sintering metal would have to be cooled before water vapor present in the gas would condense.

If the atmosphere has a higher moisture content than a dew point of about −10° F., there is a greater tendency toward oxidation of the sample during sintering which consequently greatly reduced strength properties. Dew points dryer than −80° F. are uneconomic and are best avoided.

It is believed that the presence of a limited amount of moisture results in the formation of an internal skeleton supporting structure which imparts strength at the sintering temperatures and prevents the sagging, and in some cases, even the melting that would normally occur at the sintering conditions. This phenomenon is of greatest importance in connection with relaitvely heavy objects since the sintering of heavy objects under conditions fostering formation of a liquid phase, even briefly, frequently leads to sagging in the prior art methods. The exact point at which such sagging occurs is difficult to predict. Accordingly, the use of the semi-dry atmosphere in accordance with this invention obviates the problem.

The preferred range of dew points is between −20° F. and −60° F. When the powder composition contains small quantities of metals such as magnesium, the dew point can occasionally be raised to be above −10° F. without deletrious results, but even then, dew points much above +10° would not be desired.

The minimum heat-up rate required for this invention varies with the conditions of the sintering environments. As a guide to determining the minimum heat-up rate, an equation has been developed for a pure powdered aluminum-lubricant mixture. Use of this equation results in the minimum heat-up rate possible for a given system. The optimum heat-up rate for the particular system may lie somewhat above this value predicted by the equation, but not below it. In any case, the minimum heat-up rate will not be less than 20° C./min.

Using a ten-minute sintering time, a heat-up rate of 45° C. per minute and a dew point in the furnace atmosphere of −20° F., the following sintering temperatures were employed for particular aluminum-copper mixtures varying weight percent of copper flake and compressed to a density of 95% of theoretical density with ½ of an organic lubricant.

Weight percent copper	Sintering temperature, C.
0	644–648
2	630–632
4	620–635
5	610–615
6	600–605
8	595–600

It is significant to note that in accordance with this invention, there is no need to separately lubricate the forming die or mold in order to prevent lamination to the mold wall, a procedure which has been indispensable in many other processes.

After sintering, the method of cooling the sintered product is relatively immaterial. It can be slowly cooled to room temperature by allowing it to remain in the furnace or it can be rapidly cooled by quenching or the like.

In practice, the process of this invention can be adapted for operation on a continuous basis. After a large batch of powder is mixed, means can be provided for automatically ejecting predetermined quantities into suitable molds, compacting, if desired, and placing the molded product with or without the mold on a conveyor belt and passing the conveyor belt through a series of temperature zones in which, successively, the lubricant is allowed to volatilize, rapid heat-up is promoted in a high temperature zone, the sintering temperature is maintained and thereafter the sample is passed through a cooling or aging zone. The same results could, of course, be achieved in a one-zone furnace.

An actual embodiment of a furnace capable of use in the work described herein has a straight muffle measuring 5″ x 4″ in cross-section, and is provided with a set of baffles at inlet and outlet, the baffles consisting of a set of three curtains made of thin stainless steel strip. The atmosphere used is tank hydrogen with a dew point of −80° F., which is fed into the muffle through an inlet. With such an atmosphere supply, an average dew point of −20° F. or better can be maintained within the furnace muffle. The actual value maintained will depend upon the hydrogen flow rate used.

The furnace is 5 feet in over-all length, and is provided with two sets of silicon carbide heating element arranged above and below the muffle. Parts are carried through the muffle on a continuous belt mounted on pulleys. Three individually controlled heating zones are available, and the power input to each zone may be separately controlled. The center zone has, in addition, separate control of power input to the elements located at the top and bottom of the muffle. This arrangement provides a uniform sintering zone 21 inches in length, and allows for a maximum of about 8 minutes sintering time when belt speed is adjusted to provide a heat-up rate of 50° C. per minute.

The following examples represent, in the opinion of the inventors, the best mode of carrying out their invention.

Example 1.—(Self-lubricating bearings)

A mixture consisting of Type A aluminum powder containing additives of the type and quantity shown in Table II below, is mixed for ½ hour and is compacted in the form of ½″ ID x ¾″ OD x ½″ long sleeves, to a density of 77% to 78% of theoretical density. The green compacts are then continuously sintered in a hydrogen atmosphere using a belt-type sintering furnace. Belt speed is adjusted so as to obtain a heat-up rate to sintering temperature of approximately 150° C. per minute. The dew point in the furnace was −20° F.

The sintered parts are found to be possessed of excellent crush strength, on the order of 1,000 to 13,000 p.s.i. for the aluminum-4 weight percent copper alloy, and 35,000 p.s.i. for the aluminum-4 weight percent copper-0.6 weight percent magnesium alloy, as shown by the data of Table I. When sized and oil-impregnated, such parts exhibit excellent bearing characteristics, being capable of operating for 1000 hours at a PV factor of 50,000 or better, this equalling or bettering the performance of porous bronze bearings currently being used for such purposes. The lubricant was a high molecular weight organic amide.

TABLE I.—FABRICATION CONDITIONS AND PROPERTIES OF POROUS ALUMINUM—COPPER AND ALUMINUM—COPPER—MAGNESIUM ALLOY BEARINGS

Bearing Composition	Type A Aluminum Powder, 2 w/o 100/200 mesh copper powder, 2 w/o copper flake, 2 w/o lubricant.	Type A Aluminum Powder, 2 w/o 100/200 mesh Cu 2 w/o copper flake, 0.6 w/o −325 mesh magnesium powder, 2 w/o lubricant.
Sintering furnace used	Continuous belt type with straight through muffle.	Continuous belt type with Hump muffle.
Furnace temperature ° F	1,100	1,120.
Sintering time	.049 f.p.m. belt speed corresponding to 20.4 minutes in a 1 foot sintering zone.	0.293 f.p.m. belt speed corresponding to 3.4 minutes in a 1 foot sintering zone.
Density before sintering, percent	77	78.3.
Density after sintering, percent	74.9	76.3.
Dimensions before sintering	.753 OD x .4975 ID x .493 long	.753 x .4975 ID x .539 long.
Optimum dimensions after sintering.	Top: .757 OD to .757 OD, .5005 ID to .5000 ID x 0.502″ long. Bottom: .757 OD to .756 OD, .4995 ID to .5000 ID.	Top: .753 OD to .7540 OD, .4980 ID to .4985 ID x 0.548″ long. Bottom: .7550 OD to .7555 OD, .499 ID to .500 ID.
Interconnecting porosity, as sintered, percent.	About 20	20.
Crush strength at optimum sintered dimensions, p.s.i.	11,320–12,850	Approx. 35,000.

Example 2.—(High-density structural parts)

A mixture consisting of Type A aluminum powder, copper flake in amounts up to 6 w/o, and ½ w/o lubricant, is mixed for ½ hour. The mixture is pressed to 95% of theoretical density in the form of 2″ long tensile bars and the bars continuously sintered at 625° C. for about 8 minutes in a hydrogen atmosphere using a belt-type sintering furnace. Belt speed is adjusted so as to obtain a heat-up rate to sintering temperature of approximately 50° C. per minute. The dew point within the furnace is −20° F.

The resultant sintered products are found to have excellent mechanical properties, as shown by the data of Table II below, ultimate strength and elongation varying from 10,1000 p.s.i. and 35% at 0 w/o copper, to 52,000 p.s.i. and 13% at 6 w/o copper.

TABLE II.—MECHANICAL PROPERTIES OF SOLUTION TREATED AND AGED SINTERED ALUMINUM PREPARED FROM TYPE A ALUMINUM POWDER CONTAINING VARYING QUANTITIES OF COPPER FLAKE

Copper Flake Content w/o	Ultimate Tensile Strength, p.s.i.	Elongation, percent	Hardness Rockwell H
0	10,100	35	18
1	18,400	27	60
2.5	32,000	18	95
3	34,300	19	96
4	37,600	14	102
5	46,600	14	
6	52,000	13	

Examples 3 and 4

An aluminum powder, all of which passed through a 100 mesh sieve and containing 35.6 weight percent of material capable of passing through a 325 mesh sieve was mixed for 30 minutes with 4% by weight of copper flake, 0.3% by weight of −325 mesh helium-atomized magnesium and 0.5% by weight of Nopcowax (a fatty amide) lubricant. The mixture was divided into several parts and each part was compacted in a shaped die to 95% of its theoretical density, using a compacting pressure of 20 t.s.i. the compressed products being in the shape of ⅛″ x ½″ x 4″ bars. Each of the bars was then heated separately to the sintering temperature at a rate given below in an atmosphere of dissociated ammonia having a dew point of −20° F. to −30° F. in the sintering zone, sintered for a period of time, and then cooled. The physical properties of the sintered material are set out below, illustrating the very significant effect of heat-up rate and sintering conditions on ultimate properties.

Example	° C./Min.	Dew Point of Furnace Atmosphere, ° F.	Time Above 600° C., Min.	Maximum Temperature, ° C.	Time at Maximum Temp., Min.	Ultimate Tensile Strength, p.s.i.	Elongation Percent in 1″
3	89	−20 to −30	4	646	Less than 1 minute	28,000	12
4	59	−21 to −27	16	654	do	22,600	12

Example 5

An aluminum powder (Alcoa 120) was mixed with 4% copper flake and ½% Nopcowax and a quantity of magnesium powder as set forth below and compressed to 95% of theretical density, using a compacting pressure of approximately 20 t.s.i., the compressed products being in the shape of ⅛″ x ½″ x 4″ bars. Each of the bars was then heated separately in a hump furnace to the sintering temperature of aproximately 645° C. to 650° C. in an atmosphere of dissociated ammonia having a dew point in the vicinty of the sintering product of approximately −44° F. Each sample was maintained at the sintering temperature for about 5 minutes and then removed and allowed to cool at room temperature. Physical properties observed immediately after sintering and cooling were as follows:

Magnesium Content, percent	Heat-up Rate ° C/min. from 400° C. to Sintering Temperature	Elongation percent in 1 inch	Ultimate tensile Strength, p.s.i.
0.1	7	12.5	24,200
0.1	53	18	23,900
0.1	80	15	24,000
0.3	7	5.5	21,100
0.3	53	9	25,100
0.3	80	11	23,700
0	31.5	3.5	15,800

After aging the samples at room temperature for 48 hours, the following physical properties were observed:

Magnesium Content, percent	Heat-up Rate ° C/min. from 400° C. to Sintering Temperature	Elongation percent in 1 inch	Ultimate tensile Strength, p.s.i.
0.1	7	9.5	23,600
0.1	53	17	24,100
0.1	80	15	23,500
0.3	7	3	22,000
0.3	53	5.5	26,800
0.3	80	6	25,200
0	31.5	5	10,300

The foregoing illustrates the unusual effect of a very small quantity of magnesium on aluminum-copper systems and further shows that at magnesium concentrations below about 0.3%, a heat-up rate of less than 20° C. per minute can be employed although appreciably better elongations are obtained at high heat-up rates.

Example 6

An aluminum powder (Alcoa 120) was mixed with ½% Nopcowax and with varying quantities of magnesium powder as set forth below and compressed to 95% of theoretical density using the necessary compacting pressure, less than 30 t.s.i., the compressed products being in the shape of ⅛" x ½" x 4" bars. Each of the bars was then heated separately in a hump furnace to the sintering temperature of approximately 645° C. to 650° C. An atmosphere of dissociated ammonia was maintained in the furnace by passing dissociated ammonia through at a rate of 300 standard cubic feet per hour. The dew point in the vicinity of the sintering product was approximately −58.5° F. Each sample was maintained at the sintering temperature for about 3 minutes and then removed and allowed to cool at room temperature. Physical properties observed immediately after sintering and cooling were as follows:

Magnesium Content, percent	Heat-up Rate ° C/min. from 400° C. to Sintering Temperature	Elongation percent in 1 inch	Ultimate tensile Strength, p.s.i.
0.1	55	8.5	9,800
0.1	72.5	17	10,900
0.1	87	16	12,900
0.1	92.5	27	12,200
0.3	55	6	8,900
0.3	72.5	10	10,000
0.3	87	14	12,800
0.3	92.5	19	12,400
1.0	55	2.5	6,000
1.0	72.5	5	8,700
1.0	87	5	10,000
1.0	92.5	10	14,000
2.0	55	3.5	8,400
2.0	72.5	2	6,400
2.0	87	3.5	11,100
2.0	92.5	7	14,300

The foregoing illustrates some of the many combinations of elongations and tensile strengths that can be obtained at varying heat-up rates and magnesium contents.

What is claimed is:

1. In the method of preparing a cohesive and finely grained metallic article containing more than 50% by weight of a primary metal whose oxide is not reducible by hydrogen at the melting point of the metal wherein the primary metal in particulate form is shaped to achieve the desired configuration and density, and the shaped mass is heated to sintering temperature, the improvement which comprises: (a) shaping the mass to a density not exceeding 98% of the theoretical density characterized by interconnecting porosity when any lubricant therein has been volatilized by heating the mass to 400° C. by using an amount of the lubricant ranging from 0 to 5% by weight and an amount of compaction pressure ranging from 0 to 30 t.s.i., and (b) maintaining the shaped metal in a moisture-containing atmosphere having a dew point range of −60° F. to −20° F. while heating the shaped metal having interconnecting porosity at a rate of at least 20° C. per minute up to the sintering temperature of the metal.

2. The method according to claim 1 in which the primary metal is aluminum.

3. The method according to claim 1 in which the metallic article consists essentially of a aluminum.

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