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POWDER METALLURGY

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This invention relates to the manufacture of articles from powdered materials, more specifically, from powdered metals, particular reference being had to the production of tool elements, bearings, etc., from powdered refractory metal compounds, such as carbides, nitrides, borides, etc., of tungsten, tantalum, boron and the like, though not limited thereto.

The present invention is in part an improvement over the invention disclosed in the copending application of William W. De Lamatter, Serial No. 332,066, filed April 27, 1940, entitled, "Hard metal articles and methods of making," now Patent 2,286,672, granted June 16, 1942.

In fashioning articles from powdered metals, or their compounds, it is customary to reduce the powder to the necessary particle size, and, after admixing a fluid lubricant or binder therewith, to press the powder, under high pressure, in molds which impart the final, or an intermediate, shape thereto. Thereafter, the pressed body is usually heated to the point where its particles sinter, frit, or otherwise autogenously coalesce.

The use of lubricants in powder metallurgy is very essential when products are made, inter alia, from the sintered metallic carbides; that is, if maximum density and minimum porosity are to be achieved in the ultimate section. The lubricant aids in overcoming the friction between adjacent particles when these are confined in a mold and subjected to pressure, permitting the confined particles to slip, relative to each other, into positions of maximum compactness. If this friction is not properly minimized, the resulting product will not be as dense as it should be, nor of uniform density, nor free from porosity. In some cases, another function of such a lubricant is that it acts as a temporary binder, thus assisting in initially holding the particles together.

In the co-pending application above referred to, there is disclosed the use of a binder-lubricant, such as paraffin, dissolved in carbon tetrachloride—or glycerin dissolved in alcohol—in the production of articles from metallic powder, notably tungsten carbide powder, having a particle size such as would pass a screen having 325 meshes per square inch. The carbon tetrachloride, or the alcohol, is the solvent by which the lubricant is rendered sufficiently fluid as to be distributed fairly uniformly throughout the mass of powder to which it is introduced, after which the solvent is driven off, leaving the powdered particles coated with paraffin or glycerin, as the case may be. Although powder thus treated may be pressed and sintered with some degree of suc-

cess, it has been found that it cannot be invariably depended upon for the attainment of maximum density and freedom from porosity, which it is one of the objects of the present invention to achieve.

It has been found that if a powder which is to be agglomerated by pressure is first mixed with a wetting agent, dispersant or deflocculent, applied at the time the lubricant is applied, as by being admixed with the lubricating solution, the objections hitherto encountered in the handling and pressing of the metallic powder are eliminated, and the uniformity and density of the resulting product is much improved.

It is, therefore, an object of the present invention so to treat powders for use in the field of powdered metallurgy, which are to be compressed to shape and agglomerated, that they may be handled with facility, compressed to maximum and uniform density, and be entirely free of voids or porosity.

Although many recognized dispersing agents or deflocculents will undoubtedly be found suitable for the purposes hereof, and the manner of their application will admit of much variation according to the needs and convenience of each particular case, we have chosen to set forth a specific application, illustrative of a preferred embodiment of the present invention, as an aid to the explanation, rather than as a limitation, thereof.

As is set forth in the co-pending application, above identified, paraffin is dissolved in carbon tetrachloride, and is then admixed with tungsten carbide powder of requisite fineness, after which the carbon tetrachloride is evaporated off, leaving a lubricating film of paraffin adherent to the particles. In the present invention, assuming these same conditions to obtain, we have found that by combining a wetting—or dispersing—agent, such as butyl acetate, with the solvent of the lubricant, a remarkable improvement in the subsequent handling and ultimate properties of the powder, in uncompressed and compressed form, is realized.

In preparing the lubricating solution, tests have indicated that paraffin should be present relative to the liquid vehicle in amounts not more than 1 part paraffin to 150 parts liquid by volume, nor less than 1 part paraffin to 300 parts liquid by volume. Within this range, the solvent, such as carbon tetrachloride, need be present in no greater quantity than is required completely to dissolve the paraffin, e. g., 5-10 ccs. of carbon tetrachloride per gram of paraffin.

The remainder of the solution may be composed of a suitable wetting—or dispersing—agent, such as butyl acetate. A satisfactory solution has been made by dissolving one gram of paraffin in 250 ccs. carbon tetrachloride, to which is added one-half again as much, by volume, of butyl acetate (66⅔% carbon tetrachloride-paraffin solution, 33⅓% butyl acetate, by volume). Because of its relatively high cost as compared to the other ingredients, it is advisable to use carbon tetrachloride in an amount not greater than that required to dissolve the paraffin or other lubricant; thus the balance of the liquid vehicle may be composed of the dispersant. In all cases there must be a substantial amount of liquid vehicle present in order to insure adequate wetting of the mass of powder and to insure proper distribution of the lubricant therethrough.

A solution compounded as above is applied to the powder to be treated so that a sufficient quantity of the solution is present as to insure the thorough wetting of each particle. This may be easily accomplished by placing the powder in a container, pouring in a sufficient quantity of solution to cover the powder, and briefly stirring to insure wetting of the particles. In this respect, prolonged mixing is not required, since the butyl acetate has the property of causing the solution to penetrate completely the interstitial space of the powder, insuring that each particle thereof is thoroughly wetted, whereby upon subsequent evaporation of the liquid vehicle of the lubricating solution, a residue of lubricant (paraffin) is disposed uniformly about each particle.

The surplus liquid is drained from the powder, which is then dried by exposure to air, or by the application of gentle heat insufficient to burn off the lubricant (paraffin) in a non-oxidizing atmosphere. It is important that the powder, prior to being subjected to pressure, be substantially dry, since if an appreciable amount of moisture is present during the pressing operation, the powder will be rendered fluid under pressure and tend to squirt from the mold, or will be maintained at something less than maximum density by the spacing effect of the occluded moisture. Articles so formed with a moisture content, if subjected to heat treatment, as in sintering operation, either explode or, if the moisture can find its way out without disrupting the body, become porous and filled with interstitial voids.

Because of the foregoing, it is essential that the powder be rendered substantially dry, and by this is meant insufficient moisture to characterize the powder as being even damp. Notwithstanding this degree of dryness that is imparted to the powder, the odor of butyl acetate has been found to linger even after subsequent crushing of the lumps and screening of the powder is had, thus leading to the conclusion that all of the butyl acetate is not evaporated off.

Powder treated in this manner will be found to have the lubricant, or binder, such as paraffin, distributed with great uniformity throughout its mass. Upon subsequent pressing, the powder will attain maximum compactness at the minimum pressures, as set forth in my copending application, and in the case of tungsten carbide powder which is sintered after compression, will sinter into an article having maximum density and great uniformity.

Although the above forms of the invention contemplate the inclusion of the dispersant or

wetting agent in the lubricating or binding solution, any other satisfactory method, as by infusing the powder with the dispersant preparatory to applying the lubricant or binder, is admissible, so long as the dispersant is present to assist in the distribution of the latter. Powder, particularly that of extremely fine particle size, first treated with the dispersant, is believed to exhibit better handling properties, since the tendency for the powder to ball-up or cake is minimized thereby.

Butyl acetate has proved satisfactory as one dispersant or wetting agent. The invention is not limited to this, however, since the higher alcohols, e. g., amyl- and butyl-alcohol, have proved equally satisfactory.

The acetates and alcohols do not dissolve such lubricant binders as paraffin and, accordingly, must be combined with a suitable solvent for this purpose, such as carbon tetrachloride, already mentioned herein. Other petroleum compounds, such as those commercial solvents known as hydrogenated petroleum, may be used in lieu of the carbon tetrachloride—"Solvesso" being a well known commercial example of such a solvent.

Tungsten carbide articles made in accordance herewith have been found to have a density at 60° F. of 16.05, which is only .01 of a point lower than the standard of 16.06 given in the handbooks as the density of pure tungsten carbide (W₂C). This compares with a density of 15.70 given for tungsten carbide of the double carbide form (WC) and is considerably higher than the densities of compressed and sintered tungsten carbide bodies made by any known methods, whether with a binder, or purportedly without a binder.

While we have emphasized one specific embodiment of our invention, it will be understood that we do not wish to be limited thereto, since various modifications and changes will readily suggest themselves to those skilled in the art and many of such modifications and changes may be made without departing from the scope of our invention as defined in the appended claims.

In the appended claims "lubricant" is intended also to include binders, and other similar ingredients added by infusion to powders to be pressed.

We claim:

1. In the production of hard metallic bodies of great and uniform density, the improvement which comprises providing a mass of powdered tungsten carbide having the requisite particle size; thoroughly admixing a lubricant with the powder in the presence of a wetting agent effective to wet the powder; compressing said powder into a mold to integrate it, and, thereafter, subjecting said integrated mass of powder to sintering heat autogenously to coalesce its particles.

2. In the production of hard metallic bodies of great and uniform density from powdered tungsten carbide, the step which includes impregnating a mass of tungsten carbide powder with a dissolved lubricant in the presence of a wetting agent effective to wet the powder; drying said powder to eliminate the solvent of said lubricant and at least the major part of said wetting agent; subjecting the powder to high pressure to form a concrete mass, and, thereafter, firing the concrete mass.

3. In the production of metallic bodies from metallic powders, the step which includes infus-

ing a mass of powder with a lubricant in the presence of butyl acetate preparatory to pressing the powder.

4. In the production of metallic bodies from powdered metals, the step which includes infusing a mass of powder with a solution containing a lubricant-solvent, a lubricant-solute, and butyl acetate, preparatory to pressing the powder.

5. In the production of metallic bodies from powdered metals, the step which includes infusing a mass of powder with a solution containing carbon tetrachloride, paraffin, and butyl acetate.

6. In the production of metallic bodies from powdered metals, the step which includes infusing a mass of powder with a solution containing: paraffin—in quantities not exceeding approximately 2 parts of paraffin to 300 parts of liquid, nor less than approximately 1 part of paraffin to 300 parts of liquid, by volume—; carbon tetrachloride in sufficient amount completely to dissolve said paraffin; and an amount of butyl acetate sufficient to make up the remainder of the 300 liquid parts, by volume.

7. In the production of metallic bodies from powdered metals, the step of preparing such powder for subsequent pressing which includes infusing the powder with a solution containing 1 gram of paraffin for every 250 cubic centimeters of carbon tetrachloride (66⅔% by volume), and butyl acetate (33⅓% by volume).

8. A pressed, sintered tungsten carbide powder body of great uniformity, density, strength and hardness, which, before being committed to formative pressure, was infused with a solution con-

taining a dissolved lubricant and a dispersant characterized by a density at 60° F. of 15.50 or greater.

9. A pressed, sintered body of tungsten carbide powder, having uniformity, strength, and hardness, characterized by a density at 60° F. of 16. or greater.

10. The method of compacting discrete powdered materials into concrete masses which includes impregnating a powder with a solution containing a lubricant for facilitating relative motion between the powder particles moving in response to compression, a liquid vehicle-solvent for dissolving said lubricant that is substantially inert toward said powder and a wetting-agent capable of effecting the distribution of said solution uniformly throughout the powder mass; bringing the powder to substantial dryness, and, thereafter, compressing said powder.

11. The method of compacting discrete powdered materials into concrete masses which includes impregnating a metallic powder with a solution containing a lubricant for facilitating relative motion between the powder particles moving in response to compression, a liquid vehicle-solvent for dissolving said lubricant that is substantially inert toward said powder, and a wetting-agent capable of effecting the distribution of said solution uniformly throughout the powder mass; substantially drying said powder, and, thereafter, molding the powder under high pressure to form a concrete metallic mass.

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