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SILICON-ALLOYED CORROSION-RESISTANT METAL POWDERS AND RELATED PRODUCTS AND PROCESSES

George Stern, Mamaroneck, Seymour J. Sindeband, Chappaqua, and Joseph P. Scanlan, Yonkers, N. Y., assignors to American Electro Metal Corporation, Yonkers, N. Y., a corporation of Maryland

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This invention relates to readily compactible and moldable powders which resist corrosion, shaped bodies of such powders, and methods for producing the same, some aspects of the invention being of a broader scope.

Among the objects of the invention are powders of metal particles which are in a readily deformable plastic condition and may be compacted into relatively strong metal bodies with relatively low pressure, at least the surface layer of the metal powder particles having alloyed therewith silicon from a gaseous or liquid silicon compound at an elevated temperature.

A particular object of the invention is such readily compactible moldable powder containing essentially powder particles of at least one metal of the group consisting of iron, nickel and cobalt, and of mixtures and alloys of said metals, which particles are in a readily deformable, relatively soft plastic condition, at least the surface layer of said particles having alloyed therewith silicon which has been deposited thereon at an elevated temperature from a silicon compound.

Another object of the invention is such silicon-surface-alloyed soft moldable metal powder combined with similar surface-alloyed powder particles of at least one metal of the group consisting of molybdenum, tungsten, chromium, vanadium, manganese, or mixtures or alloys thereof.

Further objects of the invention include compacted bodies made of such surface-alloyed metal powder particles, methods for the economical production of such surface-alloyed soft moldable metal powder particles, and economical production of compacted bodies from such powder particles of the invention.

The foregoing and other objects of the invention will be best understood from the following description of specific exemplifications thereof.

For years past various corrosion-resistant products such as gears, valve parts, bearings, filters, and the like, have been made of stainless steel powders by powder metallurgy techniques. The stainless steel powders generally used for such bodies have been obtained from previously prepared stainless steel ingots, for instance, by subjecting a stainless steel ingot to grain boundary corrosion followed by disintegration, or by melting a stainless steel ingot and atomizing it in the molten state. The so-produced particles of such stainless steel powders are of high density and hardness, and require high-molding pressure of at least 40 to 45 t. s. i. (tons per square inch), which is much too high for economical production of molded products, and results in rapid wear and frequent break-down of the molding dies. As a result, the production of molded products from stainless steel powders has found only limited use.

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What has been said above about stainless steel powder, which is an alloy of iron, nickel and chromium, applies also to alloys of iron, cobalt and nickel with aluminum.

According to the invention the foregoing difficulties heretofore encountered in the production of corrosion-resistant ferrous metal bodies by powder metallurgy techniques are overcome by the provision of a readily compactible and moldable metal powder of relatively soft ferrous powder particles, at least the surface layer of the powder particles having combined therewith silicon which has been deposited thereon from a silicon compound at an elevated temperature so as to render the powder particles corrosion-resistant, the surface-alloyed metal powder particles being sufficiently soft and plastically deformable to make it possible to compact them with relatively low pressure, for instance, only 25 to 30 t. s. i. into bodies of even greater strength than molded bodies made by compacting prior art powders of corresponding composition with a molding pressure of at least 40 to 45 t. s. i.

Various commercially available soft, plastically readily deformable iron powders are suitable for producing corrosion-resistant soft moldable metal powders of the invention. Among such available and suitable soft iron powders is sponge iron powder having a carbon content of .1 to .2% (unless otherwise specifically stated, all proportions are given herein by weight), electrolytically produced iron powder, carbonyl iron powder, eddy mill iron powder, and atomized molten iron powder of silimar carbon content. The following impurities present in such available powders, to wit, .3% Mo, .2% Si, and other minor impurities usually present therein, do not impair their usefulness for practicing the invention.

In addition, many relatively hard and difficult to compact fine iron powders containing up to about .2% carbon will, in general, when subjected to the silicon-alloying process of the invention herein described, become softened and acquire characteristics which render them readily moldable.

In accordance with a phase of the invention—based on the original concept of producing silicon-surface-alloyed ferrous iron powder—relatively soft powder particles of different metals, to wit, one or more metals of the group including

iron, nickel and cobalt, are combined in proper proportion, and a desired proportion of silicon from a gaseous compound is deposited on and diffused into the interior of such metal powder particles so as to yield soft powder particles combining silicon with the other metal or metals in proportions required for producing alloyed bodies of desired characteristics and containing silicon and such metals in the desired proportions.

Any of the known methods for depositing silicon on the surface of a metal body heated to an elevated temperature below the melting temperature of the body and of silicon may be used for depositing on and diffusing silicon into metallic powder particles in accordance with the principles of the invention.

Among the known silicon-alloying processes, one that was found particularly suitable resides in subjecting the metal particles to the action of a mixture of gaseous SiCl and H_2 at a temperature in the range from 900 to 1500° C. It is believed that the resulting reaction takes place either according to the equation

$$SiCl_4+H_2 \rightarrow Si+HCl \quad (1)$$

or according to the equation

$$SiCl_4+\text{metal} \rightarrow Si+\text{metal chloride} \quad (2)$$

or simultaneously according to both these equations.

In any case, the silicon atoms of the gaseous silicon compound are deposited on the surface of the metal powder particles, and diffuse into the interior and alloy with metal of the particles at the temperature of the treatment.

In accordance with one phase of the invention, the soft iron particles are first sintered into sinter cake pieces within a reducing temperature, such as pure dry hydrogen, or purified dry cracked ammonia, at an elevated temperature so as to produce a porous sinter cake of low density, not higher than about 4 to 5 g./cc., and of low strength not higher than modulus of rupture of about 6500 to 7000 p. s. i. The powder particles of such porous low density sinter cake pieces produced by the initial sintering treatment, may be readily surface-alloyed with silicon by known processes for depositing silicon from a gaseous or liquid silicon compound at an elevated temperature at which the deposited silicon diffuses into the interior of the powder particles. The porous low-density sinter cake pieces subjected to such silicon-surface-alloying treatment do not undergo any material increase in density and strength when subjected to this surface-alloying treatment, and they may be readily comminuted with minimized work hardness into minute soft powder particles of the required size range and high degree of plastic deformability which makes it possible to compact the resulting silicon-surface-alloyed ferrous powder particles with a pressure of only 50,000 p. s. i. into strong green metal powder compacts having a nodulus of rupture of 400 p. s. i. or more, without adding any binder to the powder particles.

In accordance with another phase of the invention, powder particles of the oxides of ferrous metals, such as iron, nickel and cobalt, or mixtures of such oxides, or mixtures of such oxides and metals—with or without additional mixtures of powders of oxides of metals of the group consisting of molybdenum, tungsten, chromium, vanadium, and manganese, or powders of these metals, or mixtures of powders of these metals and their oxides—are initially sintered into porous sinter cake pieces at an elevated temperature within a reducing atmosphere so as to produce sinter cake pieces of the reduced metal powder particles having a low density not higher than about 4½ to 5 g./cc., and of low strength not higher than a modulus of rupture of about 7000 p. s. i. The metal powder sinter cake pieces obtained by such initial reducing treatment are then surface-alloyed with silicon from a silicon compound at an elevated temperature at which the deposited silicon diffuses into the interior of the metal powder particles. The low-density sinter cake pieces subjected to such silicon-surface-alloying treatment do not undergo any material increase in density or strength, and may be readily comminuted into minute powder particles of the required size range which have only a minimized work hardness, and exhibit a high degree of softness and deformable plasticity which makes it possible to compact the resulting silicon-surface-alloyed minute metal powder particles with a pressure of only 50000 p. s. i. into strong green powder compacts having a modulus of rupture of 400 p. s. i. or more, without adding any binder to the powder particles. The surface-silicon-alloyed metal powders produced in the manner described above from oxides of the different metals will yield a surface-alloyed powder body, the individual particles of which are alloys of the different metals and substances corresponding to the different metals or oxides used for making the initial sinter cake pieces and the proportion of silicon deposited on the sinter cake particles.

A convenient way for producing such sinter cake pieces for the foregoing treatment is to mix the powders of the metal oxides with a lubricant and binder and pellet the mixture into pellets, the pellets containing enough of the lubricant and binder which decomposes and is driven off at the sintering temperature so that the resulting sinter cake pellets or pieces have the required high porosity and the required low density and low strength.

Without in any way limiting the scope of the invention, there will be hereinafter described examples of satisfactory procedures for producing soft silicon-surface-alloyed metal powders of the invention from commercially available raw material.

There will now be described, by way of example, a satisfactory procedure for producing silicon-surface-alloyed corrosion-resistant soft iron powder of the invention from commercially available sponge iron powder having a carbon content of about .15%.

A typical commercial powder of this type used in the process consisted of a mixture of minute powder particles having the following particle-size distribution typical of moldable metal powders: 21%—100, +150 mesh; 30%—150, +200 mesh; 13%—200, +250 mesh; 14%—250, +325 mesh, and the balance —325 mesh powder.

The iron powder is subjected to an initial treatment which sinters it into a porous iron powder cake of low density and high porosity so as to permit ready surface-alloying of the cake powder particles with silicon deposited thereon. Such porous low-density sinter cakes may be produced as follows: A layer of the iron powder about ⅛ to ¼ inch is deposited into sinter boats of suitable heat-resistant metal such as iron-chromium sheet metal, without in any way compacting the deposited iron powder. The interior surface of each sinter boat is coated with a stick-suppress-

ing or release medium such as a water suspension of magnesium hydroxide to permit ready separation of the sintered powder body from the boat after the sintering operation. The powder within the boat is then treated within a furnace in a reducing atmosphere, such as purified dry hydrogen or purified cracked ammonia, at a temperature of about 800° to 1050° C. for one-half to three hours, and then permitted to cool within the same atmosphere to room temperature. In general, such treatment at a temperature between about 900° to 1000° C., such as 950° C., for one hour is sufficient for sintering commercial sponge iron powder into a sinter cake having a density of 1.7 to 4 g./cc., which is readily frangible into lumps or sinter cake pieces suitable for undergoing the desired silicon-surface-alloying treatment.

A good way for carrying on the foregoing sintering treatment is to push the powder containing boats through a tunnel furnace within which is maintained the desired reducing atmosphere, the heat treatment being followed by cooling within the same atmosphere as a part of a continuous process, in the course of which the boats are moved from one end of the furnace to the other.

After completion of the initial sintering treatment, the iron powder sinter cakes are removed from the boats and are broken into sinter cake fragments or pieces of a size suitable for the subsequent siliconizing treatment. Sinter cake pieces having cross-sectional dimensions in the range between about ⅛ inch to about 1¼ or ⅜ inch have been found very suitable for the subsequent surface-alloying treatment.

An example of a satisfactory procedure for surface-alloying such iron powder sinter cake pieces with silicon will now be described: The porous iron powder sinter cake pieces are packed within a pack mass which is effective in depositing silicon on the sinter cake powder particles. The pack may consist, for instance, of 50% by volume ceramic lumps, such as porcelain pieces, the balance a silicon alloy such as ferrosilicon containing about 40 to 70% (by weight) silicon.

The sinter cake pieces packed with porcelain and ferrosilicon are placed within treatment baskets of suitable heat-resistant metal, such as a chromium-nickel-iron alloy, and the packed treatment baskets which are placed in a retort. The contents of the retort are heated to an elevated temperature in the range between about 900 and 1100° C., and an atmosphere containing pure dry hydrogen and dry hydrogen chloride gas, is passed through the retort for producing reactions causing silicon atoms to be deposited on the metal powder particles of the sinter cake lumps, and to diffuse into the interior of the metal particles and alloy therewith. In such surface-alloying treatment, the hydrogen chloride gas passing through the retort interacts with the ferrosilicon to form silicontetrachloride gas. The silicontetrachloride gas is decomposed at the surface of the powder particles, and the silicon atoms deposit on the surface and diffuse into the interior of the metal powder particles and alloy with the metal of the particles.

According to the silicon content in the treated powder, the time of treatment may be varied between about 10 and 20 hours. The temperature of the treatment may be varied between about 900 and 1100° C., and must be well below the melting point of the ferrosilicon of the pack.

By way of example, the following surface-alloying procedure gave satisfactory results: The broken up sinter cake lumps were packed in circular baskets having a diameter of 22" and a height of 10". Four such baskets were stacked upon each other in a closely fitting retort of 60" height. A mixture of 3 parts of gaseous HCl and 20 parts of H_2 was passed at 1050° C. through the retort, first for 2 hours at a rate of 40 cu. ft. per hour, and then for 10 more hours at a rate of 20 cu. ft. per hour. The foregoing treatment yielded iron powder sinter cakes with a silicon content of about 12%. After being subjected to such silicon-surface-alloying treatment, the resulting sinter cakes have about the same density and the same strength or modulus of rupture which they exhibited prior to the silicon-surface-alloying treatment. Because of the low density and strength of the silicon-surface-alloyed sinter cake pieces, they may be readily comminuted with minimized work hardness into soft metal powder particles of a size range and high degree of plastic deformability required for compacting the powder particles into strong pellets with relatively low pressure of only 50,000 p. s. i. into green compacts having a modulus of rupture of at least 200 and higher.

By way of example, surface-alloyed iron powder sinter cakes prepared in the manner described above, were disintegrated by a disc crusher into silicon-surface-alloyed powder particles, 90% of which passed through a 100 mesh screen, the powder containing about 8% −325 mesh particles, about 50%−100, +200 mesh particles, balance −200, +325 mesh particles. Green tests of such silicon-surface-alloyed powder particles compacted—without any binder—under a pressure of 25 t. s. i. exhibited an average modulus of rupture of about 850 p. s. i. (pounds per square inch).

The desired surface-alloying action may be obtained without packing materials by the following procedure. The sinter-cake pieces are placed in a retort and a stream of hydrogen and a gaseous silicontetrachloride is passed through the retort while its contents are maintained at an elevated temperature. Satisfactory results were obtained by passing gas mixtures containing from 4 to 30% (by volume) of $SiCl_4$ through the retort at temperatures between about 900° and 1160° C., and at rates in the order of 30–60 cc. per second for periods ranging from 2 to 15 hours. The desired gas mixture was produced by bubbling hydrogen through liquid silicontetrachloride at room temperature, at which the vapor pressure of $SiCl_4$ is comparatively high so that sufficient amounts thereof are carried with the hydrogen into the retort.

The silicon-surface-alloyed iron powder particles of the invention, produced by the treatment of the invention, have a spongy character, tentacle-like shape. Green compacted bodies produced from such powders exhibit excellent corrosion-resistance under prolonged salt-spray tests, and they also resist attack by boiling dilute HNO_3 and H_2SO_4.

Similar results are obtained with other types of soft, plastically deformable iron powders subjected to the silicon-surface-alloying treatment of the invention. In fact, as explained above, many relatively hard and difficult to compact iron powders will, in general, when subjected to the silicon-surface-alloying treatment of the invention of the type herein described, become softened and acquire the high degree of plastic deformability which renders them readily moldable into strong green compacts of high strength of at least 200 p. s. i.

The foregoing rupture test data were obtained by test equipment and test methods described in the article of J. P. Scanlan and R. P. Seelig in Powder Metallurgy Bulletin, 44, p. 128 (1949), using test bars 1½'' long, $\frac{7}{16}$'' wide, and ¼'' thick.

The test results given above are representative of the characteristic distinguishing silicon-surface-alloyed ferrous powders of the invention over prior art powders of corresponding composition. Thus, soft silicon-surface-alloyed and corrosion-resistant ferrous powders of the invention differ from heretofore available corrosion-resistant stainless steel powders by the fact that green compacts made from powders of the invention have a materially greater rupture strength, at least two to four times greater than similar prior art stainless steel powders.

A distinguishing characteristic of the corrosion-resistant silicon-surface-alloyed soft ferrous powders of the invention is the fact that when compacted into a green test bar body of the dimensions given above under pressure of 25 p. s. i. with no lubricant or binder, such compacted green body exhibits a modulus of rupture several times greater than a similar body produced by compacting prior art silicon steel powders, under the same pressure. In particular, such green test bar compacts made from powders of the invention have a modulus of rupture of at least 300 p. s. i.

In producing surface-alloyed soft, plastically readily deformable ferrous powders of the invention, it is important that at all stages of the processing operations, the powder particles should not be subjected to any material work hardening forces. Thus, for instance, it is essential that in initially sintering the soft iron powder particles into porous sinter cake fragments suitable for packing into the silicon-alloying pack, the powder which is to be subjected to the initial sintering action should not be compacted under any substantial pressure. If a substantial compacting pressure, even as low as 5 t. s. i. is initially applied to such soft ferrous metal powder (Fe, Ni, Co) in preparation for the initial sintering process, which precedes the silicon-alloying treatment, such silicon-alloyed sinter fragments will acquire a relatively great density and strength, and large forces will be required for crushing them, and the resulting powder particles will be severely work hardened. Unless the sinter cake fragments which are to be subjected to the silicon-alloying treatment are of low density and strength—obtainable if the powder particles subjected to the preliminary sintering action have not been initially compacted under pressure—the crushing energy required for pulverizing the silicon-alloyed sinter cake fragments would be so large that the resulting silicon-alloyed powder particles would be distorted in shape and work hardened, making it necessary to apply undesirably large pressure for compacting them into bodies of the required final shape.

In other words, when producing silicon-surface-alloyed soft ferrous powders of the invention, it is essential that the sintered powder cake fragments or lumps—produced in preparation for the silicon-alloying treatment—should have a low density, and correspondingly low strength, so as to minimize any work hardening imparted to the powder particles when pulverizing the sintered powder cake fragments or lumps following the silicon-alloying treatment, thus resulting in powder particles exhibiting only minimized work hardening. Furthermore, it is also essential that the sintered powder cake fragments or lumps—produced in preparation for the silicon-alloying treatment—should have high porosity so that they are permeable to the gases by means of which the silicon-alloying treatment is carried on.

By controlling the density and strength of the sinter cake lumps—produced in preparation for the silicon-alloying treatment—it is possible to control the preparation of soft, plastically deformable silicon-surface-alloyed powders of the invention.

As explained above, the proper range of the density of the sinter cake lumps—produced in preparation for the silicon-alloying treatment—is about 1.5 to 4.0 g./cc. The modulus of rupture of such sinter cake lumps is correlated to their density, being about 50 p. s. i. for sinter cake lumps having a density of 1.7 g./cc. and increasing to about 6500 p. s. i. for sinter cake lumps having a density of 4.0 g./cc.

Furthermore, as long as the sinter cake lumps, produced in preparation for the silicon-alloying treatment, are of low density, and have a low modulus of rupture in the ranges set forth above, they will also have about the same density and the same modulus of rupture after being subjected to the silicon-alloying treatment whereby silicon is surface-alloyed with the individual powder particles of such sinter cakes.

Accordingly, to obtain silicon-alloyed powders of the invention, it is sufficient to control the density and/or modulus of rupture of the sinter cakes or sinter cake lumps produced in preparation for the silicon-alloying treatment. As long as the density of such sinter cake lumps is not more than about 4.0 g./cc., and their modulus of rupture is not more than about 6500 p. s. i. they will, after the silicon-alloying treatment, remain of sufficiently small density and strength as to permit their pulverization without material work hardening of the powder particles. As a result, the silicon-alloyed powder obtained from such sinter cakes will have the desired high degree of softness and plastic deformability as to make it possible to compact such powders into green compacts having a modulus of rupture of at least 300 p. s. i. and higher.

By sintering such green compacted bodies of silicon-alloyed soft iron powder of the invention, compacted with only a relatively small pressure, there are obtained bodies having the same strength as those produced by sintering prior art metal powders of corresponding composition which have been compacted with much higher pressure. Good results are obtained by sintering green compacted bodies of such silicon-alloyed powder at a temperature in the range of 1200° to 1350° C. within a protective atmosphere such as dry hydrogen, or of purified cracked ammonia. It is also desirable to maintain the protective atmosphere at a dew point of about —50° C. or below. Such low dew point atmosphere may be obtained by a suitable getter such as pure chromium powder or ferrosilicon powder (70% Cr) mixed with aluminum oxide.

Green compacted bodies made from soft silicon-surface-alloyed iron powder of the invention may be infiltrated with other metals such as copper and copper alloys for producing composite bodies having the desired combined characteristics. Such infiltrated bodies may be subjected to heat treatments, either at the time of

the infiltration, or subsequent thereto for causing the infiltrant and the metal particles and substances of the powder compact skeleton to diffuse into each other to any degree as desired.

According to a further phase of the invention, silicon-surface-alloyed soft plastically readily deformable powders of either iron, nickel or cobalt, or alloys or mixtures thereof, are produced in a very economical way from the oxides, or mixtures of the oxides of these metals, or mixtures of these metals and their oxides. Among the oxides suitable for this purpose is black mill-scale containing principally iron oxides (Fe_3O_4 and FeO) which is formed when rolling or forging iron and steel.

For producing silicon-surface-alloyed powders of the invention from oxides of iron, nickel or cobalt, or mixtures of the different oxides, the desired metal oxide or oxides may be subjected to a combined reducing and sintering treatment.

By way of example, there will now be described a satisfactory process for producing a silicon-alloyed powder containing soft moldable iron and nickel alloyed powder particles partially silicon-alloyed by surface-diffusion of silicon in accordance with the process of the invention.

Powder particles of iron oxide mill scale containing essentially Fe_2O_3 and FeO are mixed with nickel oxide powder and lamp black, and ball milled into a powder of —100 mesh. As an example, 200 parts of the mill scale are mixed with 22.6 parts nickel oxide and one part of lamp black. The powder mixture is then ball milled to powder of —100 mesh, and placed in treatment boats of heat-resistant metal coated on the interior with a stick-suppressing medium, and treated in a furnace under reducing atmosphere such as dry hydrogen or cracked ammonia at a temperature in the range between 800° and 1100° C. for one-half to three hours, and then permitted to cool in the same atmosphere. Satisfactory results are obtained by such treatment carried on at a temperature of about 950° C. for one hour. The powder mix may be pelleted into pellets before subjecting it to the foregoing treatment, or it may be placed into the treatment boat as a thin powder layer about ⅛ to ¼" deep, in which case the resulting sinter cake produced by the reducing heat treatment is broken up into lumps. The powder is formed into pellets by mixing it with a lubricant and binder so that the resultant mixed powder mass may be readily made up into small porous pellets, for instance, of cylindrical shape, having a diameter of about ¼ to ½ inch, and the same height, with a pellet density of 3 to 4 g./cc. Any suitable organic lubricant and binder which decomposes at elevated temperatures of about 800° to 900° C. and above, may be used in making such pellets. For instance, high fatty acids, such as stearic acid, and salts of stearic acid, such as zinc stearate or the like, are suitable for use as a lubricant. Carbohydrates, such as dextrose dissolved in water, or camphor dissolved in alcohol, may be used as a binder in making such pellets.

The reducing and sintering treatment carried out at an elevated temperature in the manner described above reduces the oxides and causes the different metal constituents of the different powder particles, i. e. of nickel and iron powder to mutually diffuse with each other, and the individual powder particles become actually alloyed.

In the particular example referred to herein, the reducing and sintering treatment was performed at a temperature of about 1050° C. for one hour, resulting in sinter cake pellets having a density of about 2.3 g./cc., with a weight loss of about .5%.

The reduced sintered powder pellets or sinter cake pieces produced in the manner described above are then subjected to a silicon-surface-alloying treatment similar to that applied to the sinter cake pieces made from sponge iron powder as described above. The silicon-surface-alloyed sinter cake pellets obtained by such treatment have about the same density as they had before being subjected to the siliconizing treatment, and they may be comminuted with minimized work hardness into soft silicon-surface-alloyed powder particles having the desired high degree of plastic deformability. By way of example, silicon-surface-alloyed sinter cake pellets produced in the manner described above, yield upon comminution, a silicon-surface-alloyed iron powder of high softness, and plastic deformability, so that a test bar of such powder compacted under a pressure of 25 t. s. i. without any lubricant or binder, exhibits a modulus of rupture of about 900 p. s. i.

By prolonging the silicon-alloying treatment, or by subjecting the silicon-alloyed sinter cake pieces, such as the sinter cake fragments or sinter cake pellets, to a suitably long additional heat or sintering treatment, the silicon content of the outer layers of the powder particles of such bodies may be caused to diffuse into the interior of the individual powder particles, and thus cause such powder particles to be substantially uniformly alloyed with silicon. Furthermore, such prolonged diffusion treatment will cause the different metals of the individual powder particles, as well as their silicon content, to mutually diffuse, thereby giving the resulting body the characteristic desired alloy composition.

By proceeding in a similar manner with a mixture of oxides of one or more of the metals iron, nickel and cobalt, with one or more of the metals molybdenum, tungsten, chromium, vanadium, manganese, or the oxides of these metals, there may be produced silicon-surface-alloyed soft, plastically, readily deformable powder mixtures of the desired different metals, to wit, iron, nickel, cobalt, molybdenum, chromium, vanadium, and manganese, exhibiting the combined characteristics of the different combined constituents.

It should be noted that when producing silicon-surface-alloyed soft metal powders of the invention from oxides of the desired metals, such as oxides of iron, nickel, cobalt, molybdenum and tungsten, and mixtures thereof, the oxide powders may be pelleted into pellets for the preliminary reducing and sintering treatment. When such pellets are subjected to the combined reducing and sintering treatment of the type described above, the admixed lubricant and binder is decomposed and driven off. As a result, the reduced sinter cake pellets have the required low density and small strength, comparable to the strength of the sinter cake fragments produced by sintering sponge iron powder deposited in a layer of about ¼" within the treatment boats in the preliminary treatment of sponge iron powder described hereinbefore. As long as the sinter cake pellets resulting from the preliminary reducing and sintering treatment have a density

not exceeding about 5 g./cc., and a modulus of rupture not exceeding about 6500 p. s. i. the sinter cake pellets will yield silicon-alloyed powders having the desired high degree of softness and plastic deformability as to make it possible to compact such powders into green compact bodies having a modulus of rupture of at least 300 p. s. i. and higher with a pressure of only 50,000 p. s. i.

As used in the specification and claims, the expression "sinter cake body" includes both sinter cake lumps and sinter cake pellets of sufficiently low density and strength that upon comminution of such silicon-alloyed sinter cake body into minute silicon-alloyed powder particles having the desired high degree of softness and plastic deformability which makes it possible to compact such powders into green compacted bodies having a modulus of rupture of at least 300 p. s. i. and higher with a pressure of only 50,000 p. s. i.

The principles of the invention described above in connection with specific exemplifications thereof, will suggest various other modifications and applications of the same. It is accordingly desired that the present invention shall not be limited to the specific exemplifications described herein.

We claim:

1. A sintered cake body of metal powder particles which is readily comminutable into soft metal powder particles, the particles of said cake body being composed essentially of at least one metal of the group consisting of iron containing at most about .2% carbon, of nickel, and of cobalt, and mixtures and alloys of said metals, said cake body having been subjected to a silicon-alloying treatment causing at least the surface layer of said particles to become alloyed with silicon deposited on the particles from a silicon compound at elevated temperatures so that said body contains at least 3% of silicon, said cake body having at most a density of about 5 grams per cubic centimeter, whereby said body may be pulverized into minute silicon-alloyed powder particles which exhibit minimized work hardness and sufficiently great softness so that when the silicon-alloyed particles are compacted into a green body under pressure of about 50,000 pounds per square inch, said green body exhibits a modulus of rupture of at least 200 pounds per square inch.

2. A sintered cake body of metal powder particles which is readily comminutable into soft metal powder particles, the particles of said cake body being composed essentially of at least one metal of the group consisting of iron containing at most about .2% carbon, of nickel, and of cobalt, and mixtures and alloys of said metals, said cake body having been subjected to a silicon-alloying treatment causing at least the surface layer of said particles to become alloyed with silicon deposited on the particles from a silicon compound at elevated temperatures, said cake body containing at least 3% silicon and having at most a density of about 5 grams per cubic centimeter.

3. A sintered cake body of metal powder particles which is readily comminutable into soft metal powder particles, the particles of said cake body being composed essentially of at least one metal of the group consisting of iron containing at most about .2% carbon, of nickel, and of cobalt, together with at least one metal consisting of molybdenum and tungsten, and of mixtures and alloys of said metals; said cake body having been subjected to a silicon-alloying treatment causing at least the surface layer of said particles to become alloyed with silicon deposited on the particles from a gaseous silicon compound at elevated temperatures, said cake body having at most a density of about 5 grams per cubic centimeter and a modulus of rupture of at most 6500 pounds per square inch, whereby said body may be pulverized into minute silicon-alloyed powder particles which exhibit minimized work hardness and sufficiently great softness so that when the silicon-alloyed particles are compacted into a green body under pressure of about 50,000 pounds per square inch, said green body exhibits a modulus of rupture of at least 200 pounds per square inch.

4. A sintered cake body of metal powder particles which is readily comminutable into soft metal powder particles, the particles of said cake body being composed essentially of at least one metal of the group consisting of iron containing at most about .2% carbon, of nickel, and of cobalt, together with at least one metal of the group consisting of molybdenum and tungsten, and of mixtures and alloys of said metals; said cake body having been subjected to a silicon-alloying treatment causing at least the surface layer of said particles to become alloyed with silicon deposited on the particles from a silicon compound at elevated temperatures, said cake body containing at least 3% silicon and having at most a density of about 5 grams per cubic centimeter.

5. The method of providing a readily compactible and moldable metal powder comprising first producing readily frangible porous sinter cake pieces composed essentially of sintered metal powder particles of at least one metal of the group consisting of iron containing at most about .2% carbon, of nickel, and of cobalt, and mixtures and alloys of at least two of said metals so that said cake pieces have at most a density of about 4 grams per cubic centimeter, and thereafter subjecting said cake pieces to a silicon-alloying treatment in which silicon from a silicon compound is deposited on the powder particles of said cake pieces and caused to diffuse into said powder particles so that said powder particles contain at least about 3% silicon, and thereafter comminuting said sake pieces into minute plastically readily deformable metal powder particles having the deposited silicon alloyed therewith so that when said silicon-alloyed particles are compacted in the absence of a binder into a green body under a pressure of about 50,000 pounds per square inch said green body exhibits a modulus of rupture of at least about 200 pounds per square inch.

6. The method of providing a readily compactible and moldable metal powder comprising first producing readily frangible porous sinter cake pieces of sintered metal powder particles composed essentially of at least one metal of the group consisting of iron, containing at most about .2% carbon, of nickel, and of cobalt, together with up to at least one metal of the group consisting of molybdenum and tungsten, and of mixtures and alloys of said metals; so that said sinter cake pieces have at most a density of about 4 grams per cubic centimeter, and thereafter subjecting said cake pieces to a silicon-alloying treatment in which silicon from a silicon compound is deposited on the powder particles of said cake pieces and caused to diffuse into said powder particles so that the said powder particles contain at least about 3% silicon, and thereafter

comminuting said cake pieces into minute plastically readily deformable metal powder particles having the deposited silicon alloyed therewith so that when said silicon-alloyed particles are compacted in the absence of a binder into a green body under a pressure of about 50,000 pounds per square inch, said green body exhibits a modulus of rupture of at least about 200 pounds per square inch.

7. The method of providing a readily compactible and moldable metal powder comprising first producing readily frangible porous sinter cake pieces composed essentially of sintered metal powder particles of the group consisting of iron having at most about .2% carbon, of nickel, and of cobalt, and mixtures and alloys of at least two of said metals so that said cake pieces have at most a density of about 4 grams per cubic centimeter, and thereafter subjecting said cake pieces to a silicon-alloying treatment in which silicon from a gaseous silicon compound is deposited on the powder particles of said cake pieces and caused to diffuse into said powder particles so that said powder particles contain at least about 3% silicon, and thereafter comminuting said cake pieces into minute plastically readily deformable metal powder particles having the deposited silicon alloyed therewith so that when said silicon-alloyed particles are compacted in the absence of a binder into a green body under a pressure of about 50,000 pounds per square inch, said green body exhibits a modulus of rupture of at least about 200 pounds per square inch.

8. The method of providing a readily compactible and moldable metal powder comprising first producing readily frangible porous sinter cake pieces composed essentially of sintered metal powder particles of at least one metal of the group consisting of iron containing at most about .2% carbon, of nickel, and of cobalt, together with up to about 30% of at least one metal of the group consisting of molybdenum and tungsten, and of mixtures and alloys of said metals so that said sinter cake pieces have at most a density of about 4 grams per cubic centimeter, and thereafter subjecting said cake pieces to a silicon-alloying treatment in which silicon from a gaseous silicon compound is deposited on the powder particles of said cake pieces and caused to diffuse into said powder particles so that the said powder particles contain at least about 3% of silicon, and thereafter comminuting said cake pieces into minute plastically deformable metal powder particles having the deposited silicon alloyed therewith so that when said silicon-alloyed particles are compacted in the absence of a binder into a green body under a pressure of about 50,000 pounds per square inch said green body exhibits a modulus of rupture of at least about 200 pounds per square inch.

9. The method of providing a readily compactible and moldable metal powder comprising first preparing a powder mass of powder particles composed essentially of at least one metal of the group consisting of iron containing at least one metal of the group consisting of iron containing at most about .2% carbon, of nickel, and of cobalt, and mixtures and alloys of at least two of said metals, at least some of said powder particles containing the metal in the form of an oxide, heating the powder mass in a reducing atmosphere at a temperature of at least about 700° C. to produce porous sinter cakes having at most a density of about five grams per cubic centimeter, and thereafter subjecting said cake pieces to a silicon-alloying treatment in which silicon from a gaseous silicon compound is deposited on the powder particles of said cake pieces and caused to diffuse into said powder particles so that the said powder particles contain at least about 3% of silicon, and thereafter comminuting said cake pieces into minute plastically readily deformable powder particles of said metals having the deposited silicon alloyed therewith so that when said silicon-alloyed metal particles are compacted in the absence of a binder into a green body having a pressure of about 50,000 pounds per square inch said green body exhibits a modulus of rupture of at least about 200 pounds per square inch.

10. The method of providing a readily compactible and moldable metal powder comprising first preparing a powder mass of powder particles composed essentially of at least one metal of the group consisting of iron containing at most about .2% carbon, of nickel, and of cobalt, together with up to about 30% of at least one metal of the group consisting of molybdenum and of tungsten, and of mixtures and of alloys of said metals; at least some of said powder particles containing the metal in the form of an oxide, heating the powder mass in a reducing atmosphere at a temperature of at least about 700° C. to produce porous sinter cakes having at most a density of about five grams per cubic centimeter, and thereafter subjecting said cake pieces to a silicon-alloying treatment in which silicon from a gaseous silicon compound is deposited on the powder particles of said cake pieces and caused to diffuse into said powder particles so that the said powder particles contain at least about 3% of chromium, and thereafter comminuting said cake pieces into minute plastically readily deformable powder particles of said metals having the deposited silicon alloyed therewith so that when said silicon-alloyed metal particles are compacted in the absence of a binder into a green body under a pressure of about 50,000 pounds per square inch said green body exhibits a modulus of rupture of at least about 200 pounds per square inch.

GEORGE STERN.
S. J. SINDEBAND.
J. P. SCANLAN.

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