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PROCESS OF MANUFACTURING SHAPED
BODIES FROM IRON POWDERSRenzo U. Volterra, Yonkers, N. Y., assignor to
American Electro Metal Corporation, Yonkers,
N. Y., a corporation of MarylandNo Drawing. Application November 8, 1940,
Serial No. 364,797

10 Claims. (Cl. 75-22)

This invention relates to the production of shaped sintered bodies from iron powder to the effect that a predetermined average content of carbon and, if desired, other additions is present in the final body. In particular, the invention concerns the production by sintering of dense bodies having the character of steel or alloyed steel, from a mixture of different kinds of iron powder.

It was difficult heretofore, or impossible, to produce by sintering shaped iron articles of desirable density and carbon content as well as mechanical specifications from powdery initial material.

Powder of steel of any description cannot be compacted by commercially practicable pressures into bodies sufficiently coherent for further manipulation. Even if pressures of the order of 100 tons per sq. in. or more are applied, brittle or fragile compacts, if any at all, result.

It was suggested therefore to produce iron powder of desired carbon content by carburizing ferritic iron powder to desired extent, quenching the hot carburized iron powder, crushing and thereafter annealing it in order to render the particles soft and briquettable. This process is relatively expensive and is not satisfactory.

It was also suggested to decarburize steel or other combined carbon containing iron powder superficially, so as to obtain particles consisting of an iron core having retained its carbon content while superficial layers consist of ferritic iron, which is capable of welding together with similarly superficial layers of other particles under high pressure. Upon sintering, however, quite porous bodies are obtained. The process is also expensive and requires great care in its performance.

It is therefore an object of the invention to increase the efficiency and lower the cost of production of shaped sintered bodies containing a desired amount of carbon and other additions.

It is another object of the invention to use iron powders containing combined carbon, in particular steel or cast iron, as available on the market, without substantially affecting and particularly changing their carbon content before compacting them under pressure.

It is a further object of the invention to produce shaped sintered bodies of predetermined average content of carbon and other admixtures from two or more kinds of iron powders of different carbon content and at least one of which, due to its carbon content and structure,

is normally, i. e. under pressures up to about 50 tons psi., not briquettable.

It is a particular object of the invention to produce shaped sintered bodies from different kinds of iron powders, one of which is normally not briquettable and contains combined carbon, and if desired, other admixtures in predetermined or given amount, while another kind of iron powder is substantially oxidic and upon reduction after compacting serves to adjust the average carbon content and the bond and structure of the sintered body obtained.

It is still a further object of the invention to produce sintered shaped bodies from two or more kinds of iron powder and a binder, one of the powders due to its carbon content and structure, being brittle and normally not briquettable, while another one consists in an iron oxide which, upon reduction after compacting serves to adjust the average carbon content and the bond and structure of the sintered body obtained, and the binder is either volatile or remains at least in part in the sintered body and consists in the latter case of ferrite powder or an organic substance.

It is still a further object of the invention to produce by sintering shaped bodies from three or more kinds of iron powders, one of which is substantially ferritic or malleable and substantially serves as a binder, another one is of the character of steel, and if desired, alloyed with other admixtures, while a third one acts to adjust the average carbon content of the body and adds to the ferritic binder and/or the other kind of iron present in the final body.

It is a particular object of the invention to produce under commercially practicable pressures and by sintering shaped iron bodies containing a desired average amount of carbon and, if desired, admixtures as used in alloy steels, from an intimate mixture of three or more kinds of iron powders, one of which is substantially ferritic in character or malleable, another one consists of iron powder containing combined carbon and, if desired, other admixtures as used in alloy steels, while the third one consists of iron oxide which together with some carbon added, during sintering operates to adjust the average carbon content of the final body and adds to the ferritic binder and/or the other kinds of iron.

These and other objects of the invention will be more clearly understood when the specification proceeds.

Substantially pure or ferritic iron can be produced commercially in different ways and is avail-

able on the market. Sponge iron, or electrolytic iron, can be used as such pure iron and crushed to desired particle size. However, these kinds of iron are relatively expensive and sometimes difficult to obtain. It is therefore preferred to manufacture such iron powder by des-
 oxidizing iron oxide in well known manner, or as particularly described in the copending application of Claus Guenter Goetzel, Ser. No. 364,814 and Paul Schwarzkopf, Ser. No. 349,996.

Crushed steel or alloy steel is available on the market. It can be produced, however, from steel or alloy steel as available on the market and containing about .1% to 1.5% carbon, by crushing and sieving it and, if particularly fine particle sizes are desired, by repeated crushing and sieving. A steel or alloy steel may be heated and quenched before crushing, if its original brittleness does not suffice for the intended powdering process.

If particular kinds of iron powder are desired containing more carbon than present in steel grades on the market, cast iron, in particular gray iron containing about 2.4% to 4% carbon or white iron, containing above about 2% carbon, can be crushed and sieved, or repeatedly crushed and sieved, so as to obtain therefrom a powder of the desired particle size.

If admixtures, such as tungsten, molybdenum, tantalum, vanadium, titanium, silicon, nickel, cobalt, chromium, and/or manganese are desired in the final body in an average amount which cannot be introduced into it by admixture of steel or iron alloys as available on the market, iron alloys of a desired content of these admixtures are to be produced, and to be powdered in any of the ways described above for steel powders. Such alloys are also available in the market as ferronickel, ferrochrome, ferromanganese, stainless steel scrap, etc. These admixtures can also be added in powdery metallic state.

Iron oxide, particularly in powdery form is available on the market, particularly as mill scale.

According to one feature of the invention, at least three different kinds of iron powders are admixed, one of the type of ferritic iron powder, another of the type of iron oxide, and a third of the type of iron containing admixtures, one of which is always carbon and the other may be, if desired, molybdenum, tungsten, chromium, nickel, cobalt, vanadium, tantalum, silicon, titanium, manganese, etc. The minimum amount of the ferritic type of iron powder depends on the processing of the mixture. The amounts of the other two types of iron powders depend upon the desired ultimate content of carbon and other admixtures in the final sintered body.

The powders are intimately mixed, preferably in a ball mill provided with a steel lining and steel balls of a composition resembling, if possible, that of the ultimate product to be obtained by sintering the powders. Dry milling is generally preferable, though wet milling can be applied with advantage even though it affords drying of the mixed powders in a preferably neutral or inert atmosphere, such as desiccated hydrogen. Ball milling may be performed at room or elevated temperatures, up to about 500 to 600° C.

The mixture is then pressed to shape at 10 to 50 tons per sq. inch, but preferably not exceeding 30 tons, which is commercially practicable.

Due to the presence of the ferritic, soft binder a compact is obtained which can be taken out of the mold or die in which it has been pressed, be subjected to presintering, if desired, and final

sintering. If the mixture was prepared by hot ball milling so that the particles of harder iron powder, in particular steel have been covered by a thin film of ferrite, small amounts of the binder, down to about 5%, are sufficient and permit compacting under pressures from 20 to 30 tons P. S. I. With 15% to 20% of the binder and such hot ball milling of the mixture, pressures of 5 to 15 tons P. S. I. suffice to arrive at a sufficiently coherent body. If ball milling at room temperature was applied, a minimum amount of ferritic binder from 15% to 20% is preferable for compacting under commercially practicable pressures. If the steel powders and particularly the combined iron containing powders were relatively coarse, say of a size corresponding up to about 100 mesh, a minimum amount of ferritic binder from 25% to 30% is preferable in order to obtain a sufficiently coherent compact under commercially practical pressures.

As explained more in detail in the above mentioned copending application of Claus Guenter Goetzel, Ser. No. 364,814, filed November 8, 1940, the iron powders containing combined carbon are angular and exhibit sharp edges which, upon being pressed together with soft ferrite, cut into the latter, and interlock the steel particles with the soft ferrite.

The ferrite particles are malleable and under pressure therefore adapt their shapes to those of the other harder iron powders present, so that the pressed compact, although still porous, can be subjected to handling and sintering without losing its shape.

The pressed compact may now be submitted to presintering or immediately to final sintering. Presintering serves mainly the purpose of degasifying the compact, particularly some or entire desoxidation of the iron oxide powder, and to facilitate final sintering. The presintering temperature lies about 30% below the melting point of the entire mixture and can be first established by rough calculation and thereafter verified by a few experiments, and lies between about 750° to 1050° C.

Presintering should be done preferably in an atmosphere neutral or inert to iron, such as desiccated hydrogen, which is capable, however, to desoxidize the iron oxide powder at suitable temperatures, if this be desired.

For purposes to be described later on in more detail, the use of an atmosphere containing carbon developing gases at presintering temperatures, may be advisable; as such atmosphere natural gas (methane) or mixtures of such gases and hydrogen may be mentioned by way of example.

The compacted or presintered body is then to be high sintered.

The sintering temperature depends of course upon the constitution of the mixture and particularly its carbon content. It is well known in the art that the melting point of iron decreases with increasing carbon content, from about 1150° C. for pure iron, to about 1150° C. at 4.2% carbon content, and then increases again slowly with further increasing carbon content. However, iron with such high carbon content is of no practical value to date. It is further well known in the art that high sintering of metals ordinarily occurs at temperatures about 10% lower than melting temperature and thus the sintering temperature for any mixture of iron powders can be easily established by calculation as well as a few experiments, for which the calculated average content of carbon of the mix gives a good basis. Ad-

mixtures of other type than carbon as exemplified above, may slightly affect the sintering temperature of the mix but can first be roughly calculated from the well known melting temperatures of the components of the mix, taking into consideration their ratios, and thereafter verified by a few experiments.

The ratios of the three types of iron powders to be used depend entirely upon the composition of the final body to be obtained.

Taking an initial mixture of 20% ferrite powder, 70% powder of steel containing 1.5% carbon and 10% iron oxide powder, it is to be considered that iron oxide of the formula FeO contains about 30% oxygen and 70% iron. Upon heating the mixture to sintering temperature, the iron oxide decomposes, 30% oxygen go off and 70% iron remain. Consequently, from the 10% iron oxide present in the initial mass, theoretically only 7% reduced iron (ferrite) will remain in the mass. The initial mixture of 100% constituents contains therefore after sintering only a total of 97%.

The 3% oxygen present in the initial mass combine in part with the hydrogen, if used as atmosphere neutral to iron, and in part with the carbon introduced by the steel powder into the mass as combined carbon.

The amount of carbon introduced into the mass by the steel powder in this example, amounted to little more than 1.05% in proportion to the entire mass of iron (20% ferrite, 7% recovered from the iron oxide and 68.95% from the steel) and may be reduced by the iron oxide to about 0.4% to 0.5%, calculated upon the entire iron content of the final body.

Thus, upon sintering in desiccated hydrogen a final body will be obtained containing about 0.4 to 0.5% carbon. It may be assumed that sintering is performed in a push furnace of continuous operation type in which the pressed compacts are positioned on a belt of bendable molybdenum sheet, and moved on and with the belt through a horizontal tube of nicrom, tungsten or molybdenum which is surrounded over a part of its length by a refractory in which wires or foils of nicrom, molybdenum, tungsten, etc., are embedded and heated to desired temperature by an electrical current in an adjustable way. By controlling the amount of hydrogen passed through the tube in the time unit, by further controlling the speed of the molybdenum sheet and thereby the period of sintering of the compacts thereon, and by controlling the sintering temperature, the ultimate amount of carbon remaining in the compact upon desoxidation of the iron oxide can be readily controlled. The larger the volume per time unit of hydrogen passed under surppressure through the tube, the larger will be the amount of oxygen taken from the iron oxide and combined with the hydrogen and the smaller the amount of carbon taken by that oxygen from the combined carbon present in each individual compact.

Taking the same initial mixture as mentioned above, and assuming that a final body containing 1% combined carbon is desired, this can be achieved only if practically none of the combined carbon introduced into the mixture by the admixed steel, is burned off (oxidized) during sintering. To this effect the atmosphere in which sintering is performed, should contain carbon developing gases, such as hydro-carbons which decompose at sintering temperature and give up carbon which can combine with the

entire oxygen developed by the iron oxide at sintering temperature. Natural gas (methane) may be used to this effect, and if the carbon given up by the gas stream passing the tube in a controlled amount per time unit should be too rich in carbon. It may be diluted with a gas such as hydrogen. Here again, by controlling the period and temperature of sintering, and the amount of gas per time unit passing the tube, the desired effect can be obtained.

Instead of using a carbon developing atmosphere, the calculated amount of carbon needed for desoxidizing the iron oxide contained in the initial mixture can be intimately admixed to the initial mixture preferably in the form of lamp black before pressing. Sintering is then to be performed in an inert atmosphere, such as desiccated hydrogen.

Taking as another example a final body the carbon content of which should be 0.5% in proportion to the entire mass of the iron present, in order to fabricate gears or tools of desired more or less intricate shape ready for use upon hardening, 20% ferrite powder, 10% iron oxide (preferably FeO), 30% cast iron (free of silicon and phosphorus containing 3.7% combined carbon and 40% steel containing 1.2% carbon may be admixed, pressed and sintered. Final sintering is to be performed in a hydrogen atmosphere and controlled in the way described above, so that about $\frac{2}{3}$ of the oxygen developed by the iron oxide combine with hydrogen while $\frac{1}{3}$ of the oxygen serves to reduce the total carbon content of 1.5% (of the original mass) introduced by the cast iron and steel, to the desired 0.5% carbon content of the resulting total iron mass. The same effect can be obtained by sintering in a hydrocarbon containing atmosphere, whereby, however, also a desired carburization of the iron mass can be effected in controllable manner.

Before going more into detail into the operation of the sintering process, the outstanding advantages of this feature of the invention may be explained. Ferrite powder is used for the purpose of compacting the initial powder into a coherent body which can easily be taken out of the mold in which it was pressed, and subjected to sintering. Ferrite of any origin is relatively expensive and its amount should be reduced therefore to the minimum just necessary for satisfactory compacting or briquetting the initial mixture. A higher amount of ferrite is however often desirable in order to arrive at the proper structure of the final body. This additional ferrite is introduced according to the invention by the use of iron oxide powder, which is the least expensive raw material for this purpose available on the market, and reduced to ferrite during the presintering and/or sintering process in which the use of either hydrogen or carbon containing gases is necessary in any event for protecting the slug undergoing sintering against adverse effects of the surrounding air. It is difficult, if possible at all, in commercial production to entirely recover that hydrogen, natural gas, etc., it is usually burned off at the discharge end of the furnace and lost. According to the invention, this protective gas is used for the total or partial desoxidation of the iron oxide admixed to the initial mixture. In other words, a part of the expensive ferrite which is needed in the final sintered product but not for compacting the mixture previous to sintering, is produced according to the invention from non-expensive iron oxide admixed

to the initial mixture, during sintering of the shaped mixture, and for this purpose a gas which in any event has to be used in that sintering process and which heretofore was lost at least in part, is utilized for converting the admixed iron oxide into the desired ferrite.

As it appears from the copending application mentioned above, of C. G. Goetzel, Ser. No. 364,-814, some ferrite is developed in the iron mass of the final sintered body from the steel or other iron containing combined carbon which has been added to the initial mixture. However, such added steel or cast iron is to be produced from iron oxide in a blast furnace process which is mostly to be followed by refining processes. By introducing the iron oxide immediately into the initial powdery mixture, expensive conversion processes of the iron oxide are dispensed with. Moreover, iron oxide scrap can be used for this purpose which is available in the market in vast amounts and at lowest prices. Thus, according to the invention the cost of the initial mixture subjected to pressing and sintering is considerably reduced. Though the amount of iron oxide which can be admixed is relatively small, 10% in the examples given above and generally from about 5% to 40% (resulting in the first case in a theoretical minimum amount of ferrite in the sintered body of about 20% to 25% and a maximum amount of about 60%), it should be considered that the invention is primarily for mass production in which even relatively slight savings in the production of individual objects count.

It is to be understood that the possible combination of different kinds of iron powder are in no way exhausted by the above few exemplifications. Thus, instead of steel powder, or a part of it, powder or powders of alloy steel may be added, if the final sintered body is to contain certain admixtures as usually contained in steel alloys. The dilution of those admixtures contained in the alloy steel powder by the mass of other iron in the final body should be duly taken into consideration. Taking as an example a final body which is to contain 0.2% manganese, and that 30% of an alloy steel powder is introduced into the initial mass containing manganese, the amount of manganese in that alloy steel should be little less than 0.7%. Such steel alloys can be manufactured in well known manner, preferably using a prealloy of ferro-manganese which commercially contains from 80% to 82% manganese.

Instead of alloy steel, to the same effect, e. g. white iron can be admixed which is commercially obtainable with any desired manganese content up to about 20%.

Although no theory of the operation of the invention is to be submitted here, it should be clearly understood that the ferrite powder added to the initial mixture according to the invention serves primarily for compacting it into a coherent body which can be handled and sintered after pressing and shaping, and that the amount of the ferrite should therefore be limited accordingly. During presintering and in any event during sintering, the added iron oxide is first reduced to ferrite. However, while in ordinary high sintering processes the binder is added both for the purpose of agglomerating the other harder particles of the mixture during pressing and thereafter during sintering, the ferrite initially added and subsequently recovered during presintering and/or high sintering does not act merely to agglomerate the other harder particles

during sintering. It must be considered that ferrite though softer than the other admixed particles is usually of the highest melting point of the mixture, while in ordinary agglomerating or cementing processes the binder is both softer and of a lower melting point than the other particles of the mixture. Therefore, during sintering the ferrite binder and the other hard powders, such as of steel or cast iron, exchange their roles, and first the steel or cast iron become plastic or they even melt partially, while the ferrite remains solid and becomes plastic to the same extent as the steel or cast iron powders only at a higher temperature. However, during sintering a diffusion starts of the combined carbon present in the admixed steel, alloy steel or cast iron powders into the ferrite powders, both originally admixed and recovered by reduction of the iron oxide powder added, and thereby the ferrite is partially or entirely converted into iron containing combined carbon; upon gradual incorporation of carbon into the ferrite, it is gradually converted into austenite until about 1.7% carbon are absorbed, or into austenite plus cementite if a larger percentage of carbon is absorbed at high sintering temperature. If controlled sintering is performed until all the ferrite present in the mass is thus converted, eventually a sort of equilibrium will be attained and the mass will consist throughout of particles containing a substantially equal percentual amount of carbon and be of austenitic or austenitic and cementitic character. If controlled sintering is carried out only to such an extent that a desired part of the ferrite both initially admixed and recovered from the reduction of the iron oxide, has absorbed carbon from the other powders contained in the initial mixture, then the mass will eventually consist of the balance of ferrite which has not absorbed carbon, of austenite or austenite plus cementite resulting from the conversion of that ferrite, and of austenite or austenite plus cementite originating from the steel, alloy steel or cast iron powders added to the initial mixture.

In calculating the theoretical sintering temperature of the entire mass from the sintering temperature of its individual components and the ratio in which they are present in the mass, it will appear that the sintering temperature is the higher, the higher the amount of ferrite initially admixed and recovered from the iron oxide during presintering and/or sintering, and particularly during heating the mass up to high sintering temperature is.

It will also be found that at such theoretical sintering temperatures iron powders which contain combined carbon are highly plastified or even start to melt. However, the higher the final sintering temperature is, the faster the combined carbon diffuses into the ferrite and thereby reduces its melting and sintering temperature which is on an average about 10% below the melting temperature though this does by no means form as fixed a value as the melting temperature but includes a range close to but below melting temperatures. It is therefore safe for most purposes to work at a high sintering temperature close to that theoretically calculated for an iron mass containing carbon of an amount introduced by the iron powders containing combined iron, that mass of iron to include ferrite and recovered iron. Thus, depending upon that combined carbon content calculated on the entire iron mass, the final sinter-

ing temperature will be within a range of about 1150° to 1390° C.

In this respect as well as regarding the preparation of the mixture from fine and coarse powders, reference is made to the broad explanations in the copending application of C. G. Goetzel, Ser. No. 364,814.

It must be understood that in all these events the carbon content of the initially admixed steel, alloyed steel or cast iron powders is reduced by the amount of combined carbon which diffused into part or all of the ferrite present in the mass during sintering at high sintering temperatures.

It should be further understood that other admixtures than carbon which were contained in the admixed alloy steel or cast iron powders, either diffused in part from those powders into the ferrite particles initially admixed and recovered during sintering from the iron oxide, if they are capable of diffusing into the ferrite at high sintering temperature, as is the case for instance with molybdenum, cobalt, nickel, or remained substantially in those alloy steel or cast iron particles, as is the case for instance with manganese particularly if present in large percentage.

It should further be clearly understood that upon cooling the mass thus highly sintered in a controlled manner, the mass undergoes transformation when passing the temperature corresponding to the transformation point A3 of the well known iron-carbon-temperature diagram. Depending upon the carbon content of the particles of the mass thus cooled, they will result in pearlite if their carbon content at the end of high sintering was 0.9%, or into pearlite plus ferrite, if their carbon content was lower, or into pearlite plus cementite, if the carbon content was higher. If cooling down to about 300° C. was performed by particular quenching, a martensitic structure of the combined carbon containing particles will result.

The ferrite if retained from that added to the initial mixture and recovered from the reduction of the iron oxide, will firmly bond the other particles which contain combined carbon. The body will be dense, and certain properties can be developed by subsequent treatment, such as heat treatment of the nature of annealing, and/or mechanical treatment, such as forging, rolling, extruding and drawing. The shaped body can also be hardened by heating and subsequent quenching, or by case hardening.

According to another feature of the invention, a binder is used which is volatile entirely or in part. By "volatile" the invention understands the quality of a liquid or viscous binding fluid to evaporate or escape upon heating to a temperature below high sintering temperature, without leaving residues in the mass. In this sense this term is also used in the appended claims.

Under "partly volatile" the invention understands the quality of a liquid or viscous binding fluid to evaporate or escape at or below high sintering temperature, and preferably at or below presintering temperature, leaving desirable residues in the mass. In this sense this term is also used in the appended claims.

As an example of a volatile binder water may be mentioned.

As examples of a partly volatile binder glycerin, glycol, glucose, dextrine, tar and other liquid or viscous preferably organic substances or solu-

tions may be mentioned, which upon heating to a few hundred degrees C. decompose and partly evaporate and partly carbonize.

According to this feature of the invention, ferrite as a binder can be dispensed with entirely or in part.

It has been found that by intimately admixing water to a powdery mixture of iron oxide and combined carbon containing iron, such as steel, alloy steel, cast iron of any description, a paste is formed which can easily be pressed in molds to desired shape, under commercially practicable pressures, and upon removing from the mold forms a briquet of sufficient cohesion to be manipulated and heat treated.

To the same effect admixtures of partly volatile binders can be used.

Thus, into a powdery mixture of 30% iron oxide and 70% cast iron containing 3.7% carbon and corresponding to 150 mesh, water was stirred until a paste resulted. The paste was pressed to shape in a mold under hydraulic pressure amounting to about 30 tons P. S. I. A shaped briquet resulted which then was subjected to presintering in hydrogen at a temperature of about 900° C. After about 30 minutes a body resulted which was porous but coherent throughout and could be dropped on the floor without breaking or crumbling. An analysis showed that practically all the iron oxide present in the body was converted into ferrite.

Taking into consideration that 30% of iron oxide (FeO) constitutes oxygen, it appears that by the admixture of 30% iron oxide to the mixture, 21% iron and 9% oxygen were introduced into it. This amount of oxygen would suffice to burn off all the carbon introduced into the mass by the admixture of cast iron. Therefore, during presintering desoxidation of the iron oxide had to be effected mainly by the hydrogen. The oxygen of the oxide also reacted with the carbon introduced into the mixture by the cast iron, which amounted to 4.2% calculated upon the entire iron mass present in the mixture (21% from the iron oxide and 67.41% from the cast iron). The ultimate total carbon content of the presintered mass was found to be about 1.5%. The water was of course entirely evaporated at the start of the presintering process.

The presintered body was then pressed in a die under 50 tons P. S. I. and then sintered at 1300° C. for about one hour, hot pressed and sintered again under the same conditions. A protective atmosphere was used containing hydrogen and some admixed methane in order to prevent decarburization.

The finally sintered body was dense throughout and exhibited satisfactory properties resembling steel of equivalent carbon content.

This feature of the invention offers manifold advantages. Expensive ferrite is entirely dispensed with. Iron oxide which is the cheapest raw material available is used instead. Desoxidation of the iron oxide can be performed easily and as completely as desired during the presintering step which is always desirable in powder metallurgy for degasifying and preshrinking the compact, and during which a porous and well permeable body is exposed to the action of the desoxidizing fluid, such as hydrogen. As iron powder containing combined carbon, the equally cheap cast iron can be used for the balance of the mixture, and in spite of the high carbon content of the latter a body can be finally obtained which resembles steel, because the high carbon

content of cast iron is also reduced during controlled presintering.

It should be understood that instead of hydrogen, any other desoxidizing gas can be used such as cracked gases, methane, hydro-carbons, which can be diluted with hydrogen, etc., so as to reduce in a controlled manner all or part of the iron oxide present during presintering. If desired, part or complete desoxidation during presintering can be effected by admixing solid carbon, such as lamp black, in sufficient amount to the initial mixture and to burn it off during presintering by all or part of the oxygen combined in the iron oxide. In the first case an inert and in the second case a reducing or even carburizing atmosphere is used.

While desoxidation of a porous presintered body is preferable, the invention is not limited to this. The compacted body taken from the mold can also be subjected immediately to final high sintering. While the body is heated to final sintering temperature, the water evaporates or the partly volatile binder is driven off and decomposed, respectively. A large part of or the entire oxygen combined in the oxide will react during this heating-up period with the hydrogen, or the carbon contained in the atmosphere used, and before the body has shrunk during proceeding sintering so far that gases cannot penetrate the body anymore, desoxidation will be completed. During the balance of the sintering period, densification of the desoxidized body will be completed.

The fact that iron oxide (Fe_2O_3) melts at a temperature even higher than FeO and close to the melting temperature of ferrite is particularly advantageous. It will stay substantially solid as the admixed cast iron does, up to high temperatures, thus maintaining the porosity of the briquet and allowing the hydrogen to penetrate it thoroughly and the gaseous reaction products to escape. Upon completed reduction, the iron oxide is converted into ferrite of slightly lower melting temperature and the further operation of the sintering process as to diffusion of carbon and other admixtures, etc., is the same as described herein with reference to the first feature of the invention.

It appears that iron oxide admixed with water develops qualities similar to those observed in the preparation of slips of ceramic, silicate containing materials. The water forms colloids or compounds with the oxide capable of uniting the cast iron particles, and upon drying, a coherent body results which can be handled. However, the inventor does not confine himself to any theory of the operation of his invention in this respect. In contradistinction hereto, cast iron or steel powders admixed with water are not briquettable even under far higher pressures than used in the example mentioned above.

It is understood that instead of cast iron powders, or parts of them, powders of steel, alloy steel, and other iron alloys and metallic admixtures can be used, of the kind and nature and to the effects as herein-before described more in detail with reference to the first feature of the invention, and that the ultimate material, its structure and composition will not differ from that obtained by the use of ferrite powder as a binder, in addition to iron oxide and iron powders containing other alloying constituents.

In general, the iron oxide may amount from about 5% to about 70% of the mixture. If water is used as a binder, a minimum amount of about 10% to 15% iron oxide is preferable. The pres-

ures for compacting the body should amount from about 5 to 50 tons P. S. I., the lower values preferably applied when partly volatile binders are used, while for water as a binder minimum pressures from 15 to 20 tons P. S. I. are preferable, depending on the particle size. The finer the iron oxide powder, the lower the pressure can be. As to the amount water admixed, it should be such that upon stirring eventually a thick paste is obtained. Certain limits cannot be given therefor, but in general 2% to 6% by weight of the mixture will suffice, considering the low specific weight of water; the larger the iron oxide amount of the mixture is, the larger should be the amount of water admixed. As to partly volatile binders, an admixture of a few per cent suffices, about 1% by weight of the mixture as a minimum.

In the appended claims, the terms "volatile" and "partly volatile" are used in the sense defined above, while the term "normally briquettable" means briquettable in the cold under normal or commercially practicable pressures up to about 50 tons P. S. I., and the phrase "atmosphere capable of affecting the final average carbon content of the body" is to mean gaseous or vaporous fluids of decarburising, inert or carburising effect upon the compact undergoing sintering, the decarburising fluids being exemplified by normal non-desiccated hydrogen, sometimes diluted by oxygen derived from decomposition of oxides contained in the compact, while inert fluids are exemplified by desiccated hydrogen and carburising fluids by natural gases (methane), cracked gases, hydro-carbons and mixtures thereof with hydrogen.

It is to be understood that the invention is not limited to any particular exemplification hereinbefore described, but to be derived in its broadest aspects from the appended claims.

What I claim is:

1. A method of producing from powdery material by compacting under pressure and sintering shaped bodies of iron containing combined carbon, preferably of the character of steel, comprising the steps of intimately admixing normally not briquettable iron powder containing combined carbon with about 5% to 70% powdery iron oxide and a binder, compacting the mixture under normal pressure into a coherent body of desired shape, and heat treating the shaped body below its melting temperature under controlled conditions effecting reduction of the iron oxide contained in and thereafter final sintering of the body, said conditions including controlled application of temperatures between about 1150° to 1390° C. close to but below the prevailing lowest melting temperature of any iron component contained in said body during final sintering until a dense and strong body of predetermined average carbon content is obtained and predetermined diffusion of part of combined carbon contained in said iron powder into iron recovered from said iron oxide is effected.

2. A method of producing from powdery material by compacting under pressure and sintering shaped bodies of iron containing combined carbon, preferably of the character of steel, comprising the steps of intimately admixing normally not briquettable powder of iron containing combined carbon with about 5% to 70% powdery iron oxide and solid carbon in an amount sufficient to desoxidise at least part of said iron oxide, and about 1% to 6% of a binder, compacting the mixture under pressure from about 3 to 50 P. S. I. into a

coherent body of desired shape, and heating the shaped body in a protective substantially inert atmosphere so as to first desoxidise at least part of said iron oxide by said admixed solid carbon and thereafter finally sinter said body, the temperatures of heating not to exceed and at least said final sinter to be effected at temperatures between about 1150° to 1390° C. close to but below the prevailing lowest melting temperature of any iron component contained in said body until desoxidation of any iron oxide if still present is completed, a predetermined diffusion of carbon into iron recovered from said oxide is effected and a dense and strong body is obtained.

3. A method of producing from powdery material by compacting under pressure and sintering shaped bodies of iron containing .1% to 1.5% combined carbon and of the character of steel, comprising the steps of intimately admixing normally not briquettable powder of iron containing .1% to 1.7% combined carbon with about 5% to 70% powdery iron oxide and about 1% to 6% binder, compacting the mixture under pressure from about 3 to 50 tons P. S. I. into a coherent body of desired shape, presintering the shaped body at temperatures between about 750° to about 1050° C., so as to reduce at least part of said iron oxide, and thereafter heat treating the presintered body at temperatures between about 1150° C. to 1390° C. close to but below the prevailing lowest melting temperature of any iron component contained in said body in a controlled manner in an atmosphere capable of affecting the final average carbon content of the body under treatment in a predetermined way, until predetermined diffusion of carbon into iron recovered from said iron oxide is effected and a dense and strong body structurally resembling steel and of predetermined average carbon content between .1% to 1.5% is obtained.

4. A method of producing from powdery material by compacting under pressure and sintering shaped bodies of iron containing combined carbon and of the character of steel, comprising the steps of intimately mixing normally not briquettable powder of iron containing combined carbon with about 5% to 70% powdery iron oxide and solid carbon in an amount sufficient to desoxidise at least a substantial part of said oxide, and about 1% to 6% of a binder, compacting said mixture under pressure of about 3 to 50 tons P. S. I. into a coherent body of desired shape, presintering the shaped body at temperatures between about 750° C. to about 1050° C. until at least part of said iron oxide is desoxidised, and heat treating the presintered body at temperatures between about 1150° C. to 1390° C. close to but below the prevailing lowest melting temperature of any iron compound contained in said body in an atmosphere capable of affecting the final average carbon content of the body in a predetermined way, until desoxidation of iron oxide if still present is completed, predetermined diffusion of combined carbon into iron recovered from said oxide is effected and a dense and strong body structurally resembling steel and of predetermined average carbon content is obtained.

5. A method of producing from powdery material by compacting under pressure and sintering shaped bodies of iron containing .1% to 1.5% combined carbon and of the character of steel, comprising the steps of intimately admixing normally not briquettable powder of iron containing .1% to 1.7% combined carbon with 5% to 70% powdery iron oxide and about 5% to 30% of a

powdery ferritic binder, compacting the mixture under pressure of about 5 to 50 tons P. S. I. into a coherent body of desired shape, and heat treating the shaped body below its melting temperature under conditions effecting reduction of the iron oxide contained in the body and thereafter final sintering of the body, said conditions including controlled application of temperatures between about 1150° C. to 1390° C. close to but below the prevailing lowest melting temperature of any iron component contained in said body and of an atmosphere capable of affecting the final average carbon content of the body in a predetermined way, until said iron oxide is completely reduced, a predetermined diffusion of carbon into said ferritic binder and iron recovered from said oxide is effected, and a dense and strong body structurally resembling steel and of predetermined average carbon content between .1% to 1.5% is obtained.

6. A method of producing from powdery material by compacting under pressure and sintering shaped bodies of iron containing combined carbon, preferably of the character of alloy steel, comprising the steps of intimately admixing normally not briquettable iron powder containing combined carbon and other constituents of alloy steel, with about 5% to 70% powdery iron oxide and a binder, compacting the mixture under normal pressure into a coherent body of desired shape, and heat treating the shaped body below its melting temperature under controlled conditions effecting reduction of the iron oxide contained in the body and thereafter final sintering of the body, said conditions including controlled application of temperatures between about 1150° C. to 1390° C. close to but below the prevailing lowest melting temperature of any iron component contained in said body during final sintering until a dense and strong body of predetermined average carbon content is obtained and predetermined diffusion of part of combined carbon contained in said iron powder into iron recovered from said iron oxide is effected.

7. In a method of producing from powdery material by compacting under pressure and sintering shaped bodies of iron containing combined carbon, preferably of the character of alloy steel, comprising the steps of intimately admixing normally not briquettable powder of iron containing combined carbon and other constituents of alloy steel, with 5% to 70% powdery iron oxide and about 1% to 6% of a binder, compacting the mixture under pressure from about 3 to 50 tons P. S. I. into a coherent body of desired shape, and heat treating in a controlled manner the shaped body in a carbon discharging atmosphere at temperatures not exceeding about 1000° C. to 1390° C., so as to desoxidise said iron oxide, and finally sintering the body at temperatures between about 1150° C. to 1390° C. close to but below the prevailing lowest melting temperature of any iron component contained in said body in an atmosphere capable of affecting to predetermined extent the total carbon content of the final body, until a dense and strong body of predetermined average carbon content is obtained and predetermined diffusion of carbon into iron recovered from said oxide is effected.

8. A method of producing from powdery material by compacting under pressure and sintering shaped bodies of the character of alloy steel, comprising the steps of intimately admixing normally not briquettable powder of iron containing combined carbon and other constituents of alloy steel with 5% to 70% powdery iron oxide and

about 5% to 30% of a powdery ferritic binder, compacting said mixture under pressure of about 3 to 50 tons P. S. I. into a coherent body of desired shape, and heat treating the shaped body below its melting temperature under conditions effecting reduction of the iron oxide contained in the body and thereafter final sintering of the body, said conditions including controlled application of temperatures between about 1150° C. to 1390° C. close to but below the prevailing lowest melting temperature of any iron component contained in said body and of an atmosphere capable of affecting the final average carbon content of the body in a predetermined way, until said iron oxide is completely reduced, a predetermined diffusion of carbon and other alloying constituents into said ferritic binder and iron recovered from said oxide is effected, and a dense and strong body structurally resembling alloy steel and of predetermined average carbon content is obtained.

9. A method of producing from powdery material by compacting under pressure and sintering shaped bodies of iron containing combined carbon, preferably of the character of steel, comprising the steps of intimately admixing normally not briquettable iron powder containing combined carbon with about 5% to 70% powdery iron oxide and about 1% to 6% of an at least partly volatile binder, compacting the mixture under normal pressure into a coherent body of desired shape, and heat treating the shaped body below its melting temperature under conditions for driving off volatile constituents of said binder, effecting reduction of the iron oxide contained in the body and thereafter final sintering of the body, said conditions including controlled application of

temperatures between about 1150° C. to 1390° C. close to but below the prevailing lowest melting temperature of any iron component contained in said body during final sintering, until a dense and strong body of predetermined average carbon content is obtained and predetermined diffusion of part of combined carbon contained in said iron powder into iron recovered from said iron oxide is effected.

10. A method of producing from powdery material by compacting under pressure and sintering shaped bodies of the character of alloy steel, comprising the steps of intimately admixing normally not briquettable powder of iron containing combined carbon and other constituents of alloy steel with about 5% to 70% powdery iron oxide and about 1% to 6% of an at least partly volatile binder, compacting the mixture under pressure from about 3 to 50 tons P. S. I. into a coherent body of desired shape, and heat treating the shaped body below its melting temperature under conditions for driving off volatile constituents of said binder, reducing said iron oxide contained in the shaped body and thereafter finally sintering it, said conditions including controlled application of final temperatures between about 1150° C. to 1390° C. close to but below the prevailing lowest melting temperature of any iron component contained in said body and of an atmosphere capable of affecting the average carbon content of the final body in a predetermined manner, until predetermined diffusion of carbon and other alloying constituents into iron recovered from said oxide is effected and a dense and strong body structurally resembling alloy steel is obtained.

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