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## METALLURGY OF FERROUS METALS

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4 Claims. (Cl. 75—22)

This invention relates to the fabrication of hardened or hardenable metals from metal powders and is a continuation in part of our application Ser. No. 225,844, filed August 20, 1938.

It has been proposed heretofore to fabricate metallic products from pressed and sintered powders. Such procedure, however, has not been possible in the production of steel since the commercially available iron powder consists of nearly pure iron and therefore when pressed and sintered the resulting bodies are soft.

One of the objects of the present invention is to provide a process of producing hardened steel articles in which the disadvantages in the prior methods will be eliminated and more even and uniform hardness obtained.

Another object is to obtain homogeneity in the structure of the parts.

Another object is to produce hardened steel parts made from pressed metal powders.

A still further object is to provide a method of rendering hardenable steel powders compressible.

Other objects and advantages of the invention will hereinafter appear.

In our aforesaid application Ser. No. 225,844, we have disclosed a method of producing steel from powdered metals based upon the discovery that when carbon is once combined with iron in the form of iron carbide, the carbon is mobile at elevated temperatures, and may be uniformly diffused throughout the composition. By mixing iron powder and iron powder containing iron carbide and pressing the mixture in a die at high pressure, one can attain very close mechanical contact of the two constituents. If this compact is then heated above the critical range where the microstructure is austenitic, the carbon in the combined form readily diffuses through the iron powder and a steel results which has the carbon distributed in a uniform or zero concentration gradient, i. e., the microstructure is the same throughout the whole piece. This alloy, on cooling, has a pearlite structure and can be treated like an ordinary steel. It can be reheated at normalizing temperatures and quenched in water or oil to produce a hardened structure, like tool steel, and tempered subsequently to relieve the hardening strains.

Other elements for alloying the steel can be added to the iron powder and iron carbide mixture to produce the whole range of alloy steels as they are known commercially today. These include for example: nickel, chromium, manganese, vanadium, tungsten, etc., and even silicon, phosphorus and sulphur which are ordinarily considered as impurities in steels. In the aforesaid application the carbon in the combined form of iron carbide is added to the iron powder in the form of a powdered ferro alloy, high in combined carbon, such as ferro-chrome, ferro-nickel, ferro-silicon, etc., or in the form of carburized iron powder. The present invention is particularly concerned with the latter method of introducing combined carbon into the composition.

The carburized iron powder may be produced by carburizing sponge iron in a rotary or other type gas carburizing furnace, as hereinafter more fully described. The percentage of carburized iron powder may be varied in accordance with the particular characteristics desired in the ultimate product. In the production of steels we prefer to add carburized iron powder to pure iron powder in the proper proportion to give a carbon content in the finished product of from .1 to 1.5 percent combined carbon. Such mixtures will produce steels exhibiting varying degrees of hardness, increasing with the increase in carbon content, the 1.0 percent combined carbon steel being extremely hard throughout. Those with the lower carbon content, such as 0.1 percent, may be carburized by the usual carburizing processes to produce a case hardening, consisting of a core of extremely tough and partially hardened steel with an extremely hard surface portion.

The pure iron powder and carburized iron powder may be mixed in the proper proportions in a ball or baffle mill, preferably the latter, since it results in a more general and intimate mixing without changing the shape or size of the individual metal or carburized iron particles. After intimate mixing thereof, the powders may be pressed into the desired shape by hydraulic or other forms of presses under a pressure of the magnitude of 100,000 lbs. per square inch. The pressed parts are then sintered in a non-oxidizing atmosphere at a temperature above the critical range of the metal, for a sufficient period to obtain a thorough diffusion of the carbide through the mass, whereby a homogeneous structure is obtained. The time of heating is dependent upon the size of the article, the percentage of carbide and other alloying ingredients therein, and may vary from a few minutes to an hour or more. During this period the powdered particles coalesce and a fine, uniform and homogeneous structure is produced. After sintering, the parts may be reheated, quenched and hard-

ened like ordinary steel, and then tempered in accordance with the practice employed in the ordinary metallurgy of carbon and alloy steels.

The iron carbide used in the mixture may be produced by carburizing powdered sponge iron, as by heating the same in a suitable container of iron, alundum, etc. in a gas carburizing furnace, such as a rotary gas carburizing furnace or a reverberatory furnace of the type used in roasting ores. Agitation of the powder during carburization decreases the time required to effect the desired result. We prefer to employ a soft iron which is comparatively free of impurities. The length of heating depends mainly upon the degree of carburization desired, the nature and rate of flow of the carburizing gas employed, the agitation of the material and the particle size of the powder. The carburizing process may be substantially the same as that employed in the carburizing of solid iron or steel and may be conducted in such gases as propane, natural gas, carbon-monoxide, or other well known commercial carburizing fluids by which carbon is introduced into iron by the gaseous method. It is necessary, of course, to exclude oxygen during the carburizing process. In one instance carburizing in propane gas flowing at the rate of 1.3 per cubic foot per hour at 1700 degrees F. for a period of one hour resulted in a 1 percent combined carbon in the product. However, by extending the period to 2 or 3 hours it is possible to carburize up to 2 or 3 percent combined carbon. In some cases it may be desirable to conduct the treatment at a lower temperature for a longer period in order to reduce the balling up of the powder. After carburizing the powder should be ground to break the lumps that may have formed.

The carburizing of the iron powder results in a case hardening inwardly from the surface thereof which hardness increases with the increase of carbon content. Due to the hard nature of such carburized powder, it is extremely difficult to effect cohesion of the particles during the pressing operation. We have found, however, that this difficulty can be overcome by properly annealing the carburized powder prior to pressing thereof into the desired form. This may be effected by reheating the powder after carburization to the annealing temperature thereof, this temperature depending upon the carbon content of the powder and being of the order of magnitude of from 1500 to 1700° F. The powder is then permitted to cool very slowly, over a period of an hour or more.

Annealing may also be effected by a very slow cooling of the powder following the carburization thereof. This annealing results in a softening of the carburized powder whereby it more readily coheres during the subsequent pressing operation. If the carburized powder is diluted with pure iron powder, the cohesion of the particles is further enhanced.

In place of using carburized iron powder, powdered steel may be employed, providing the same is annealed prior to compression thereof, as above set forth. The carburizing may be conducted to a point just sufficient to produce the required amount of combined carbon in the finished part in which case the powders may be pressed directly into the desired form following the carburizing and annealing process. Or the carburizing may be sufficient to produce a mixture rich in combined carbon, requiring dilution of the same with iron powder so as to obtain the desired carbon content in the steel. Other materials containing combined carbon, such as ferro alloys, may also be added if desired in addition to the carburized iron powder. During the gas carburizing described above each particles of iron powder forms its allotment of carbide, thereby facilitating the diffusion of the carbides homogeneously through the sintered mass.

For fabrication of an article the powdered material is placed in a suitable container which is filled to a depth of approximately three times the depth of the finished article. The plunger is thereafter forced into the container under the pressure of approximately one hundred thousand pounds per square inch, which pressure compacts the powder to approximately one-third of its original bulk. The exact reduction in volume of the powder, upon compression, is dependent upon the particular mixture employed. After the pressing operation the compressed powders are coherent and may be readily removed from the mold, by means of an ejector of the usual type. The compact may then be sintered at a suitable temperature in an inert atmosphere, such as hydrogen, for a sufficient period to coalesce the constituents into a uniform mass. The sintering temperature should be above the critical range of the particular composition so that the carbide will be dissolved in the austenite, as in the ordinary heat treating process of steels. After sintering the part may be quenched from the sintering temperature, in the accepted manner ordinary to steel treatment.

As an example of the specific process a mixture comprising 50 percent pure iron and 50 percent carburized and annealed iron, which contains one percent combined carbon in the iron carbide, was pressed at a pressure of approximately one hundred thousand pounds per square inch and sintered in a non-oxidizing atmosphere at a temperature of 1200 to 1300° C. for a period of two to four hours in a high frequency induction furnace. The resulting metal was subsequently hardened in accordance with the usual hardening practice and oil quenched. The structure was typical of a carbon tool steel of the particular composition produced and had a hardness and tensile strength commensurate with the amount of combined carbon employed. The micro-constituents were typical of tool steel, as for instance martensite.

It is obvious, of course, that many changes may be made in the method of compacting the powders and uniting powders of different compositions, those shown being by way of illustration only. Therefore, we do not desire to be limited to the specific processes disclosed, but contemplate all variations thereof as coming within the scope of the appended claims.

We claim:

1. The method of producing steel which comprises mixing uncarburized powdered iron and carburized and annealed iron powder in the proper proportion to give a content of combined carbon in the mixture of from 0.1 to 1.5%, pressing said mixture to render it coherent, sintering at a temperature above the critical range at which the microstructure is austenitic but below the temperature at which the metal becomes plastic, and continuing the heating beyond the time required for sintering for a period sufficient to cause diffusion of the combined carbon substantially uniformly through the mass.

2. The method of producing steel which comprises carburizing iron powder, mixing uncarbu-

rized iron powder therewith, pressing the mixture into coherent form and sintering at a temperature above the critical range where the microstructure is austenitic but below the temperature at which the metal becomes plastic, and continuing the heating beyond the time required for sintering for a period sufficient to cause diffusion of the carbide substantially uniformly through the mass.

3. The method of producing steel which comprises carburizing iron powder, annealing the carburized iron powder, diluting the same with uncarburized iron powder to produce a desired percentage of combined carbon of from 0.1 to 1.5% in the mixture, pressing the mixture into coherent form and sintering at a temperature above the critical range where the microstructure is austenitic but below the temperature at which the metal becomes plastic, and continuing the heating beyond the time required for sintering for a period sufficient to cause diffusion of the carbide substantially uniformly through the mass.

4. The method of producing steel which comprises carburizing iron powder, diluting the same with uncarburized iron powder to produce a desired percentage of from 0.1 to 1.5 percent of combined carbon in the mixture, pressing the mixture into coherent form and sintering at a temperature above the critical range where the microstructure is austenitic but below the temperature at which the metal becomes plastic, and continuing the heating beyond the time required for sintering for a period sufficient to cause diffusion of the carbide substantially uniformly through the mass.

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