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METHOD FOR PRODUCING ALLOY-STEEL ARTICLES

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ABSTRACT OF THE DISCLOSURE

This invention relates generally to the production of high-alloy steel articles, having extremely low oxygen contents and thus improved cleanliness, by powder metallurgy techniques. More specifically, the desired high-alloy steel articles having extremely low oxygen contents are achieved in accordance with the invention by placing alloy steel powder in a gas-tight container, evacuated to a pressure less than atmospheric, in the absence of further evacuation, heating said container and contents to an elevated temperature until the pressure within said container levels off, further evacuating said container to remove gaseous products therein, and thereafter compacting said powdered material while the same is in said container at elevated temperature and low pressure.

It is well known, that with high-alloy steels, such as high-speed tool steels, that their susceptibility to hardening upon quenching from the austenitizing temperature is dependent upon a fine, even carbide distribution within the material. For this purpose, it is known to produce high-alloy tool steel articles by powder metallurgy techniques. These operations include broadly the steps of providing a powder of the desired high-alloy steel, heating the powder in a container to an elevated temperature, at which sintering or fusion of the material results, and thereafter compacting said material by the application of pressure. In operations such as this, the alloy is in finely divided form, and consequently an extremely large surface area thereof is exposed to the atmosphere. This results in extremely rapid and substantial oxidation of the material. Because the alloying elements commonly found in the material, such as chromium, molybdenum, tungsten and vanadium, are readily oxidizable and since such an extremely large surface area thereof is exposed to the atmosphere, substantial oxidation will occur even under conditions of extremely short exposure to an oxidizing atmosphere. As a result of this oxidation, the products resulting after the final compacting step are characteristized by extremely high oxygen contents, which as is well known result in a product of poor cleanliness.

It is accordingly the primary object of the present invention to provide a method for producing high-alloy steel articles having fine, uniformly dispersed carbides by powder metallurgy techniques.

A more particular object of the invention is to provide a combination of heating and evacuating steps prior to compacting that result in the removal of oxides present in the powder material to extremely low levels prior to compacting.

A further object of the invention is to provide a method for removing oxygen from high-alloy steel powder prior to compacting thereof wherein the material is heated to a temperature and under conditions at which the oxides are reduced without causing substantial agglomeration of carbides and sulfides, the resulting gaseous products of reduction are then removed, and the material is maintained in a non-oxidizing environment until compacting is achieved.

These and other objects, as well as a complete understanding of the invention, may be obtained from the following description and examples.

In the practice of the invention, a charge of finely divided high-alloy-steel powder, which may for example be AISI M–2 tool steel of for example, —100 mesh, is placed in a gas-tight container. The container may, for example, be a thin-walled, mild steel cylinder having a flat top and bottom. The interior of the container is connected to an evacuating means, which may be any type vacuum pump well known for this purpose, and the container is evacuated to a pressure of at least less than atmospheric, for example about 50 microns or less. During this pumping operation, the interior of the container should be at ambient temperature so that any moisture present in the container will be removed rather than result in further oxidation of the alloy particles. Pumping is discontinued and the container is placed in a furnace at high temperature, for example 2100° F., and heated to an elevated temperature of, for example, a temperature substantially equal to that of the furnace or at least about 2000° F. Temperatures on the order of 2000° F. are required in the operation to insure reduction of the oxides of the alloying elements. Heating is continued until the pressure within the container ceases to rise. The time required for this operation will, of course, depend upon the quantity of material, the oxygen level, and the furnace temperature. The pressure within the container levels off at, for example, about 300–400 mm.; however, as with heating times this will depend upon the quantity of material, the oxygen level, and the furnace temperature. When the pressure within the container ceases to increase, as described above, pumping of the container is begun, while the same is maintained at elevated temperature. The evacuation or pumping is continued until the pressure within the container is decreased to a level at which the rate of decrease levels off, which indicates that the outgassing rate has stabilized. Typically, the rate may level off at about 300–350 microns per minute and at a pressure of about, for example, 25 microns. The stabilization of the outgassing rate indicates that the gaseous products in the container have been removed as completely as possible with the specific equipment being used. The container and its contents are then subjected to compacting at elevated temperature and low gas pressure. For this purpose, any convenient means may be employed. For example, if the material is contained within a cylindrical container having flat ends, compacting may be achieved by placing the container within a die conforming susbtantially to the cross section of the container, and a plunger may be inserted within the die to effect the desired consolidation of the material within the container. After compacting, the container is removed by a machining or pickling operation or a combination of both. Alternately, consolidation may be achieved by placing the container within a gas-pressure vessel wherein gas in introduced to the vessel under pressure to collapse the container and thereby compact the material. Also, the container may be forced through a die, whereby the material is compacted by extrusion prior to removal of the container.

It is not necessary that the material be compacted to 100 percent density by a single operation. For example, it is possible to compact the material by one of the methods discussed above to an intermediate density of, for example 95 percent, remove the container from the material by machining or pickling, and then subject the compact to a conventional reduction operation, such as rolling, to achieve the desired final shape and density. By compacting in this manner, close control of the dimensions of the final product is readily achieved.

To insure that moisture removal from within the container has been completed prior to heating to elevated temperature, it is preferred that prior to this heating step,

the container be heated to a relatively low temperature, for example 300° F., for a short time while being evacuated. After this operation, evacuation is discontinued and then the container is transferred to a furnace for heating to the elevated temperature desired for compacting and oxide reduction. The pumping at ambient temperature followed by pumping for a short time at a slightly elevated temperature has resulted in almost complete removal of moisture from the container, which of course greatly minimizes oxidation within the container during the initial stages of heating. In this manner, the processing time may be accordingly reduced, because there is less oxide to be reduced and correspondingly less gaseous product thereof to be removed during the evacuating stage.

The maximum temperature and heating time are such as to avoid substantial growth and agglomeration of carbides, as well as sulfides. For this purpose, heating under sintering conditions wherein substantial melting and fusion occurs must be avoided. In this manner, a compacted article having the desired fine, uniformly dispersed carbides is produced.

Example I

AISI M–2S —100 mesh powder in an amount of 2.25 pounds was placed in a mild steel cylindrical container having a 2.5-inch inside diameter and a three-inch length. The container had a flat top and bottom closure and was, of course, gas tight. The container with the powder therein was placed in a furnace and heated for one hour to a temperature of approximately 2200° F. While at elevated temperature, the container and material were extruded in a single operation to achieve a compact with 100 percent density. Specimens were obtained from the extruded material and tested for oxygen content and carbon content. The carbon content was 0.89 percent and the oxygen content was 443 p.p.m.

Example II

A container of AISI M–2S powder was prepared as described in Example I, except that carbon was added to the particle charge by coating the particles with lampblack in an amount of 0.10 percent by weight. After heat treating and extruding as described in Example I, specimens were taken and tested for oxygen and carbon contents. The carbon content of the specimens was 0.98 percent and the oxygen content was 299 p.p.m.

Example III

A charge of AISI M–2S powder was placed in a container as described in the above two examples, except that the container was adapted for connection through a stem to a vacuum pump. The container while at ambient temperature was evacuated to a pressure of about 10 to 15 microns. The container was then placed in the furnace at a temperature of approximately 2200° F. and pumping was contined for about five minutes to insure complete moisture removal from the interior of the container. Pumping was then stopped. The temperature of the container increased to about 2200° F. and the pressure therein increased from about 10 to 15 microns to about 48 mm., at which point the pressure leveled off and ceased to increase. At this time, pumping was begun and continued for about four hours and 30 minutes at which time the outgassing rate leveled off at about 354 microns per minute. The pressure within the container at this time was 25 microns. The container and material were compacted by extrusion as described above while at a temperature of 2200° F., and samples were taken from the extrusion for carbon and oxygen determinations. The carbon content was 0.87 percent and the oxygen content was 29 p.p.m.

Example IV

An additional operation was performed which was identical to that of Example III above, except that 0.15 percent carbon was added to the particle charge by coating the particles with lampblack. In addition, the following differences prevailed during the operation over those described in Example III. During the heating to elevated temperature, the pressure buildup leveled off at 79 mm. This was probably caused by the additional moisture introduced by the lampblack and also because of the additional reaction with oxygen caused by the additional carbon present as lampblack. In addition, during the pumping stage, the outgassing rate leveled off when the pressure within the container reached about 20 microns; the outgassing rate leveled off at 300 microns per minute. As in Example III, specimens were taken from the finally extruded material and analyzed for carbon and oxygen. The carbon content was 1.03 percent and the oxygen content was 40 p.p.m.

Example V

Iron powder of —100 mesh in an amount of 49.5 grams was mixed with 0.5 gram of lampblack and placed in a container as described with respect to the above examples. The container at ambient temperature was evacuated to a pressure level of about 15 to 20 microns. Evacuation of the container was continued while the same was heated at 300° F. for 20 minutes. As described above, this insured the complete removal of moisture from the container interior. Evacuation of the container was continued while the same was placed in a furnace at 2100° F. where it was heated for one hour. During the first 10 minutes of heating, the pressure within the container increased to about 300 to 400 microns and during the remaining 50 minutes of heating and pumping was reduced to 55 microns, at which time the outgassing rate leveled off. It is to be emphasized that pumping continued uninterrupted during the entire heating cycle. The container was then removed from the furnace and compacted by pressing with a 200-ton press. The compact, while still within the container, was heated to about 2000° F. for about one hour for the purpose of attempting to distribute any carbon present within the material. Polished cross sectional specimens of material were obtained and examined. The examination of a polished specimen showed substantial quantities of free graphite and iron oxides which indicated that there was substantially no reaction between the carbon and the oxides during heating. The samples were then reheated at 2000° F. for about one hour and then re-examined; after this heat treatment there was no visible difference in the structure. The presence of the graphite particles, rather than iron carbides, conclusively shows that there was no substantial carbon reaction during heating. The specimens were again heated to about 1700° F. for about three to four minutes, water quenched, and R_c hardness determinations were made. The random hardness throughout the test specimens ranged from 12 to 24 R_c. The same specimens were heated for about one hour at 2000° F. and thereafter tested for hardness. All hardness determinations were below zero R_c. The above lack of hardening in the quenched condition is a further indication of the absence of carbon reaction during the simultaneous heating and evacuating of the container.

Example VI

An additional operation identical to that described above with reference to Example V was performed except that during heating at 2100° F., pumping was discontinued for thirty minutes of the one hour heating time. Examination of polished specimens of the material showed a substantially complete absence of free graphite and iron oxides. This shows that under these conditions the carbon reacted with the oxides to form gaseous products thereof, which were removed during the evacuation stage of the operation. When the material was tested for hardness in the quenched condition as described in Example V above, determinations of 59 to 61 R_c were obtained. After heating at one hour for 2000° F., the hardness determinations were from 53 to 56 R_c. This shows that the

carbon reacted with the iron during heating to elevated temperature.

By comparison of Examples I and II with Examples III and IV, it is apparent that by heating the particle charge within the container to an elevated temperature and then evacuating the container when the pressure therein reaches a maximum, in accordance with the method of the present invention, a drastic reduction in the oxygen content of the final compacted article is achieved over that obtained by heating to elevated temperature without subsequent evacuation, as is the case in Examples I and II. It may be seen by comparing Example V with Example VI that it is necessary to discontinue evacuation of the container while the same is being heated to elevated temperature and until such time as the pressure within the container levels off at a maximum level. If this condition does not prevail, as may be seen from the results of Example VI, the conditions for carbon reaction with oxygen are not present as evidenced by the presence of graphite and iron oxide in Example V wherein the container and contents were subjected to pumping during the entire heating cycle.

Although various embodiments of the invention have been shown and described herein, it obvious that other adaptations and modifications may be made by those skilled in the art without departing from the scope and spirit of the appended claims.

What is claimed is:

1. A method for producing fine-grained, homogeneous articles characterized by extreme cleanliness from oxygen-contaminated, carbon-bearing metal particles comprising confining a charge of said particles in a substantially moisture-free gas-tight container, in the absence of continuous evacuation heating said charge and container to an elevated temperature and for a time sufficient to substantially reduce oxides present on the powder without causing substantial agglomeration of carbides and sulfides, beginning further evacuation of said container when the pressure therein reaches a predetermined level, continuing evacuation until removal of gaseous reaction products is substantially complete, and compacting said charge within said container while at low pressure and elevated temperature.

2. A method according to claim 1 wherein evacuation of said container is continued for a short time during the beginning of heating of said container to elevated temperature.

3. A method according to claim 1 wherein said pressure sufficient to effect substantially complete removal of moisture from said container is less than atmospheric.

4. A method according to claim 1 wherein said charge and container are heated to an elevated temperature of at least about 2000° F.

5. A method according to claim 1 wherein said predetermined pressure level is the level at which the pressure ceases to increase.

6. A method according to claim 1 wherein said charge is compacted within said container to form an article of intermediate density and is thereafter subjected to additional compacting to a final density.

7. A method according to claim 6 wherein said article of intermediate density is removed from said container prior to additional compacting to a final density.

8. A method according to claim 1 wherein said powders are coated with a carbon-containing material prior to charging the container.

9. A method according to claim 8 wherein said carbon-containing material is lampblack.

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