

June 17, 1969

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3,450,528

METHOD FOR PRODUCING DISPERSIONED HARDENABLE STEEL

Filed July 25, 1968

FIG. 1.

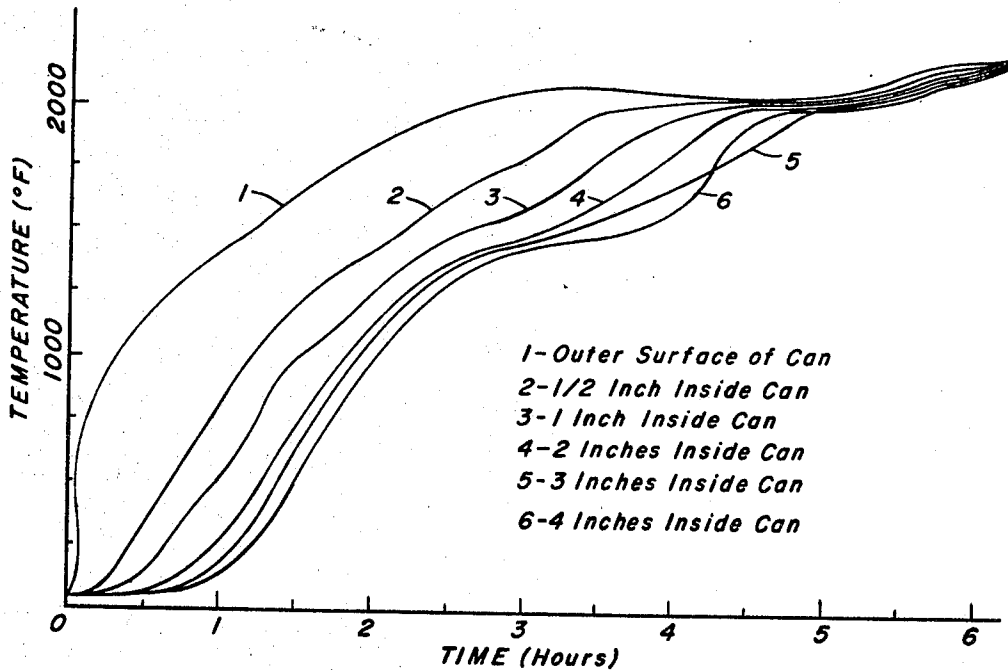
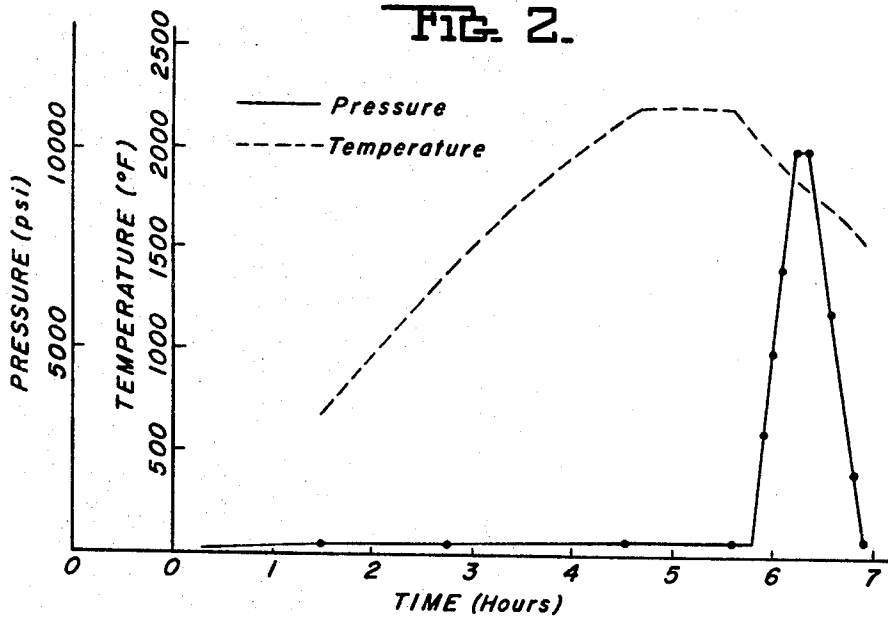


FIG. 2.



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METHOD FOR PRODUCING DISPERSIONED HARDENABLE STEEL

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Continuation-in-part of application Ser. No. 641,034,
May 24, 1967. This application July 25, 1968, Ser.
No. 766,656

Int. Cl. B22f 3/14

U.S. Cl. 75—203

10 Claims 10

ABSTRACT OF THE DISCLOSURE

The invention relates generally to the art of powder metallurgy and in more particular aspects to a powder-metallurgy practice wherein compacting of finely-divided metal powders into dense articles is achieved by the use of a fluid-pressure vessel, commonly termed an "autoclave." The method of the invention includes the basic steps of placing a charge of powdered metal consisting of spherical particles not larger than about —30 mesh in a container. The container, with the powdered metal charge therein, is heated to a temperature above about 0.7 homologous fusion temperature of the powdered metal but below the fusion temperature thereof. The container is then transferred to a fluid-pressure vessel for compacting to a density of at least about 95% by the application of pressure within the range of about 10,000 to 30,000 p.s.i. It is necessary that the compacting be completed before said powdered-metal charge has cooled to a temperature below about 0.7 homologous fusion temperature of said powdered metal.

This is a continuation-in-part of my copending patent application, Ser. No. 641,034, now abandoned, filed on May 24, 1967.

In the manufacture of many metal articles, and particularly tool steel articles, it is highly desirable to have the product characterized by very small, well distributed carbides in a fine-grained matrix. In tool steel articles, this structure provides maximum hardness upon quenching from austenitizing temperature. Although the above-described properties are highly desirable in tool steel articles, cast high-alloy tool steels are very susceptible to eutectic-type carbide segregation during solidification. This effect becomes even more pronounced in the presence of cooling-rate decreases. Consequently, to minimize this effect a small size ingot is desired. These eutectic carbides are very difficult to dissolve upon austenitizing and if such is attempted by employing long austenitizing times, excessive grain growth will be the expected result. If a cast ingot characterized by eutectic-type carbide segregation is rolled, the segregate is elongated rather than being uniformly dispersed. It is therefore common practice prior to rolling such material to forge the metal to break-up and disperse any segregates present therein. Even with this technique, however, it is not possible to achieve completely random dispersion of carbides.

It has been discovered, in the practice of the present invention, that the desired random complete dispersion of carbides in high-alloy tool steel articles is produced by using atomized particles having the desired randomly dispersed carbides and compacting these particles by uniform pressure application at a temperature and pressure sufficient to cause bonding and substantially complete densification without substantial carbide agglomeration. The temperature to which the particles are heated for compacting must be below that at which substantial carbide agglomeration will occur.

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The metal powder as described above is compacted by the use of a fluid-pressure vessel, commonly termed an "autoclave." In vessels of this type, high pressures are developed by the use of a gas such as helium. The gas pressure within the autoclave acts uniformly throughout a charge of powdered metal placed in a container for compacting. Pressure transmitted to the powdered-metal charge in this manner creates the metal flow that is necessary for compacting without destroying the desired completely random dispersion of carbides. More specifically, by the application of the fluid pressure, the particle surfaces are held in contact for a time sufficient to permit solid-state bonding to occur. To achieve bonding, however, it is necessary that the particles be at an elevated temperature and under pressure above a minimum level, which is dependent upon the temperature. If the temperature is too high, the carbides will tend to agglomerate by sintering and consequently the desired random carbide dispersion will not be achieved, even under conditions of uniform pressure application within the autoclave. The temperature at which this occurs with a particular metal may be termed as the fusion temperature. Alternately, if the temperature is too low, thorough bonding and compacting to densities approaching 100% will not be achieved. The density of the powder prior to compacting should be relatively high, for example, at least about 60%, to permit subsequent compacting to densities of substantially 100% while avoiding buckling or irregular inward folding of the walls of the container for the powdered metal being compacted. For this purpose it is necessary that the metal particles be substantially spherical and of a size not larger than —30 mesh. Since the container must be removed from the compact, such as by acid pickling or machining, after compacting has been completed, any substantial inward folding of the container during compacting must be avoided. Otherwise, substantial quantities of the compact will be removed during removal of the container. If the particles are not substantially spherical to the extent necessary for satisfactory compacting, such may be to some extent overcome by precompacting the powder prior to placing the same in the autoclave for complete compacting to a density approaching 100%. This preliminary or precompacting may be achieved by a mechanical operation, such as the use of a die into which the powder-filled container is placed for compaction by the application of a ram to the die. For a rapid commercially practical compacting operation, it is necessary to heat the powdered metal charges to satisfactory compacting temperatures within the containers prior to placing the same in the autoclave for compacting. Heating prior to compacting may require two hours or more, because to achieve adequate bonding and compact density, thorough "soaking" is advantageous, because the temperature of the powdered metal must be substantially uniform throughout the container. Therefore, it would not be practical to place the containers at room temperature within the autoclave and then heat for the long times required before the application of fluid pressure for compacting. Hence, in the practice of the invention, prior to compacting it is necessary to heat the powder-filled containers to a temperature below that at which carbide agglomeration results but yet sufficiently high that transfer to the autoclave, pressurization of the autoclave, and compacting to substantially final density is achieved before the temperature of the metal powder drops below that necessary for adequate bonding and compacting. An additional factor that adds considerable time to the heating cycle prior to compacting is that the powder-filled container should be completely "outgassed" before compacting to provide substantial oxygen removal from the container. This requires connection of the container interior to a

suitable vacuum pump for removal of gaseous reaction products produced during heating of the powdered metal within the container. If "outgassing" is not achieved, the resulting compact will be characterized by the presence of detrimental oxides and other surface impurities that adversely affect bonding of the metal powder and the quality of the final product.

It has been found that these purposes may be achieved by heating the metal powder charge to a temperature above 0.7 homologous temperature of the metal powder, but not above the temperature of fusion of the metal powder. When maintained within these temperature limits, the metal powder can be satisfactorily compacted by the application of pressure within the range of 10,000 to 30,000 p.s.i., with the amount of pressure varying inversely with the temperature of the material being compacted. If difficulty is encountered in maintaining temperature until compacting is completed, it is possible to provide heating means within the autoclave to maintain temperature or substantially decrease the rate of temperature drop of the material during pressurizing of the autoclave and compacting therein.

For the purposes of the invention, the homologous fusion temperature may be defined as the actual absolute temperature of the powdered metal, for example degrees Renkin, divided by the absolute temperature of fusion of the metal particles. For materials such as tool steel, this will typically result in a lower temperature limit of about 1800° F. and an upper limit of about 2200° F. As pointed out above, the pressure necessary to achieve compacting to the required density and adequate bonding will vary inversely, within the range 10,000 to 30,000 p.s.i., with the temperature of the material being compacted.

A more complete understanding of the invention may be obtained from a consideration of the following description, specific examples and drawings, in which:

FIGURE 1 is a graph showing the heating times required to heat powdered metal in various cross-sectional areas of a filled container to a required compacting temperature; and

FIGURE 2 is another graph showing a heating and compacting cycle for powdered metal.

To demonstrate the long heating times involved in heating a charge of powdered metal to satisfactory compacting temperature within a container, the following experimental work was performed. A tubular carbon steel container measuring eight inches in diameter by eight inches in length was filled with M2S tool-steel powder of about -100 mesh. Moisture was removed from the container by evacuation, and it was sealed against the atmosphere. Thermocouples were positioned at the center and on radials of 1, 2, 3, 3.5 and 4 inches extending to a mid-length position. The container was placed in a cold globar furnace and the thermocouple leads were connected to a recorder.

As may be seen from FIG. 1, it took approximately five hours for the entire contents of the container to reach a temperature of about 2200° F. As explained hereinabove, it is necessary to insure that the center material within the container is at satisfactory temperature, because otherwise the density of the material in the center of the resulting compact will not be adequate. As would be expected, and as may be seen from FIG. 1, the material in the outermost portions of the container reached the required compacting temperature much sooner than the material relatively closer to the center of the container. These data show the long heating times required prior to compacting, which precludes heating of the powder metal to the high temperature required for compacting within the autoclave prior to compacting.

Additional testing was performed by providing a charge similar to that described above in a container of mild carbon steel having an outside diameter of 3.5 inches and a length of 6 inches. The prepared specimen had a thermocouple positioned near the geometric center. The tem-

perature reading at this position was used as a reference during the heating cycle shown in FIG. 2. The specimen was heated to a temperature of about 2200° F., at which time it was subjected to fluid pressure in an autoclave on the order of 10,000 p.s.i. It may be noted from FIG. 2 that the temperature of the specimen, as measured by the thermocouple, decreased to about 1800° F. prior to compacting by fluid-pressure application. Subsequent metallographic analysis of the resulting compact indicated that the density was relatively low throughout the entire compact as contrasted with the higher-density compacts that may be achieved by conducting the compacting at higher temperatures within the limits of the present invention.

Although one embodiment of the invention has been shown and described herein, it is obvious to those skilled in the art that other adaptations and modifications may be made without departing from the scope and spirit of the appended claims.

I claim:

1. A method for producing hardenable steel articles from ingredients metals comprising placing a charge of steel particles containing a random dispersion of carbides to be compacted in a container, heating said powdered-metal charge within said container to an elevated temperature, said elevated temperature being above a selected compacting temperature but below the temperature at which substantial carbide agglomeration will occur, placing said container and powdered metal charge in a fluid pressure vessel, increasing the fluid pressure within said vessel to a level sufficient to compact said powdered-metal charge to a density of at least 95 percent, said compacting to this density being completed before said powdered-metal charge has cooled to a temperature below said selected compacting temperature.

2. The method of claim 1, wherein said selected compacting temperature is above about 0.7 homologous fusion temperature of the powdered-metal charge.

3. The method of claim 1, wherein the fluid pressure within said vessel is increased to a level within the range of 10,000 to 30,000 p.s.i.

4. The method of claim 3, wherein said fluid pressure varies inversely with compacting temperature within said pressure range.

5. The method of claim 1, wherein said charge of powdered metal comprises substantially spherical particles.

6. The method of claim 5, wherein said particles are not larger than about -30 mesh.

7. The method of claim 1, wherein said powdered-metal charge is compacted to an intermediate density prior to being placed in said pressure vessel.

8. The method of claim 1, wherein said powdered-metal charge is subjected to additional heating within said pressure vessel prior to completion of compacting.

9. The method of claim 1, wherein said powder-filled container is outgassed prior to compacting within said pressure vessel.

10. A method for producing hardenable steel articles from powdered ingredients comprising placing a charge of powdered metal having a particle size not larger than about -30 mesh and having a random dispersion of carbides in a container, heating said powdered metal charge within said container to a temperature above about 0.7 homologous fusion temperature of the powdered metal and below the temperature at which substantial carbide agglomeration occurs, compacting said powdered-metal charge to a density of at least 95 percent by increasing the fluid pressure within said vessel to a level within the range of 10,000 to 30,000 p.s.i., said pressure level varying inversely with said temperature, said compacting being completed before said powdered metal charge has cooled to a temperature below about 0.7 homologous fusion temperature of said powdered metal.

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References Cited

UNITED STATES PATENTS

2,725,228	11/1955	Dodds -----	75—226
3,053,706	9/1962	Gregory -----	75—203 X
3,235,958	2/1966	Gautheron -----	75—214 X
3,340,056	9/1967	Hodge -----	75—226
3,369,892	2/1968	Ellis -----	75—203 X
3,380,861	4/1968	Frehn -----	75—203 X

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FOREIGN PATENTS

616,393 3/1961 Canada.

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U.S. Cl. X.R.

75—204, 214, 226

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,450,528

Dated June 17, 1969

Inventor(s) Vernon R. Thompson

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 4, line 22, after "from" insert -- powdered --;
same line 22, after "ingredients" cancel "metals".

SIGNED AND
SEALED
JUL 21 1970

(SEAL)

Attest:

Edward M. Fletcher, Jr.

Attesting Officer

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Commissioner of Patents