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Conta

[11] **3,894,892**

[45] **July 15, 1975**

[54] **PROCESS FOR HEATING AND SINTERING FERROUS POWDER METAL COMPACTS WITH RADIO FREQUENCY MAGNETIC FIELD**

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 285,965, Sept. 5, 1972, Pat. No. 3,779,747.

[52] **U.S. Cl.** **148/108;** 148/12.9; 148/154; 219/9.5; 219/10.41; 219/10.43; 148/103; 75/200

[51] **Int. Cl.** **C21d 1/04**

[58] **Field of Search** 148/108, 105, 154, 12.9, 148/103; 75/200, 214, 226; 264/56; 219/9.5, 10.41, 10.43

[56] **References Cited**

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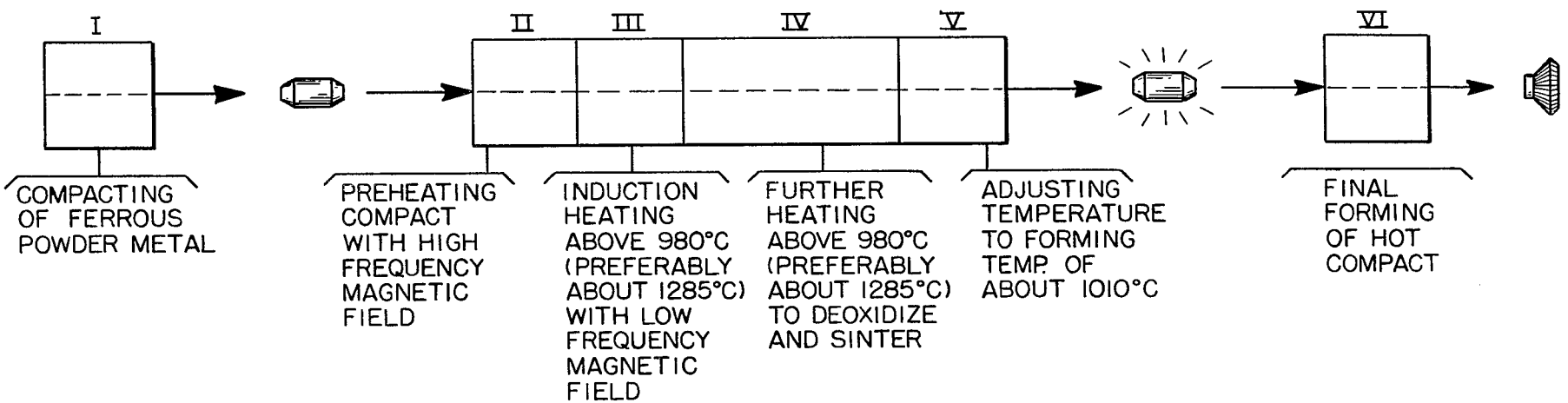
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[57] **ABSTRACT**

A relatively high frequency (in the radio frequency range) magnetic field is used for reducing electrical resistivity of ferrous powder metal compacts prior to subjecting the compacts to a relatively lower frequency magnetic field which rapidly raises the temperature level of the material of each compact to a sintering temperature.

7 Claims, 6 Drawing Figures

FIG. 1



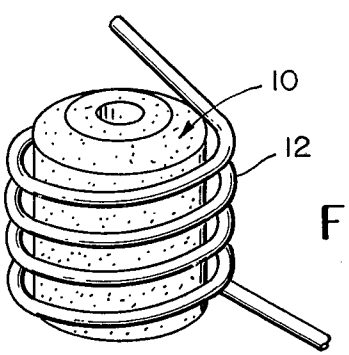


FIG. 2

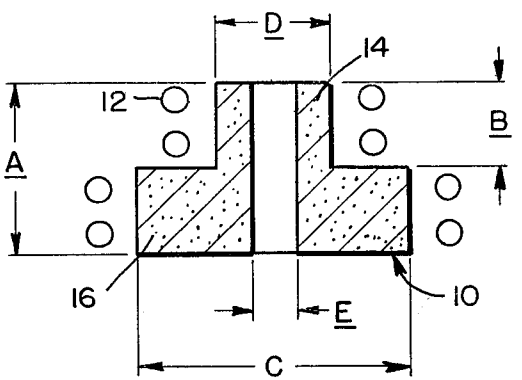


FIG. 4

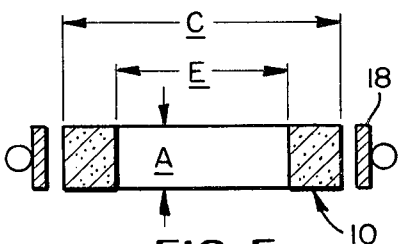


FIG. 5

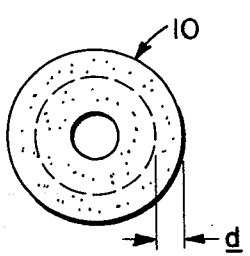


FIG. 3

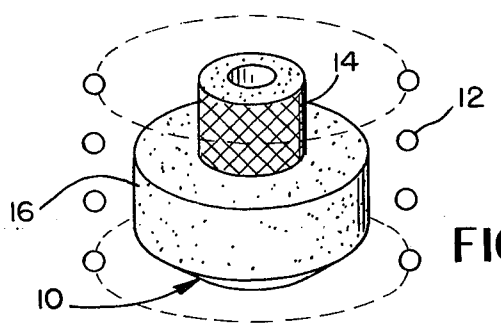


FIG. 6

Sept. 16, 1975

PROCESS FOR HEATING AND SINTERING FERROUS POWDER METAL COMPACTS WITH RADIO FREQUENCY MAGNETIC FIELD

RELATED APPLICATION

This application is a continuation-in-part of my co-pending application Ser. No. 285,965, filed Sept. 5, 1972 now U.S. Pat. No. 3,779,747, issued on Dec. 18, 1973.

BACKGROUND OF INVENTION

The present invention particularly relates to processes for heat treating and sintering powder metal compacts to prepare the compacts for final forming operations which increase the density and establish final shapes of the compacts as finished parts, but the discoveries of this invention may be applied to processes which sinter powder metal parts as a final step in a technique which does not require final forming or forging of the parts. The invention is specifically concerned with improvements in heating and sintering of compacts formed from ferrous metal powders for use in the production of high strength finished parts such as bevel and spur gears, and the like.

Various processes are known for compacting loose powder material into a coherent form which can be more easily handled during required treatments of heating and sintering prior to final forming or forging operations. Typically, a precisely measured quantity of the metal powder is compacted with isostatic equipment, or with mechanical die means, into an initial shape having characteristics of increased density and coherency. This "green" compact form is then subjected to heating and forming treatments required for driving off lubricants or binders, deoxidizing the material of the compact, coalescing particles of the compact, and increasing density and shape of the compact into a finished part.

It is also known to raise the temperature of powder metal compacts to a level at which individual particles of the compact coalesce into a homogeneous mass without actual melting of the particles (also described as diffusion bonding). This treatment is commonly carried out at about 1120°C with known sintering equipment, however, it has been recently discovered that somewhat higher temperatures can be used for effecting coalescence of particles of a powder compact and for simultaneously deoxidizing the compact. In the context of this specification and its claims, references to sintering or sintering temperatures are intended to define deoxidation of a compact as well as the formation of a homogeneous mass from a metal powder.

Various processes and means have been attempted for carrying out efficient sintering of powder metal compacts so that the compacts will be in a desired metallurgical condition or in a condition to be formed in a die to increase their densities to nearly 100% of theoretical density and to establish a final shape of a finished part. Known processes for heating and sintering compacts have been somewhat inefficient or uneconomical because of excessive heating times or energies required to carry out the necessary conditioning of a compact for final forming or other use. For example, it is known to sinter a compact with radiant heating means to a level of about 1120°C for a relatively long period of time of about 20 minutes in order to fully deoxidize and sinter the powder metal material of the compact. It is also known to preheat all of the material of a compact at a temperature below the subsequent sintering temperature so as to initially burn off lubricants which may be contained within the material of the compact as a result of a particular compacting process requiring such lubricants. It has been suggested that such a burning-off step should precede higher temperature sintering of a compact irrespective of whether sinterng is carried out with a radiant furnace or with induction heating means. Very recently, it has been suggested that surface lubricants be burned off in a radio frequency magnetic field prior to sintering in a lower frequency field, and it has been proposed (see British Pat. Specification 1,299,064, published Dec. 6, 1972) to use a radio frequency field to carry out the complete sintering of a powder metal part. Magnetic fields in the radio frequency range have also been used to harden or treat surfaces of fully dense objects.

Induction heating has been considered as an interesting way for raising the temperature of a ferrous metal powder compact because it permits a controlled addition of substantial energy into the body of the compact to thereby raise its temperature in a relatively short time. However, it has been discovered that when a commercially available, low frequency (3 kiloHertz, for example) induction field is utilized as a means for raising the temperature of a compact to a sintering temperature level, there is a characteristic "incubation" period at the onset of each heating cycle in which there appears to be a delayed temperature response to the induction field. This delay is significant, sometimes amounting to several minutes depending upon the size, density, and material of the compact, and this adds considerably to the cost of energy required for an induction heating treatment. It is not known precisely why a powder compact exhibits an incubation period at the beginning of an induction heating cycle, but it is apparent that the compact exhibits high electrical resistivity, is very inefficient in absorbing power, and that initial heating is slow in an induction field. One attempt to overcome this problem has required a presintering of the compact with resistance heating means, as described in U.S. Pat. No. 3,708,645. The theory of this process appears to be one of raising the temperature of the material of the entire compact to a point where preliminary sintering takes place between powder particles of the compact so as to improve heat conduction from outside surfaces to inside sections of the compact. However, resistance heating of a compact can be somewhat difficult to handle because of a need to directly contact each part with electrodes of the resistance heating equipment.

Thus, there is a need for an improved process for conditioning the material of a powder metal compact so as to take advantage of presently available induction heating equipment in a sintering operation.

BRIEF DESCRIPTION AND SUMMARY OF INVENTION

My co-pending patent application Ser. No. 285,965 (now U.S. Pat. No. 3,779,747) describes an invention for improving treating processes for ferrous powder metal compacts so that such compacts can be economically and efficiently heat treated in a continuous process which carries each treated compact to a final forming operation where its density is increased to approximately 100% of theoretical value and its shape is

established for a finished part. The invention described therein provides for an overall descrease in time and energy required for heat treating such compacts through a use of induction heating in an efficient and economical manner for at least a part of the heat treatment of each compact. More specifically, my co-pending application emphasized a preheating step which involved a low temperature preheating of only outside surfaces of a compact so as to produce an irreversible change in characteristic for the compact, resulting in a substantially reduced response time for the compact in an induction field. The preheating step was preferably carried out at about 205°C but below the sintering temperature for the material of the compact.

The present specification will emphasize my further discovery that the incubatioin period (or the time it takes for a compact to effectively couple with a magnetic field of an induction heating system) of a given compact in a relatively low frequency induction field can be substantially reduced or eliminated by a pretreating step which involves the placing of a powder metal compact in a magnetic field of a relatively high frequency (in radio frequency ranges of around 50 kHz or above) for a sufficient length of time to substantially reduce the electrical resistivity of the powder metal compact. It is not known just what mechanical or chemical changes take place when the compact is pretreated in a high frequency induction field, but it can be observed that the pretreated compact has substantially less electrical resistivity and responds much more readily to lower frequency induction heating than would be the case if the compact were not pretreated at all. Since the pretreating step can be carried out in a very short period of time, on the order of one second or more depending upon the size of the compact, there is an overall reduction in the total power and time required to carry out induction heating of the compact to a sintering level.

Pretreating of a compact with certain radio frequency induction fields can produce relatively high temperatures in the outer surface of the material of the compact, thereby resulting in something similar to a highly localized presintering of the compact. From this it can be theorized that such presintering has an effect of substantially reducing electrical resistivity of the compact in those sections where the internal temperatures are high enough to result in improved particle-to-particle bonding of the powder. Such a theory, however, does not completely explain the improvement in response time for compacts which do not reach sintering temperatures in their outer surfaces and for those sections of the compact which are adjacent to the high temperature areas of reaction to the radio frequency induction field. The adjacent sections appear to experience a low temperature (i.e., below sintering temperature) change of the type described in my co-pending application Ser. No. 285,965 (now U.S. Pat. No. 3,779,747), and this low temperature change could result from heat transfer from the outer surfaces to the interior sections of the compact. Thus, the use of a high frequency induction field can result in a composite change in a compact in which a relatively localized penetration of eddy currents induced by the high frequency field will result in a significant improvement in induction response time for the compact.

In accordance with the present invention, a basic process will be emphasized in which there is a pretreating of powder metal compacts with a relatively high frequency (for example, radio frequency range) field for a sufficient period of time to substantially reduce the time it takes to subsequently raise the temperature of the compact to a sintering level in a lower frequency induction field. In its broadest form the invention comprises the steps of (a) rapidly adjusting resistivity of a "green" powder metal compact with a sufficiently high frequency magnetic field to obtain a desired depth of current inducement in the compact for the particular electrical resistivity characteristics of the compact, followed by (b) a step of adjusting the frequency of a magnetic field downwardly to maintain a desired depth of current inducement to efficiently raise the temperature of the powder metal compact to a sintering level. In this sense, there could be a series of adjustments of frequency of the magnetic field, if desired and if equipment were available for offering such adjustments, however, the initial decrease in resistivity is so rapid there appears to be no real need for more than a single adjustment in frequency after the pretreating step.

Reduced to practical terms, and keeping in mind availability of commercial equipment for generating high and low frequency magnetic fields, the invention can be practiced by pretreating the powder metal compact with a radio frequency magnetic field, followed by a heating of the compact to sintering level in a low frequency (1 to 10 kHz) induction field.

Because of the relatively localized penetration of radio frequency induction fields in untreated compacts, it is possible to utilize the principles of the present invention to selectively pretreat different portions of a single compact so as to produce different response characteristics of different parts of the compact in a low frequency induction field. Such a use of selective treatment may be useful in pretreating powder metal compacts having configurations which include major and minor diameter portions which are to be heated in an induction coil of a single common diameter (that diameter being larger than the major diameter). By improving response time of one portion of the compact relative to another portion thereof, it is possible to balance out the overall response of such a compact in a relatively simple coil which does not have to be custom designed to the particular shape of the compact.

These and other features and advantages of the invention will become apparent in the detailed discussion which follows. In that discussion reference will be made to the accompanying drawings as briefly described below.

BRIEF DESCRIPTION OF DRAWINGS

FIG. 1 is a schematic illustration of a continuous process by which a compact of ferrous powder metal is prepared and treated in accordance with the present invention for use in a final forming operation;

FIG. 2 is a view of a typical powder metal compact configuration as it would be positioned in a coil for producing a radio frequency magnetic field;

FIG. 3 is a sectional view taken across the middle of the compact shown in FIG. 2 to illustrate sections which are treated by a radio frequency induction field;

FIG. 4 is an elevational view, in section, of a compact shape which was used in an experiment of the present invention;

FIG. 5 is an elevational view, in section, of another compact shape used in an experiment of the present invention; and

FIG. 6 is a view of a compact configuration in which there are major and minor diameter portions which can be selectively treated in accordance with the present invention.

DETAILED DESCRIPTION OF INVENTION

The process depicted in FIG. 1 is especially useful in producing high strength gear forms which exhibit equal or superior characteristics to present day gears manufactured by a method of cutting and removing material from a blank. The gear forms which can be produced by the process of the present invention include various known bevel gears as well as spur gears and other types of gear designs and gear tooth forms.

Station I of FIG. 1 comprises a place at which a ferrous metal powder is compacted to increase its density and to create a coherent preform or "green" compact which can be easily handled in subsequent steps of heating and forming. The compacting technique does not form a separate part of the present invention and may consist of any known isostatic or mechanical method for increasing metal powder density to about 70 to 90% of its theoretical full density. Prior to compacting, the ferrous metal powder is selected or prepared to provide for a controlled admixture of carbon for reacting with combined oxygen and for forming an alloy with the ferrous metal of the powder. For example, a commercially available, water atomized, annealed metal powder having a high ferrous content of about 98% by weight of the powder can be used. About 0.20 to 0.80% by weight of graphite is admixed with the metal powder, and the mixture is subjected to isostatic compacting to produce a preform or compact having about 75 to 90% of the theoretical full density for the selected material. It is not essential that a lubricant binder be added to powder mixture when isostatic techniques are used for compacting, and therefore, a subsequent step for removing such a lubricant or binder is not required, as is the case with certain other compacting methods.

After compacting, the powder metal compact is delivered to suitable apparatus for carrying out a series of heating treatments in accordance with the present invention. These heating treatments may be included as a part of a continuous process which provides for delivery of compacted preforms from a compacting machine to the heating apparatus which will carry out critical steps of heating as the compacts move through a series of heating zones within the apparatus. Such continuous processes include continuous delivery of preforms through a series of stations as well as delivery of batches of preforms to separate stations. Stations II, III, IV and V represent the stations at which heating treatments will be applied to compacts moving through heating zones contained therein. Upon completion of the heating treatment of this invention, the hot compacts will be rapidly delivered to a final forming or forging press at Station VI. Again, final forming can be included as part of an overall continuous process, and means would be provided for unloading hot compacts from the final heating Station V for a rapid transfer into the forming press at Station VI. A continuous process of the type depicted in FIG. 1 can be carried out in a time period of about 6 to 10 minutes (for relatively small gear parts) from the time of initial compacting until a finished form is produced at Station VI.

The heating and sintering Stations II–V provide for treatments which are necessary to carry out the reactions and physical changes in a powder metal compact before it can be formed into a high strength finished product. The Stations III–V are arranged to maintain an inert (or reducing) atmosphere around each compact as the compact advances from station-to-station.

Induction heating is used at Station III to add substantial heat energy to a powder compact in a relatively short period of time so as to bring the material of the compact to a sintering temperature at or above 980°C (and preferably to about 1285°C). However, it has been observed that ferrous metal compacts exhibit a characteristic incubation period at the beginning of a heating treatment in a low frequency induction field, and this period is of sufficient duration to result in a costly expenditure of energy without attainment of the desired rapid change in the temperature of the compact. In accordance with the present invention, the incubation period is substantially reduced or eliminated by subjecting each compact to a pretreating step in a relatively high frequency induction field.

Pretreating is carried out at Station II with known radio frequency generating equipment which operates, for example, at a frequency in the range of 50 to 450 kHz. It is preferred that a frequency at the lower end of the range, for example at about 50 kHz, be used in certain applications. An inert atmosphere may be provided at this Station but in certain applications may not be necessary.

FIG. 2 illustrates a typical relationship between a compact **10** and a coil **12** for carrying out high frequency treatment of the compact at Station II. The effect of pretreating in a radio frequency magnetic field appears to one of substantially reducing electrical resistivity of at least the skin of the powder metal compact in a very short time duration. For example, pretreating may take only 1 or 2 seconds for compacts typically used in the formation of bevel gears. In the art of induction heating, it is known that the reference depth d of induced current in powder metal compact should be one-fourth or less than the maximum thickness of the compact load for reasonable efficiency of the system. A typical preferred reference depth is illustrated between the arrows shown in FIG. 3, and this depth is less than one-fourth of the wall thickness of the compact shape which is shown. It is also known that there is a direct relationship between depth of induced current and resistivity of the compact in accordance with the formula:

$$d = K\,(r/mf)^{1/2}$$

d = reference depth of induced current
K = dimensional constant
r = electrical resistivity of the material
m = magnetic permeability of the material
f = frequency of heating current

It can be seen that once there is an adjustment in resistivity r of the material resulting from the use of a relatively high frequency f magnetic field, it is necessary to shift to a lower frequency f magnetic field to maintain a desired reference depth d of induced current in the part. Thus, the relationship between the pretreating and heating steps of the process is one of determining desired depth of induced current in a given size of part

and then adjusting frequency to compensate for a change in electrical resistivity of the material of the compact.

FIGS. **4** and **5** illustrate specific configurations of powder metal compacts which were treated in accordance with the process of the present invention. Each of the illustrated compacts were formed from a commercially available, water atomized, ferrous metal powder marketed as "Hoeganeas 2000" powder and having the following composition:

Carbon	- 0.018%
Manganese	- 0.350%
Phosphorus	- 0.007%
Sulfur	- trace
Silicon	- 0.013%
Oxygen (O_2)	- 0.120%
Nickel	- 0.500%
Molybdenum	- 0.48%
Iron	- balance to make 100%

The compacts were prepared isostatically without addition of a lubricant and their densities were approximately 75% of the theoretical density for the material used.

EXAMPLE 1:

The compact configuration illustrated in FIGS. **4** and **5** included a minor diameter hub portion **14** formed on a common axis with a major diameter body portion **16.** The FIG. **4** configuration of such a "hubbed" preform included the following parameters:

Overall Height	A	=	1⅞ inches
Height of Hub	B	=	1 inch
Major Diameter	C	=	2½ inches
Minor Diameter	D	=	1½ inches
Bore Diameter	E	=	1 inch
Weight		=	500 grams

This preform was heated in a nominal 450 kHz system for approximately **5** seconds. Actual machine operating frequency was unknown but could vary from 250 to 600 kHz during the heating cycle. Power consumed was about 5 kilowatts. The coil **12** included four turns, and clearances between the coil and outside surfaces of the preform (for both major and minor diameter portions) were in the range of ¼ to 3/**16** inch. After heating, the treated compact was heated to a sintering temperature in a stack of such compacts which were moved through a coil **12** having a single cross-sectional diameter throughout (as shown in FIG. **6**), and the compact exhibited very efficient (essentially instantaneous) coupling with a 3 kHz magnetic field induced by the coil. The illustrated "hubbed" preform can be used to produce a hubbed bevel gear (such as an automotive differential side gear) with a final forming step after sintering.

EXAMPLE 2:

FIG. **5** illustrates a clutch ring type of preform which included the following parameters:

Overall Height	A	=	1¼ inches
Overall Diameter	C	=	4¾ inches
Bore Diameter	E	=	3½ inches
Weight		=	900 grams

This preform was heated in a nominal 450 kHz coil (four turns of tubing having a square cross section of ¼ inch) for about 16 seconds. A clearance of about ¼ inch was provided between the preform and the coil during heating at high frequency. Power consumption was about 12 kilowatts peak. The treated preform was subsequently sintered in a low frequency (3 kHz) magnetic field, and the treated preform exhibited very rapid coupling and heating up to sintering temperature level.

In both of the examples above, the time cycle for treatment was determined by a change in plate current level of the power supply rather than by using a constant time for each size and shape of compact. For example, when the samples exhibited an initial plate current of 0.2 to 0.3 amperes, treatment was continued until the plate current reading was 1 ampere. The samples were then maintained at the 1 ampere level for approximately 1 to 2 seconds before removal from the field. Other electrical parameters in the heating circuit may be used to detect a change in the efficiency of coupling between the field and the sample.

It has been discovered that use of a 450 kHz field can present certain limitations in the treating of relatively large parts where depth of penetration of the field may be very shallow. This results in poor pre-treatment of such large parts and a slower response time of the parts to a lower frequency field. Since longer exposure to the 450 kHz field can result in rupturing of the sample (from thermal hoop stresses developing in the outer surface of the sample), techniques were studied to improve the treatment of larger parts. It was determined that the 450 kHz field could be used if the field were either (a) "pulsed" by turning the field on and off several times, (see U.S. Pat. No. 3,331,686 wherein pulsing during high frequency sintering is suggested) to allow surface heat to transfer deeper into the sample without developing large thermal hoop stresses, or (b) operated at a lower power level for a longer period of time, as was done in Example 2 above. These techniques required longer cycle times or more complex control of equipment.

Another technique involves the use of a lower radio frequency to increase the depth of penetration of the field without developing large thermal stresses. The disadvantage of this technique is that it can require more power than is required with higher frequency levels, however, the increase in power usage may be acceptable with certain large parts. This technique was tested with the following example.

EXAMPLE 3:

A clutch ring preform of the type and dimensions used in Example 2 above was treated in a plate coil **18** of the type illustrated in FIG. **5.** The plate coil was one and one-half inches in height and the clearance between the preform and the plate was about 3/**16** inch. The preform was treated with equipment having a nominal rating of 50 kHz for 6 to 7 seconds. Power consumption was 25 kilowatts. Preforms treated in this manner coupled almost immediately into a 3 kHz field. Thus, the use of a 50 kHz frequency (or a frequency at the lower end of the radio frequency range) may be preferred for larger parts where a shorter cycle time is desired.

The high frequency treatment of this invention produces an irreversible change in heating characteristics of powder metal compacts, and the compacts can be stored after such treatment if desired.

Although the invention has been described above with reference to a specific ferrous metal powder and specific treatment steps applied thereto, it should be appreciated that the principles of this invention can be utilized in improving characteristics of other ferrous metal compositions which are subjected to induction heating processes. Of course, composition, or other characteristics of a particular powder metal selected for use will change some of the parameters discussed above.

What is claimed is:

1. A process for heating and sintering ferrous powder metal compacts, comprising the steps of

pretreating a powder metal compact in a magnetic field having a relatively high frequency selected from a radio frequency magnetic field at about 50 kHz or above to induce current in the compact to a depth distance of about one-fourth of the maximum thickness of the compact,

continuing said pretreating for a sufficient time to effect a substantial decrease in electrical resistivity of said compact without significant sintering of the compact, and thereafter

subjecting the compact to a magnetic field having a relatively low frequency of about 10 kHz or less which provides for an efficient heating of said compact to a sintering temperature level in accordance with the changed electrical resistivity of the compact.

2. The process of claim 1 wherein said pretreating step is carried out for a time duration of less than 20 seconds for a compact which weighs about 900 grams.

3. The process of claim 1 wherein said pretreating step comprises an application of a radio frequency magnetic field to only surface areas of said compact.

4. The process of claim 3 wherein said application of a radio frequency magnetic field to surface areas of the compact is carried out with a sufficient energy input to result in a low temperature preheating of sections of the compact adjacent to said surface areas.

5. The process of claim 4 wherein said sections adjacent to said surface areas are preheated to a temperature of at least 150°C but less than the sintering temperature for the material of said compact.

6. The process of claim 1 wherein said pretreating step comprises an application of a radio frequency magnetic field to selected areas of a compact so as to provide for a differential response time for separate areas of the compact when the compact is placed in a lower frequency induction field.

7. The process of claim 1 wherein the duration of said pretreating step is determined by a change in an electrical parameter of the radio frequency heating circuit.

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