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POWDER METALLURGY

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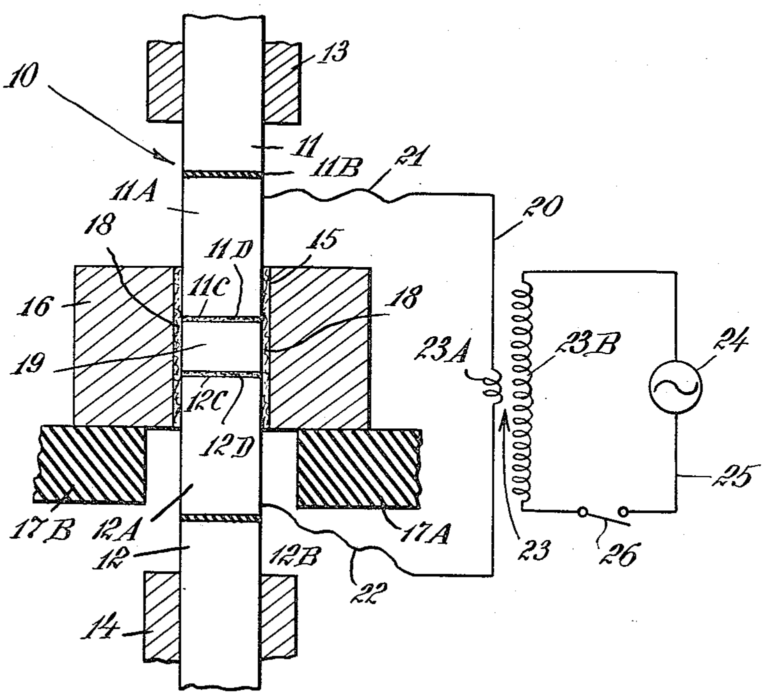

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POWDER METALLURGY

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This invention is concerned with powder metallurgy and particularly with the sintering of metal powder compacts. The invention provides improvements in methods of and apparatus for sintering such compacts to the end that sintering time is substantially reduced, with resultant increases in capacity of equipment and decreases in operating cost.

In the practice of powder metallurgy, it is customary to compress metal powders in a mold to form a coherent mass or compact and to sinter the compact to form a metallic bond between the particles. The heat treatment, as practiced heretofore, has required considerable time, and has been a matter of hours and, in some instances, of days. This makes for high cost of operation and, in some instances, renders the process non-competitive. Moreover, inasmuch as the compact usually is sintered in an unconfined state after withdrawal from the forming die, changes in dimension and distortion are likely to occur to such an extent that required tolerances for the finished piece are not met.

As a result of my investigations, I have developed an improved sintering technique whereby metal powder compacts are sintered adequately and substantially instantaneously, while they are confined in a die, thus minimizing opportunity for distortion and saving much time. Moreover, my invention permits objects of high density (i. e., little or no porosity) to be formed by powder metallurgy with lower pressures than those employed customarily heretofore. In accordance with my invention, the bonding together of the particles of the metal powder compact is accomplished by heating zones or points of contact between particles in the compact to a temperature in the neighborhood of, but preferably not exceeding, the melting point by passing through the compact an electric current of such amperage that the contact points attain this temperature substantially instantaneously, the heat treatment being conducted while the compact is compressed between electrodes carrying the current and while the compact is confined between the electrodes by insulated walls.

In the practice of my invention, the points or areas within the compact at which the metal particles are in contact with each other may be brought instantaneously to a temperature at which bonding occurs, while the bulk of the metal in the compact remains in a solid and relatively cold state. In consequence, the entire mass of the compact is not highly heated, although it becomes sufficiently plastic to permit the elimination of porosity in the compact with the exertion of only moderate pressure. Thus, the amount of heat employed in bonding together compacts in accordance with my invention is so small that the temperature of the entire compact does not rise greatly, so that it can be handled with the bare hands almost immediately following heat treatment and ejection from the die.

As indicated above, it is necessary to compress the compact between the electrodes during the heat treatment and to confine it between the electrodes by insulated wall surfaces. The electrodes should be of a conductive material, preferably a metal that is adequately strong and which has a melting point substantially higher than that of the compact. Moreover, it is desirable to employ electrodes the metal of which does not tend to alloy substantially with the metal of the compact. Otherwise, the electrodes tend to be welded to the compact during heat treatment.

The tendency of the electrodes to become welded to the compact can be reduced substantially by coating the surfaces of the electrodes that are to be placed in contact with the compact with finely-divided conductive material of high melting point and preferably of flaky character, for example graphite.

Ordinarily the electrodes will take the form of a pair of plungers in a die within which the compact to be heat treated is confined. The inside surface of the die walls which confine the compact must be insulated to avoid leakage of current. It is possible to make the die walls of an insulating or non-conductive material such as Bakelite or glass, since the die walls need not be heated highly. However, I have discovered that it is desirable to employ metal dies and to line these with a thin insulating coating.

The die walls may be lined with an insulating material such as paper. It is preferable, however, to coat the die walls with a flake material which has a high insulating or dielectric value, at least in a direction transverse to the die walls. For example, the interior of the die may be coated with flakes of mica or other micaceous material, that lie flat against the die wall and are at least in part overlapping.

The die lining may also be formed of metallic flakes. In fact, a metallic flake material, especially if it is relatively malleable and coated with a lubricant or leafing agent of insulating character, affords numerous advantages. Thus, the side of the mold or die, and in fact all interior surfaces of the die except the electrode surfaces in contact with the compact, may be lined with fine metal flakes which lie flat against the mold surfaces and are at least in part overlapping. The resulting thin coating of overlapping flakes of metal (which should have reasonably high malleability) is not broken when the compact is compressed in the die, so that actual contact between the die walls of steel or the like and the powder of the compact is substantially prevented and ejection of the compact from the die is facilitated. Moreover, the flakes on the die wall, especially if they are coated with a non-conductive lubricant or leafing agent such as stearic acid, are substantially non-conductive in a direction transverse to the die wall. Current introduced into the compact by the electrodes is thus confined within the compact.

Although I prefer to employ malleable metallic flake material as a die lining (for example aluminum flake mixed with stearic acid), flakes of non-metallic insulating substances, such as for example mica, may be employed. Such flakes do not aid in ejection of the piece to the same degree that the metal flakes do, but they serve as better insulation. The micaceous material preferably is mixed with a non-conductive binder having lubricating properties, for example a long chain fatty acid.

The use of the insulating flake die lining is not limited to the instantaneous sintering process described above, for it may be employed in any operation in which current is passed through a mass of metal powders confined in a die from which the mass should be insulated in order to avoid current leakage. Thus, the lining may be used when metal powder compacts are sintered by passing through them currents of relatively low density for substantial periods of time while the compact is confined by walls. Naturally enough, when currents of relatively high voltage are employed, the dielectric character of the flake lining in a direction transverse to the die wall must be better than when currents of low voltage are employed. The requisite increased insulation may be obtained by using flakes of high dielectric properties, such as mica or by increasing the thickness of the lining or both.

As disclosed and claimed in my co-pending application Serial No. 422,426, filed December 10, 1941, the use of die linings composed of oriented overlapping flakes of malleable material, for example aluminum flake, greatly facilitates ejection of compacts from a die in which it has been compressed, and reduces the force required for ejection to but a small fraction of that required in bare dies or those lubricated in accordance with customary powder metallurgical procedure. At the same time, such linings effectively prevent welding of the compact to the die wall (which tends to occur with many metals, particularly aluminum, even when cold powders are compressed) and also tend to reduce the occurrence of laminations and planes of weakness in metal powder compacts during compression and ejection.

Although either direct or alternating current may be employed in the practice of my invention, provided that the current density is adequate, I prefer to employ alternating current of commercial frequency, say 60 cycles. With alternating current of such frequency, the current is applied for a few cycles only. Ordinarily, assuming that the amperage is sufficiently high, adequate bonding together of the particles may be accomplished within the time of one or two cycles of current.

The current density to employ will depend, at least to some extent, upon the nature of the metal of the compact, the degree of porosity in the compact and the thickness of the compact, i. e., the depth of the compact in the direction of current passage. Ordinarily, compacts of most non-ferrous metals are amenable to substantially the same treatment and, other conditions remaining the same, may be sintered at substantially the same current density. Thus, non-ferrous compacts about ¼" in diameter and ⅛" to ⅜" thick may be adequately sintered in about 1/60 to about 1/30 of a second, i. e., with one or two cycles of 60 cycle current, the amperage of the current being about 20,000 and the compact being pressed between the electrodes with a force of the order of 5 to 10 tons per square inch. In such case, the current density is approximately 400,000 amperes per square inch of compact cross-section per ⅛" to ⅜" of compact thickness.

An important feature of my invention resides in the fact that the metal particles of which the compact is composed may be bonded together adequately without bringing about substantial diffusion in the interior of the powder particles. Generally speaking, the shorter the time of treatment the less diffusion occurs. Thus when it is desired to bond together particles of dissimilar metals which alloy readily with each other, for example tin and copper, it is possible to bring about the formation of a strong compact with very little intersolution of the tin and the copper, provided that the sintering time is adequately short. On the other hand, by repeated application of the current for short intervals, the diffusion of the metals can be increased and controlled. In the case of a compact composed of tin and copper powder, the application of current of high amperage for about 1/60 of a second brings about adequate bonding without diffusion. If the compact thus sintered is kept in the die and the current is again applied for one or more short intervals, say 1/60 of a second, diffusion occurs with resultant formation of tin-copper alloys. By repeated applications of such shots of current, it is possible to convert the entire compact to bronze.

If the current is applied for a sufficiently long interval which, in general, will not exceed half a second, the metal powder in the die may melt entirely. This is not desirable, for the molten metal tends to be forced out of the die and may also become welded to the electrodes. Consequently, if diffusion is desired it is preferable to treat the compact with a series of quick shots of current with intervals of a second or more between shots. In this way, any melting which occurs does not cause difficulties.

The ability of the process to promote adequate bonding without much alloying between readily alloyable metals present as powder particles in a compact is desirable because it permits the manufacture of improved metal objects with new properties. For example, bearings high in finely-distributed free tin and free copper are superior in bearing properties to bronze bearings containing the same ratio of copper to tin, provided that the "free-metal" bearings are adequately strong. The requisite strength is obtainable through the practice of my invention.

In the case of brass, the process of my invention permits the development of a peculiar metallographic structure in that a brass compact when treated in accordance with my invention develops a dendritic structure with the dendrites oriented in the direction of current passage.

The apparatus of my invention, as indicated above, comprises a die with a cavity having an insulated interior wall surface (which may be provided by an insulating lining or by making the die of non-conductive material) and adapted to confine a metal powder compact to be sintered, a plurality of electrodes which serve to close the openings in the die cavity, at least one of the electrodes being slidable in the die cavity, means for compressing the electrodes toward each other to compress the compact in the die, and means for passing a current of high amperage from one electrode to another through the compact while the compact is under pressure exerted by the electrodes.

In my preferred form of apparatus two opposed electrodes are slidable with respect to the die to facilitate ejection of the sintered compact.

The apparatus of my invention can be constructed from a spot welding machine of the press type, for example a Thompson-Gibbs machine of the K–18–40 type or that of the Eisler Engineering Company—Press type #525. Such machines, when equipped with suitable electrodes which act as punches in a die made of non-conductive material or in which the cavity is lined with insulating material, are admirably suited for instantaneous sintering of relatively small metal powder compacts in accordance with my invention. Naturally enough, the bigger the compact to be sintered, the bigger is the required machine, since the greater must be the electrical input. Satisfactory results have been obtained with small compacts having a cross-section of less than one square inch transverse to the direction of current passage, with machines having ratings of 40 to 125 k. v. a.

My invention will be understood more thoroughly in the light of the following detailed description of practices thereof, taken in conjunction with the accompanying single figure which is a schematic illustration, partly in section, of apparatus adapted for the practice of the invention.

Referring to the drawing, it will be observed that the apparatus comprises a press 10 equipped with an upper piston 11 and a lower piston 12 that are slidable, respectively, in an upper cylinder 13 and a lower cylinder 14. The pistons are adapted to be forced toward each other or in the opposite direction by hydraulic pressure or the like exerted in conventional chambers (not shown) in the cylinders.

A pair of plunger electrodes 11A, 12A are mounted respectively on the outside ends of the upper and lower pistons and insulated therefrom by insulating layers 11B, 12B. The electrodes are concentric and slide in the bore 15 of a die 16 that is disposed between the two cylinders and is supported on insulators 17A, 17B.

The bore of the die and the cross-section of the electrodes may be of any desired shape. The bore of the die is lined with a layer 18 of oriented overlapping metal flakes coated with a non-conductive lubricant or leafing agent, such as stearic acid, which also serves as a binder to hold the flakes flat against the die wall and thus provide the insulating film.

The ends 11C, 12C of the electrodes which enter the die are coated with layers 11D, 12D of finely-divided and preferably flaky material of high electrical conductivity and high melting point, for example flakes of graphite.

The two plunger electrodes and the die enclose a compact 19 of compressed metal powder to be heat treated. The compact and the plungers are insulated from the bore of the die by the insulating layer of metal flakes, but the compact and the plungers are in good electrical contact through the conductive layers 11D, 12D.

The electrodes are connected in series in a secondary circuit 20 by means of flexible connections 21, 22, which may be ribbons of woven copper wire, to a secondary coil 23A of a step-down transformer 23. The primary coil 23B of this transformer is disposed in inductive relationship with the secondary and is connected in series in a circuit 25 with a conventional alternating current source 24, such as a 60 cycle 110 volt lighting circuit, through an automatic switch 26 of the type employed in spot welding machines and the like for making the circuit and breaking it automatically after a predetermined number of cycles.

The apparatus illustrated may be made by converting a spot-welding machine of the press type by equipping it with suitable plunger electrodes, preferably of tungsten, and a suitable insulated die which may be of steel. Spot welding machines that are suited to such adaptation include the 40 k. v. a. machine made by Thompson-Gibbs and identified as type K–18–40 (equipped with G. E. thyratron control and employing a primary 60 cycle 220 volt alternating current of 180 amperes) and the 125 k. v. a. machine of the Eisler Engineering Company, identified as Press type #525 and equipped with 60 cycle Neotrol control.

As indicated above, suitable electrodes may be made of a variety of materials and should have high electrical conductivity and mechanical strength coupled with an ability to withstand alloying with the metal of the compact. A composition consisting predominantly of tungsten and sold by P. R. Mallory Company under the name Elkonite 100W is suitable, especially for non-ferrous compacts.

The compact to be heat-treated can be formed in situ from loose powder in the bore of the sintering die of the apparatus illustrated or it may be pre-formed by compression in a forming die. In any event, the compact is pressed between the electrodes while confined in the bore of the sintering die and, while the pressure is exerted, current of high amperage, say of the order of a million or more amperes per square inch of cross-section, is passed through the compact for a short time, say $\frac{1}{60}$ to $\frac{1}{30}$ of a second. Under these conditions, the compact becomes plastic, especially at the points of contact between particles and can be made substantially non-porous if relatively low pressure, of the order of 7 tons per square inch is exerted, so as to compact the powder still further and fill up the voids between particles.

As indicated above, the die may be of a non-conductive material, such as Bakelite or other plastic of relatively high dielectric strength. However, because of its greater mechanical strength, I prefer to employ a metal die, even though this requires the use of an insulating lining. The current involved in the practice of my invention is high, but the voltage of this current is relatively low, say of the order of 5 to 20 volts. Consequently, a very thin insulating film is satisfactory. A paper lining may be employed but experience has shown that such a lining may be charred in the sintering and may have to be replaced each time a fresh compact is sintered.

The insulating lining of flake material, for example finely-divided mica or metal flake, is preferred. Although in some instances overlapping metal flakes without a leafing agent may be employed, I prefer to use metal flakes that have been coated with a non-conductive binder, such for example as stearic acid, because such agent increases the dielectric value of the lining and also holds the lining to the die wall.

A great variety of metallic flake material may be used. Thus, flakes of various malleable metals, such as copper, gold, zinc, lead and aluminum may be employed. Ordinary aluminum flake material, such as that employed in paints, is satisfactory. Such material contains a small proportion (of the order of 3%) of a leafing agent such as stearic acid and is usable in the practice of my invention without addition of more leafing agent. However, I prefer to add to such flake a substantial proportion of finely-divided stearic acid or the like employing about 1 volume of stearic acid powder to 2 volumes of aluminum flake. The powdered stearic acid and the aluminum flake, the individual particles of which have already been coated with stearic acid, should be suspended in a suitable vehicle. A vehicle in which the leafing agent is substantially insoluble, say benzol in the case of stearic acid, is preferable. However, other volatile vehicles, such for example as acetone in the case of stearic acid, may be employed even though the leafing agent is soluble therein.

The flake lining of the die may be applied in any convenient manner, for example by an automatic wiper or atomizer in the case of commercial operation or with a brush in laboratory work. A single coat is all that is required, although care should be taken to see that all parts of the bore are adequately coated. The vehicle should be permitted to evaporate before the loose powder or the pre-formed compact is introduced into the bore of the die. It may be unnecessary to re-line the die each time, for the lining is relatively durable and may survive during treatment of several compacts. However, it is desirable to reline the die each time.

With many types of metal powder, it is unnecessary to coat the ends of the electrode plungers with a finely-divided conductive material. However, when there is any tendency for the compact to become welded to the end of the electrode, it is desirable to paint the ends of the electrodes with graphite flake or the like.

To consider my invention in greater detail, a solid copper object was formed in accordance with the following procedure:

A spot welding machine of the press type with a rated capacity of 125 k. v. a. was equipped with a cylindrical die having a bore about ¼'' in diameter and adapted to form the apparatus illustrated in the drawing. The die was insulated from the rest of the apparatus, including the two cylindrical plunger electrodes which slid therein. Electrolytic copper powder was pre-compacted in the cold state in a die at a pressure of 5 to 10 tons per square inch to form a cylindrical slug about ¼'' thick and of a diameter only slightly less than that of the bore of the die. The die was lined by painting it with a suspension of aluminum flake and finely-divided stearic acid in benzol so as to form a continuous lining of oriented flat-lying overlapping flakes over the entire bore surface.

The precompacted copper slug was placed in the lined die and the electrodes, coated at their ends with graphite flake, were pressed against the compact in the die with a force of about 7½ tons per square inch. Alternating current of 60 cycles and about 20,000 amperes was shot through the compact for an instant, i. e., for 1 to 4 cycles of current time.

The current thus applied brought about a thorough welding together of the particles of copper to form a dense metallic slug. Microscopic examination of a cross-section of the slug indicated that the metal at the surfaces and points of contact between the particles in the compact had been heated highly and perhaps in some instances melted. However, it appeared that the bulk of the metal had not been highly heated, for there was little evidence of diffusion or change in crystalline character in the interior of the particles.

The foregoing test was repeated employing dies of various materials including glass, Bakelite and Transite. The test was also conducted with metal dies which had a paper lining. In general, the results obtained with the lined metal dies were superior to those obtained with the non-metallic unlined dies, and the metal flake lining was superior to the paper lining.

Electrodes of various compositions, including copper, were used. It was found that tungsten electrodes were preferable in that they did not tend to become welded to the compacts. Thus, plungers made by turning down welding electrodes composed principally of tungsten and identified as Elkonite 100W-K475 were found to be satisfactory, especially if the ends in contact with the compact are coated with graphite.

With compacts of the size indicated, i. e., about ¼ of an inch in diameter and 1/16 to ¼ of an inch thick, adequate bonding was attained with current of a density of about 400,000 amperes per square inch applied for one cycle or 1/60 of a second. The pressure exterted on the compact during the passage of the current therethrough being about 7½ tons per square inch. It was found that these conditions of operation were satisfactory for compacts made of bronze powder (specifically a 90 copper, 10 tin bronze), brass powder (specifically 70—30 brass) and aluminum, in addition to copper.

Thus a number of brass, bronze and aluminum compacts were treated in the apparatus described above under the conditions found suitable for the treatment of copper compacts. In substantially all instances the results were satisfactory and dense compacts were formed.

In the case of the brass compact, it was discovered that the compact developed an oriented dendritic structure when it was subjected to a current of 5 to 20 volts and of a density of about 400,000 amperes per square inch for 1 cycle of current, i. e., 1/60 of a second. Microscopic examination of sections of the specimen showed the dendrites extending through the compact as more or less parallel bundles in the direction of current passage.

When a brass slug having this oriented dendritic structure was subjected to a second treatment under the conditions outlined above for one cycle of current, i. e., a total of two cycles, the dendritic structure tended to disappear.

The compacts of bronze powder were made of a bronze powder in which the copper and tin were not completely diffused. A single shot of current for 1 cycle at a voltage of 5 to 20 and a current density of about 400,000 amperes per square foot brought about adequate bonding between the particles but did not cause further substantial alloying of the free copper and tin, indicating that the time of treatment was so short that no substantial diffusion of the metallic constituents was permitted.

The aluminum powder compacts were adequately welded into dense compacts under the conditions found to be suitable for copper, brass and bronze.

I claim:

1. The improvement in the sintering of a metal powder compact to bond together the particles thereof which comprises heating points of contact between particles in the compact to a temperature in the neighborhood of the melting point by passing through the compact an electric current of such amperage that the temperature at the points of contact is attained substantially instantaneously while the balance of the particles remains in a relatively cold condition, the compact being heated by the current while it is compressed between electrodes carrying the current and confined between the electrodes by a die wall coated with flat-lying overlapping metal flakes having thereon a leafing agent of insulating character.

2. The improvement in the sintering of a metal powder compact to bond together the particles thereof which comprises heating points of contact between particles in the compact to a temperature in the neighborhood of the melting point of the metal of the compact by passing through the compact an electric current of such amperage that the points of contact with such temperature substantially instantaneously while the interior of the particles remains in a relatively cold condition, the compact being heated by the current while it is compressed between electrodes carrying the current and confined between the electrodes by die walls coated with metal flake lying flat against the die walls, the flakes being covered with a relatively non-conductive lubricant.

3. The improvement in the sintering of a metal powder compact to bond together the particles thereof which comprises heating the compact to a temperature at which the powders tend to weld together by passing through the compact an electric current while the compact is confined by walls coated with a thin film of flat-lying metal flake coated with a lubricant of insulating character.

4. An apparatus for sintering metal powder compacts which comprises a die of conductive material with a cavity having interior wall surfaces covered with an insulating layer composed of metallic flakes lying flat against the wall surfaces and adapted to confine the metal powder compact to be sintered, a plurality of electrodes which serve to close the openings in the die cavity, at least one of the electrodes being slideable in the die cavity, means for compressing the electrodes toward each other to compress the compact in the die, and means for passing a current of high amperage from one electrode to another through a compact while the latter is under pressure exerted by the electrodes.

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