SINTERING OF SHAPED CEMENTED METAL BODIES
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This invention relates to the production of shaped cemented metal particle bodies containing ingredients which at sintering temperature, form a liquid phase.

In the past, difficulties have been encountered in producing shaped cemented refractory metal particle bodies having ingredients of relatively low melting temperature, because of the deformation of the shape of such bodies which occurred in the course of the final sintering treatment to which they have to be subjected in order to give them the desired physical characteristics and strength.

Thus, by way of example, in the case of a small gas turbine rotor formed of titanium carbide bound by an alloy of nickel, chromium and cobalt forming more than about 30% of the rotor, it was found that its shape becomes deformed when it is subjected to the final sintering treatment at the required high temperature, such as 1300° C. to 1350° C. (Throughout the specification and claims, all proportions are given by weight unless specifically stated otherwise.)

Among the objects of the invention is a sintering treatment for sintering cemented metal particle bodies containing ingredients which form a liquid phase at the sintering temperature, which treatment will suppress and eliminate the deformation of the desired shape of the body caused by the final sintering treatments as carried on in the past.

The foregoing and other objects of the invention will be best understood from the following description of exemplifications thereof, reference being had to the accompanying drawing, the single figure of which shows, by way of example, in a partially diagrammatic and partially cross-sectional view, one type of equipment for sintering a cemented refractory metal particle body in accordance with the principles of the invention.

The present invention is based on the discovery that the deformation difficulties encountered in the final sintering of cemented refractory metal particle bodies are caused by the differences in the gravitational forces acting on the different parts of the body as a result of partial softening of the lower melting phase thereof when heated during the final sintering treatment. In accordance with the invention, the deformation difficulties encountered in the final sintering treatment of such cemented refractory metal bodies, are eliminated and suppressed by rotating the body during the sintering operation so that the gravitational forces acting on different portions of the cemented body balance each other. For best results, the cemented body should be rotated about one of its axes of inertia, which are also the axes of symmetry of the body. Thus, in the case of a cemented body which is symmetric with respect to an axis of rotation thereof, the body should be rotated around such axis of rotation during the sintering treatment. In the case of bodies which have in one direction materially smaller dimensions than in other directions, such as disc-like bodies, it is desirable to rotate the body during the sintering operation about a horizontally disposed inertia axis thereof corresponding to its largest moment of inertia.

In the drawing is shown by way of example, one type of equipment which has been found satisfactory for carrying out the process of the invention when producing a gas turbine rotor out of cemented refractory metal particle material. In the form shown, the gas turbine rotor generally designated 10 comprises a generally circular disc-like rotor body 11 which is symmetric about its axis of rotation 12 and has a cylindrical seating opening 13 coaxial with its axis of rotation 12. The rotor body 11 is provided along its circular periphery with an array of fluid-guiding buckets 14 symmetrically arranged with respect to the axis of rotation 12. Such gas turbine rotors, which have to operate in oxidizing combustion gas atmospheres at elevated temperatures, are produced out of cemented refractory particle material which exhibits the required strength and corrosion resistance at the elevated temperatures at which it is exposed to the combustion gas atmosphere. The process of the invention may be used to produce gas turbine rotors of the entire range of sizes in which they are required. As an example, the process of the invention may be used for making gas turbine rotors as small as about 5 centimeters or even less in diameter, as well as larger sizes such as up to 30 centimeters in diameter.

As an example, good results are obtained with a gas turbine rotor made by the process of the invention out of titanium carbide particles cemented by a binder alloy metal of high corrosion resistance but having a lower melting temperature than titanium carbide. As a more specific example, good results are obtained with such gas turbine rotor having 30% to 70% titanium carbide content, the balance binder metal of high corrosion resistance and good binder properties. Good results are obtained with the binder metal formed of nickel, cobalt and chrom- 

The process of the invention will now be described in more detail in connection with a specific example thereof. Thus, a cemented gas turbine rotor is produced by the process of the invention as follows:

Example 1

There is prepared a mixture of powder particles of titanium carbide with powder particles of desired proportion of the binder metal ingredients, to wit: nickel, chromium and cobalt. The powder mixture is then compacted in a die having a die cavity of a shape corresponding to the rotor body 10 shown in the drawing, with a solid peripheral annulus extension corresponding to the row of buckets 14. Good results are obtained with a compacting pressure of about 800 kg./cm.2. After removing from the die, the green compact is pre-sintered
in vacuum or in a protective atmosphere at a temperature between about 800° C. and 900° C. for three to four hours, depending on size, so as to yield a pre-sintered body of substantial strength but sufficient softness to permit forming conventional tools to the desired final shape. The pre-sintering temperature is so chosen as to prevent softening of the shaped body while securing substantially full shrinkage thereof, and minimize any further shrinkage thereof in the final sintering treatment. In the design of the dies for the green compact, allowance is made for the shrinkage of the compact in the pre-sintering treatments and for the shaping of the presintered body. The machined and finished pre-sintered rotor body so obtained is thereafter subjected to the final sintering treatment at a selected temperature between about 1300° C. to 1350° C. for 1 to 1½ hours depending on the size. By way of example, a gas turbine rotor of high desired thermal shock resistance was made by the process described above of a powder mixture containing 35% titanium carbide and 65% binder metal consisting of 60% nickel, 20% chromium and 20% cobalt. The resulting rotor bodies had a transverse rupture strength of 180 to 200 kg./mm.² and a specific weight of 6.9 g./cm.³.

There will now be described in connection with the drawing, how a gas turbine rotor of the type described above was subjected to the final sintering operation at a temperature of about 1300° C. to 1350° C. for about one hour or more, without causing it to be deformed, in accordance with the principles of the invention. The desired shaped cemented bodies, such as turbine rotors 10, are subjected to the final sintering operation within the interior of a furnace which, in the form shown, comprises a double-wall casing enclosure 21 formed of two spaced walls 22, 23 of heat-resistant material, such as heat-resistant metal, forming a cooling space through which a cooling liquid, such as water, is circulated for cooling the furnace walls. The furnace enclosure 21 is shown provided with a detachable double-wall section 24, likewise formed of two spaced walls 22-1, 23-1 confining a cooling space through which cooling liquid is similarly circulated. The detachable enclosure wall section 24 is joined to the main enclosure 21 along the interfitting junction walls 25, 26, arranged so as to permit joining them with a hermetic seal which permits maintenance with the interior enclosure space 20 of the furnace either a vacuum or a desired protective atmosphere, such as hydrogen, during the sintering treatment. The turbine rotor 10 is shown held on a rotor shaft 31 arranged to be rotated on bearings 32. The rotor shaft is formed of refractory material. The left side of the rotor shaft 31 is shown arranged to be joined by detachable coupling 36 to a drive shaft 34 which is rotatebly driven by a suitable drive mechanism on the interior of the furnace enclosure 21. The rotor shaft 31 shown is connected by a similar coupling connector 33 to a similar rotor shaft of the adjacent of a series of rightwardly disposed additional turbine rotors which are to be sintered within the furnace enclosure 21 in accordance with the principles of the invention. It is to be understood that the part of the specification below, which deals with the turbine rotor 10 mounted adjacent to the left side wall of the furnace enclosure 21, apply also to each of the other similar turbine rotors which are subjected simultaneously to a similar final sintering operation within the furnace enclosure 21.

The drive shaft 34 by which the rotor shaft 31 of the turbine rotor is driven, is shown seated for rotation in a bearing 35 mounted in the left side wall of the furnace enclosure 21. The bearing 35 is of a type well known in the art that provides hermetic seal between the interior and the exterior of the furnace enclosure 21 while permitting the shaft 34 passing therethrough to rotate therein for driving through coupling connector 33, the rotor shaft 31 together with the turbine rotor 10 held thereon, and other similar turbine rotors which are being sintered within the furnace enclosure 21.

The region of each of the turbine rotors 10 positioned within the furnace enclosure 21 is to be heated by an array of heater bodies shown in the form of heater rods 41 of a material such as molybdenum or molybdenum disilicide, which are arranged so as to be supplied with electric heating current as through electric connector bars 44 extending from water cooled electric connector terminals 45 insulatingly mounted and passing through the wall portions of the furnace enclosure 21. In order to protect the interior wall surfaces of the furnace enclosure 21 against the heat radiated by the heater bodies 41, a plurality of heater screens, shown by dash lines 46 and formed of sheets of refractory metal, such as molybdenum, are interposed between the heater bodies 41 and the interior wall surfaces of the furnace enclosure 21. In order to maintain the sintered body 10 at a desired uniform and balanced treatment temperature, there is provided within the furnace enclosure 21, a further inner heat-balancing enclosure 51 enclosing and surrounding the space occupied by the rotor 10.

The inner enclosure 51 may be formed of refractory metal such as molybdenum sheet material and may have a detachable wall section 52 on the upper side of the adjacent region of the detachable double-walled enclosure 21. In the form shown, the bearings 21 on which the rotor shaft 31 is suitably mounted within the walls of the inner enclosure 51 surrounding the cemented rotor 10 which is to be sintered, may be formed of refractory or ceramic material of the type used in similar applications, which will retain its strength at the high temperatures at which the interior of the furnace is heated. The general details of the structural features of the sintering furnace of the type shown, except for the arrangement whereby the sintered body is to be rotated therein, are of well known construction generally used in the past for finally sintering cemented refractory metal particle bodies and need not be described in more detail. The rotor shaft 31 on which the cemented rotor 10 is seated, the drive shaft 34 and the shaft connector 33 are made of a solid material that will retain its strength at the elevated furnace temperature, such as tungsten or molybdenum.

The interior of the sintering furnace 21 is heated by heating current supplied to the heater bodies 41 for bringing and maintaining the rotor 10 at the desired sintering temperature. The sintering body 10 is heated in a furnace of about 1300° C. to 1350° C. The interior space within the furnace enclosure 21 is evacuated by a suitable vacuum connection, not shown, so as to maintain therein a vacuum of about 0.1 to 0.05 mm. of mercury column. Throughout the time the rotor 10 is held at a raised temperature at which the binder metal of the rotor might develop a liquid phase or might soften, the rotor 10 is rotated by applying a rotary driving force to the outer end of the drive shaft 34. The rotor 10 is kept rotating for the entire period of 1 to 2 hours required for completing the final sintering treatment wherein its body is given the desired physical characteristics. After performing the sintering treatment the heating is stopped and the interior of the furnace is cooled, the rotor 10 being kept rotating until its temperature has been brought down considerably below the temperature at which the binding metal thereof has any liquid phase or a tendency to soften, such as a temperature of 800° C. or lower.

In making gas turbine rotors by a process of the invention of the type described above, it has been found advantageous to incorporate small amounts of molybdenum in the mixture of the powder ingredients out of which the gas turbine rotor is formed. The addition of a small amount of molybdenum eliminates free carbon and oxygen which if present would detract from the physical properties of the formed gas turbine rotor. As stated above, the amount of the metal binder addition may vary
between 25% and 65% by weight. The sintering operations are carried out in what is known as a "technical" vacuum of the order of 0.1 mm. of mercury column. In general, the compaction pressure applied to form out of the powder mixture the gas turbine compact, ranges between 2 to 5 t.s.i. (tons per square inch). The compact is sintered at the pre-sintering temperature of 600° C. to 900° C. for a pre-sintering time of about 1 hour and up to 3 or 4 hours, depending on the size. The machined and finished pre-sintered rotor body is subjected to a final sintering temperature of 1200° C. to 1400° C. for 1 to 1½ hours. Such results have been obtained with the process of the invention carried on within the range of conditions outlined above, with a gas turbine rotor having an outside diameter of 8 inches and larger.

In the sintering operations, during which the gas turbine rotor is subjected to elevated temperatures at which its binder must become softened, the gas turbine rotor is rotated around an axis of symmetry thereof to prevent flow of the softened or liquefied binder metal due to the gravitational forces acting thereon. In practice, slow rotation of the gas turbine rotor such as at 1 r.p.m. (revolutions per minute) or a somewhat higher rate of such 5 r.p.m. is sufficient to prevent gravitational flow or creep of the heated softened binder metal. By such slow rotation, the direction of the gravitational forces acting on the softened binder metal is continuously changed at a rate at which they are insufficient to cause any movement or displacement of the softened binder metal of the rotor while it is heated during the sintering operations. By rotating the rotor in the manner described above to prevent flow or creep of binder metal while the rotor is subjected to the elevated temperature of the sintering furnace, uniform and homogeneous distribution of the binder and the danger of non-homogeneity avoided, a critical factor for rotors which operate at high speed. The rate at which the gas turbine rotor is rotated while it is maintained at an elevated temperature during the sintering operation, is not critical, and is related to and depends on the size of the cemented-particle rotor and the composition of the cemented material out of which it is formed. Thus, in the case of a gas turbine rotor of 35% TIC cemented by 65% of the binder metal, as described above, having an outside diameter of 20 centimeters, the rotor is maintained at a rate of about 4 to 5 r.p.m. while it is maintained at the elevated sintering temperature. On the other hand, in the case of a similar gas turbine rotor of the same material having an outside diameter of about 14 centimeters, good results are obtained by rotating it at a rate of 2 to 3 r.p.m. Rotors made with a smaller content of the binder metal, may be rotated at a smaller speed to prevent gravitational flow or creep of the binder metal which is somewhat softened at the elevated sintering temperature. In general, a relatively low speed of rotation, of at most 200 revolutions per minute, will be sufficient for preventing deformation of a cemented body as a result of softening of a liquid phase thereof during the sintering operation. The sintering medium of the invention is not limited to forming shaped cemented bodies of the type described above but is of great value generally in the production of a wide range of sintered cemented metal particle bodies containing ingredients which form a liquid phase at the high sintering temperature to which they are subjected, because the action of the gravitational forces on the liquid phase of such body during the sintering may cause distortion of the liquid phase, thereby disturbing the homogenous character of the sintered body, a factor which is of great practical importance in many applications of sintered cemented refractory bodies. Thus, for instance, lack of homogeneity in a rotary body will develop large unbalancing forces during high-speed rotation of such body and will require careful balance.
face of the steel rotor. After an initial presintering at about 800° C. to 1000° C. for 5 to 10 minutes, the pre-
sintered compact is subjected to a grooving operation for forming out of the cylindrical sintered compact a
plurality of spirally-shaped elements joined and supported
at the opposite ends by a continuous end ring collar.
After placing the so-obtained generally cylindrical body
on a cylindrical graphite rod on which its end collars
are seated, it is sintered in vacuum for 20 minutes at
1450° C. to 1500° C., while the cylindrical body held
on the graphite rod is rotated 5 turns per minute.
In such sintering treatment, the sintered body is softened
by the liquid phase formed of some of the ingredients.
Without rotation during the sintering treatment, the in-
dividual spiral tool elements would become deformed
under the gravitation forces acting thereon, and would
require a great deal of additional machining to remove the
deformation defects.

Example 5
For shield containers for radioactive substances formed
of tungsten bound with 5% nickel and 2% copper, it is
essential to secure absolutely uniform distribution and
homogeneity of the binder metal content throughout the
body of the container. A cylindrically shaped radiation
shield is prepared by subjecting the compact formed of
a mixture of several powder ingredients to sintering for
10 to 15 minutes at 1400° C. to 1450° C. under hydro-
gen while the shield body is being rotated at a rate of
five turns per minute. During the sintering operation,
the body is supported on a cylindrical ceramic support
which forms the rotating support thereof. Because of
the great difference between the specific weight of tung-
sten and its copper-nickel content, the gravitational forces
acting thereon during sintering, while it contains a liquid
phase, will cause non-uniform distribution of its nickel-
copper content, if it is not rotated during the sintering
process in accordance with the invention.

Example 6
Airfoil shaped turbine buckets or vanes are formed
out of titanium carbide cemented with 50% of a nickel-
chromium-cobalt alloy. The powder compact formed of
the titanium carbide and the alloy, is provided at its
opposite ends with rotary support projections which are
ground off therefrom after completing the sintering.
The rotary end portions of the compact are arranged coaxial
with its inertia axis. After preliminary sintering of the
compact at about 800° C. - 1000° C., it is subjected to
an additional shaping operation. Thereupon it is sub-
jected to additional sintering while it is being rotated
on its end projections under vacuum for 10 minutes at
a sintering temperature of 1250° - 1300° C., at which a
liquid phase is formed of some of its contents. Although
such bucket may only be 7 inches long, such sintering treat-
ment will cause slight deformation thereof as a result of
the gravitational forces acting thereon while it is
softened by the liquid phase formed therein during the
short sintering treatment. The removal of the defects
caused by the deformation involves expensive, careful
machining, which is eliminated by rotation thereof during
the sintering, in accordance with the invention.
Instead of providing a direct mechanical coupling con-
nection between the exterior drive shaft and the rotor
shaft 31, the mechanical driving connection may be
provided in some other way, for instance by a magnetic
coupling between a magnetic member of the external drive
shaft and a magnetic member in the interior of the fur-
nace enclosure 21 which has a mechanical connection to
the rotor shaft 31.

This application is a continuation-in-part of our co-
pending application Serial No. 381,598, filed September 22,
1953, now abandoned.
The features and principles underlying the invention
described above in connection with specific exemplifica-
tions, will suggest to those skilled in the art many other
modifications thereof. It is accordingly desired that the
 appended claims be construed broadly and that they
shall not be limited to the specific details shown and de-
scribed in connection with exemplifications thereof.

We claim:
1. The method of sintering a homogeneous cemented body consisting of cemented refractory particles selected
from the group consisting of the refractory metals, the
carbidies, the borides and the silicides of the refractory me-
tals and mixtures thereof, which body contains ingredi-
ents which form a liquid phase at the sintering tempera-
ture, the procedure comprising rotatably supporting said
body along an axis, subjecting said body while so sup-
ported to sintering for at least 10 minutes at a sintering
temperature of at least about 1100° C. at which a liquid
phase is formed of some of its ingredients tending to
soften said body, and slowly rotating said body about
said axis while it is being sintered at said sintering tem-
perature in such manner that the direction of the grav-
itational forces acting on said liquid phase and other por-
tions of said body is continuously changed at a rate at
which said gravitational forces are insufficient to cause
any movement of portions of the liquid phase and of any
other portions of said body relatively to each other be-
cause of softening of said liquid phase while being sub-
jectied to said sintering treatment.
2. The method of sintering a cemented body as
claimed in claim 1, wherein said body is rotated about
an axis having the greatest moment of inertia.
3. The method of sintering a homogeneous cemented
body which is symmetric with respect to a central axis
and consists of cemented refractory particles selected
from the group consisting of the refractory metals, the
carbidies, the borides and the silicides of the refractory me-
tals and mixtures thereof, which body contains ingredients
which form a liquid phase at the sintering temperature,
the procedure comprising rotatably supporting said body
along said axis, subjecting said body while so supported
to sintering for at least 10 minutes at a sintering tem-
perature of at least about 1100° C. at which a liquid
phase is formed of some of its ingredients tending to
soften said body, and slowly rotating said body about
said central axis while it is being sintered at said sinter-
ing temperature in such manner that the direction of the grav-
itational forces acting on said liquid phase and other por-
tions of said body is continuously changed at a rate at
which said gravitational forces are insufficient to cause
any movement of any portions of the liquid phase and of
any other portions of said body relatively to each other
because of softening of said liquid phase while being sub-
jectied to said sintering treatment.
4. The method of sintering a cemented body as
claimed in claim 1, wherein said body is rotated about
the temperature of said body is lowered from the elevated
temperature of sintering to a level at which all
binder metal content of said body is solidified.
5. The method of sintering a cemented body as claimed
in claim 3, wherein said body is rotated until the tempera-
ture of said body is lowered from the elevated tem-
perature of sintering to a level at which all binder metal
content of said body is solidified.

No references cited.
UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 2,894,836

July 14, 1959

Franz Kolbl et al.

It is hereby certified that an error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

In the heading to the printed specification, between lines 7 and 8, insert "Claims priority, application Austria October 1, 1952 --."

Signed and sealed this 8th day of December 1959.

(SEAL)

Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents
UNITED STATES PATENT OFFICE
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