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(54) METHOD OF SINTERING ELECTRICALLY CONDUCTING POWDERS

VERFAHREN ZUM SINTERN VON ELEKTRISCH LEITFÄHIGEN PULVERN

PROCÉDÉ DE FRITTAGE DE POUDRES ÉLECTRIQUEMENT CONDUCTRICES

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Description

TECHNICAL FIELD

[0001] The present invention belongs to the field of methods for obtaining sintered parts, which consists in the application of heat and pressure to powders for finally obtaining dense parts, in particular wherein the heat is obtained via electrical currents that are forced to pass through conductive powders.

STATE OF THE ART

[0002] ECAS (electric current assisted sintering) gathers a family of consolidation methods in which mechanical pressure is combined with electric and thermal fields to enhance interparticle bonding and densification. The starting materials can be in the form of either powders or green compacts. The primary purpose of imposed electric currents is to provide the required amount of resistive heat.

[0003] ECAS techniques can be classified with respect to the discharge time. Conventionally, 0.1 s discharge time can be assumed as the threshold between fast and ultrafast ECAS. However, confusion should be avoided between fast termed herein and the FAST acronym (field activated/assisted sintering technique) frequently encountered in the scientific literature. Here, fast simply refers to either a high processing rate or a low processing time.

[0004] On one hand, fast ECAS techniques are known with different acronyms mainly depending on the electric current waveform: SPS (Spark Plasma Sintering), PECS (Pulsed Electric Current Sintering), RS (Resistance Sintering), etc. The bases of the process were developed by INOUE (US3241956 A). The electric current is normally applied as pulses and also was reported the possibility of the application of various superimposed currents with different frequencies and two stages of pressure to increase densification. The application of the load was also described in US3508029 A. These two processes are characterized by the low intensity of the electric pulses (< 1 kA/cm^2) and high duration of the cycles (from seconds to minutes). In addition, the manufacturing of fast ECAS equipment is complex with the need of protective atmosphere (or vacuum).

[0005] On the other hand, ultrafast ECAS techniques typically employ either one or up to three repeated (capacitor) discharges. Each discharge lasts less than 0.1 s. The current pulse density can be on the order of 10 kA/cm^2. Ultrafast ECAS is generally referred to as electric discharge compaction (EDC) or EFS (Electro Forging Sintering). Representative examples of these methods were explained in the following patents: EP 2198993 A1 from Fais, US4929415 and US4975412 from Okazaki. Main problem of these methods is that the capacitors discharge the stored energy in a sudden an uncontrolled way and thus they did not permit tailoring of the power input to the powder mass. The use of high current and high voltage resulted in inconsistent densification and inhomogeneity of parts manufactured using these consolidation processes, because the resistance of the powders is not homogeneous due to porosity, surface oxidation, compaction or bonding between the particles, and it is well known that the current always follows the lowest resistive path. Other problems are the low size of the samples that can be produced and the sparks in the electrodes produced by the high current and high voltage.

[0006] Other ultrafast processes used low voltage equipment like the patents developed by Cremer (US2355954), Knoess (US5529746) and Bauer (US7361301). Knoess and Bauer obtained good densification with high conductive powders like iron and copper. Knoess used various pulses of very high current (>100 kA/cm^2) and Bauer an intensity lower that 10 kA/cm^2, voltage lower than 10V using a sintering time around 1 s. The problem may occur when high electrical resistance samples are manufactured (due to the high resistivity of the powder or because of the large size of the parts), it will not be possible to close the electric circuit so the current can pass the whole material of the parts. For that reason, with these techniques it will not be possible to obtain larger samples or the consolidation of powders with higher resistivity due to the low voltage used.

[0007] JP H03 236402 discloses an apparatus for carrying out the sintering of electrically conducting powders.

DESCRIPTION OF THE INVENTION

[0008] To overcome the drawbacks of the prior art, the present invention proposes a method of sintering electrically conducting powders in an air atmosphere for obtaining a sintered product, comprising the following sequence of steps:

a) placing the powders in an electrically isolating mold,
b) applying a pressure to the powders between 100 and 500MPa,
c) applying to the powders a sintering current density at a sintering voltage during a sintering time, for sintering the powders.

[0009] The method of the present invention comprises, between step b) and step c), applying to the powders an activation current density lower than the sintering current density at an activation voltage greater than the sintering voltage during an activation time lower than the sintering time, to reduce the electrical resistance of the powders, the activation current density and the sintering current density being constant.

[0010] The application of the activation current density and sintering current density is carried out while the pressure is being applied to the powders.

[0011] The activation current density applied to the powders at a voltage greater than the voltage used for sintering in step c) during a lower time than step c) produces a current discharge that breaks the oxide layer in the surface of the powders and creates bridges between the particles of powders, obtaining a more uniform and cleaner particle surface which reduces the electrical resistance to the flow of the current through the powders such that the sintering current density applied in step c) is distributed more homogeneously through the powders in the mold. Thus is possible to sinter large size parts and parts made of material with a high electrical resistivity.

[0012] Preferably the activation current density is greater than 0,5 kA/cm^2, the activation voltage is greater than 10V and the activation time is lower than 300ms, for generating a current discharge of low intensity at a high voltage in a very reduced time, to assure an homogeneous superficial de-oxidation of the powders and formation of bridges among particles.

[0013] According to the invention, the time between the removal of the activation current density and the application of the sintering current density is lower than 20 ms to assure an optimal distribution of the sintering current density. Most preferably the sintering current density is applied immediately after the application of the activation current density, i.e just after the activation time is run out.

[0014] According to a preferred aspect of the invention:

- The activation current density is applied by employing a first electrical power unit.
- The sintering current density is applied by employing a second electrical power unit.
- The first and second electrical power units are operated independently.

[0015] The method of the invention comprises the control of the two electrical power units which enables to optimize the processing time and the energy consumption, altogether with the installation costs.

[0016] Further it allows to program and monitor at all times the power that is being introduced in the powder, thus allowing the process to be controlled very accurately and repetitively, both in the application of the activation current density and in the application of the sintering current density.

[0017] Furthermore, it has been found that the precise control enabled by this method, allows a considerable increase in the parts size, their geometry, and the types of materials that can be sintered.

[0018] According to a preferred embodiment of the invention:

- The activation current density is comprised in the range going from 0.5 to 5 kA/cm^2;
- The activation voltage is comprised in the range going from 10 to 100 V;
- The activation time is comprised in the range going from 50 to 300 ms;
- The sintering current density is comprised in the range going from 3 to 15 kA/cm^2;
- The sintering voltage is lower than 15 V;
- The sintering time is comprised in the range going from 500 to 1500 ms,

but these parameters must fulfil the conditions that the selected values for the sintering current density applied to the powders must be greater than the selected values for the activation current density and the selected values for the activation voltage always must be greater than the sintering voltage applied.

[0019] According to a most preferred embodiment of the invention:

- The activation current density is comprised in the range going from 0.5 to 4 kA/cm^2;
- The activation voltage is comprised in the range going from 10 to 100 V;
- The activation time is comprised in the range going from 90 to 200 ms;
- The sintering current density is comprised in the range going from 3 to 10 kA/cm^2;
- The sintering voltage is lower than 10 V;
- The sintering time is comprised in the range going from 500 to 1500 ms,

but these parameters must fulfil the conditions that the selected values for the sintering current density applied to the powders must be greater than the selected values for the activation current density and the selected values for the activation voltage always must be greater than the sintering voltage applied.

[0020] The skilled person will select the precise values of these parameters for each conductive powder, but always bearing in mind that the activation current density applied to one conductive powder must be lower than the sintering current density applied and the activation voltage greater than the sintering voltage. Thus it is not possible to choose a value of 4 kA/cm^2 for the activation current density and a value of 3 kA/cm^2 for the sintering current density. Same applies to the activation and sintering voltages where is not possible to apply a activation voltage of 10 V and a sintering voltage of 15 V. Some examples of parameters selections are disclosed in the description of preferred embodiments.

BRIEF DESCRIPTION OF THE DRAWINGS

[0021] To complete the description and in order to provide for a better understanding of the invention, a set of drawings is provided. Said drawings form an integral part of the description and illustrate an embodiment of the invention, which should not be interpreted as restricting the scope of the invention, but just as an example of how the invention can be carried out. The drawings comprise the following figures:

FIG. 1 is a block diagram of an apparatus according to a preferred embodiment.

FIG. 2 is a time plot of the pressure and the voltage/current when the inventive method is applied to a WC-Co powder.

DESCRIPTION OF A WAY OF CARRYING OUT THE INVENTION

[0022] In FIG. 2 the time pressure/ current/voltage diagram corresponding to the implementation of the method according to the invention for obtaining a sintered WC-Co is shown.

[0023] The process starts with the step consisting in placing an electrically conducting powder in an electrically insulating mold.

[0024] Then a pressure between 100 and 500 MPa is applied inside the mold, preferably with two pistons, in this case around 300 MPa.

[0025] Then an activation step is carried out, consisting in applying an activation current density at an activation voltage for an activation time and carried out by employing a first electrical power unit (2). As shown, in this step a low current density (around 2 kA/cm^2) and a high voltage (around 30V) are applied. The pulse is about two tenths of a second.

[0026] Then a waiting step is carried out wherein no current and/or voltage are applied. This step consists in the switching of the power units, that is, to switch from a power unit (2) to another power unit (3). The waiting time is that needed for carrying out said switching by the control unit (4), in the present case a PLC. In FIG. 2 the switching time is about 2 tenth of a second. A technical possibility would be to use a single power unit but with instantaneously variable current and voltage. However, the control requirements for the current and voltage levels and the discharge times would imply very sophisticated equipment that would make the method uneconomic at industrial level.

[0027] Then, the proper sintering step is performed, which consists in applying a sintering current density at a sintering voltage during a sintering time carried out by employing the second electrical power unit (3). In this case the intensity is higher (around 10 kA), but the voltage is reduced to 5 V.

[0028] The current density is applied using two opposite electrodes. In an embodiment the pistons can be used as opposite electrodes.

[0029] As shown in FIG 1, and according to a preferred embodiment, the invention also relates to an apparatus (1) for carrying out the inventive method.

[0030] The apparatus comprises:

- means for applying current and voltage to the powders, represented by the power units (2, 3);
- an electrically insulating mold (5) containing the conductive powders (6), which is closed in its ends by two pistons for applying mechanical pressure and which form the electrodes (7) as well.

[0031] As shown in FIG. 1, the means for providing current and voltage for an activation step is a first electrical power unit (2) and the means for providing current and voltage for a sintering step is a second electrical power unit (3).

[0032] The first power unit (2) is arranged to provide through the electrodes (7) an activation current density comprised between 0.5 and 5 KA/cm^2 and an activation voltage comprised between 10 and 100 V whereas the second power unit (3) is arranged to provide through electrodes (7) a sintering current density comprised between 3 and 15 kA/cm^2 and a sintering voltage lower than 15 V. These ranges allow to sinter most of the commercially interesting conductive powders for typical applications, with a single machine, which parameters have to be set prior to the sintering.

[0033] The apparatus further comprises:

- Means for switching between the first 2 and the second 3 electrical power unit;
- Means for controlling the duration of the current density and voltage provided by the first power unit (2);
- means for controlling the duration of the current density and voltage provided by the second power unit (3);
- connections (23, 33) between each of the power units (2, 3) and the electrodes (7) of the mold (5).
- means for controlling the pistons that apply pressure in the mold.

[0034] The means for controlling the duration of the current density and voltage provided by the first power unit (2) are able to control a predetermined discharge time (activation time) comprised in the range going from 50 to 300 ms and the means for controlling the duration of the current and voltage provided by the second power unit (3) are able to control a predetermined discharge time (sintering time) comprised in the range going from 500 to 1500 ms.

[0035] Each power unit (2, 3) comprise a transformer (21, 31) and an inverter (22, 32), and the two power units (2, 3) are controlled by a single control unit (4), which is preferably a programmable logic controller.

[0036] This PLC includes:

- means for switching between the first (2) and the second (3) electrical power unit,
- means for controlling the duration of the current and voltage provided by the first power unit (2),
- means for controlling the duration of the current and voltage provided by the second power unit (3); and
- means for controlling the pistons that apply pressure in the mold.

[0037] Now, specific examples of application of the method of the invention to different metal powders are described.

EXAMPLE 1: WC-6Co / WC-10Co

[0038] A WC-6Co or WC-10Co disk is produced with the disclosed apparatus with a thickness of 16 mm and a diameter of 22 mm. The agglomerated powder was spherical with an agglomerate size of less than 100 microns.
In the activation step a current density between 2 and 4 kA/cm^2 during 100-200 ms was applied in order to activate the powder. A voltage between 15-50 V is needed for this activation step.
[0039] In the subsequent sintering stage a current density between 6-10 kA/cm^2 was applied to obtain a densified sample with a voltage lower than 10 V during 500-1000 ms. Between stages, activation and sintering, a minimum time of 10 ms was established. Pressure, from 100-500 MPa, was applied from the beginning of the process.
[0040] The density of the final disk, measured by the Archimedes method, is around 13-14.8 g/cm^3. It is possible to obtain fully dense samples with hardness around 1800-2100 HV30.

EXAMPLE 2: Titanium

[0041] A titanium disk is produced with the disclosed apparatus with a thickness of 10 mm and a diameter of 22 mm. The shape of the particles of the powder was irregular with a maximum particle size around 75 microns.
[0042] In the activation step a current density between 1-3 kA/cm^2 was applied during 90-100 ms in order to activate the powder. A voltage between 10-50 V is needed for the activation stage.
[0043] In the sintering stage a current density between 4-7 kA/cm^2 was applied during 500-1000 ms to obtain a densified sample with a voltage lower than 10 V. Between stages, activation and sintering, a minimum time of 10 ms was established. Pressure, from 100-500 MPa, was applied from the beginning of the process.
[0044] The density of the final disk, measured by the Archimedes method, is around 3.5-4.4 g/cm^3. It is possible to obtain fully dense samples.

EXAMPLE 3: TiC-25Ni and TiC-25Fe

[0045] A TiC-25Ni and TiC-25Fe disks are produced with the disclosed apparatus with a thickness of 16 mm and a diameter of 22 mm. The agglomerated powder was irregular with a particle size of less than 30 microns.
[0046] In the activation step a current density between 1-3 kA/cm^2 was applied during 100-200 ms in order to activate the powder. A voltage between 15-50 V is needed for this activation stage.
[0047] In the subsequent sintering step a current density between 6-9 kA/cm^2 was applied during 500-1000 ms to obtain a densified sample with a voltage lower than 10 V. Between stages, activation and sintering, a minimum time of 10 ms was stablished. Pressure, from 100-500 MPa, was applied from the beginning of the process.
[0048] The density of the final disk, measured by the Archimedes method, was around 5.1-5.5 g/cm^3 for TiC-25Ni and 5.1-5.4 g/cm^3 for TiC-25Fe. It is possible to obtain fully dense samples with hardness around 1600-2000 HV30.

EXAMPLE 4: Aluminium

[0049] An aluminium disk was produced with the disclosed apparatus with a thickness of 12 mm and a diameter of 12 mm. The powder was irregular with a particle size of less than 150 microns.
[0050] In the activation step a current density between 0.5-2 kA/cm^2 was applied during 100-200 ms in order to activate the powder. A voltage between 30-80 V is needed for this activation stage.
[0051] In the subsequent sintering stage a current density between 3-4 kA/cm^2 was applied during 500-1000 ms to obtain a densified sample with a voltage lower than 10 V. Between stages, activation and sintering, a minimum time of 10 ms was established. Pressure, from 100-300 MPa, was applied from the beginning of the process.
[0052] The density of the final disk, measured by the Archimedes method, was around 2.5-2.7 g/cm^3.
[0053] In this text, the term "comprises" and its derivations should not be understood in an excluding sense, that is, these terms should not be interpreted as excluding the possibility that what is described and defined may include further elements, steps, etc.
[0054] The invention is obviously not limited to the specific embodiments described herein, but also encompasses any variations that may be considered by any person skilled in the art, within the general scope of the invention as defined in the claims.

Claims

1. Method of sintering electrically conducting powders in an air atmosphere for obtaining a sintered product, comprising the following sequence of steps:

a) placing the powders in an electrically isolating mold,
b) applying a pressure to the powders between 100 and 500MPa,
c) applying to the powders a sintering current at a sintering voltage during a sintering time, for sintering the powders,

characterized by applying to the powders, between step b) and step c), an activation current density lower than the sintering current density at an activation voltage greater than the sintering voltage during an activation time lower than the sintering time, to reduce the electrical resistance of the powders, the activation current density and the sintering current density being constant

2. Method according to claim 1, wherein the activation current density is greater than 0.5 kA/cm^2, the activation voltage is greater than 10V and the activation time is lower than 300ms,

3. Method according to any of the previous claims, wherein:

- The application of the sintering current density and sintering voltage are carried out by employing a first electrical power unit (2);
- The application of the activation current density and activation voltage are carried out by employing a second electrical power unit (3);
- The first and second electrical power units (2, 3) operate independently.

4. Method according to any of the previous claims, wherein:

- The activation current density is comprised in the range going from 0.5 to 5 kA/cm^2;
- The activation voltage is comprised in the range going from 10 to 100 V;
- The activation time is comprised in the range going from 50 to 300 ms;
- The sintering current density is comprised in the range going from 3 to 15 kA/cm^2;
- The sintering voltage is lower than 15 V;
- The sintering time is comprised in the range going from 500 to 1500 ms,

and wherein the activation current density is lower than the sintering current density and the activation voltage is greater than the sintering voltage.

5. Method according to claims 1 to 4, wherein:

- The powders are WC-6Co powders or WC-10Co;
- The activation current density is comprised in the range going from 2 to 4 kA/cm^2;
- The activation voltage is comprised in the range going from 15 to 50 V;
- The activation time is comprised in the range going from 100 to 200 ms;
- The sintering current density is comprised in the range going from 6 to 10 kA/cm^2;
- The sintering voltage is lower than 10 V;
- The sintering time is comprised in the range going from 500 to 1000 ms.

6. Method according to claims 1 to 4, wherein:

- The powders are titanium powders;
- The activation current density is comprised in the range going from 1 to 3
- KA/cm^2;
- The activation voltage is comprised in the range going from 10 to 50 V;
- The activation time is comprised in the range going from 90 to 110 ms;
- The sintering current density is comprised in the range going from 4 to 7 kA/cm^2;
- The sintering voltage is lower than 10 V;
- The sintering time is comprised in the range going from 500 to 1000 ms.

7. Method according to claims 1 to 4, wherein:

- The powders are a mixture of TiC-25Ni powders and TiC-25Fe powders;
- The activation current density is comprised in the range going from 1 to 3 kA/cm^2;
- The activation voltage is comprised in the range going from 15 to 50 V;
- The activation time is comprised in the range going from 100 to 200 ms;
- The sintering current density is comprised in the range going from 6 to 9 kA/cm^2;
- The sintering voltage is lower than 10 V;
- The sintering time is comprised in the range going from 500 to 1000 ms.

8. Method according to claims 1 to 4, wherein:

- The powders are an aluminium powders;
- The activation current density is comprised in the range going from 0.5 to 2 kA/cm^2;
- The activation voltage is comprised in the range going from 30 to 80 V;
- The activation time is comprised in the range going from 100 to 200 ms;
- The sintering current density is comprised in the range going from 3 to 4 kA/cm^2;
- The sintering voltage is lower than 10 V;
- The sintering time is comprised in the range going from 500 to 1000 ms.

Patentansprüche

1. Verfahren zum Sintern elektrisch leitfähiger Pulver in einer Luftatmosphäre zum Erhalten eines Sinterprodukts, umfassend die folgende Abfolge von Schritten:

a) Einbringen der Pulver in eine elektrisch isolierende Form,

b) Anlegen eines Drucks zwischen 100 und 500 MPa auf die Pulver,

c) während einer Sinterzeit Anlegen eines Sinterstroms an die Pulver bei einer Sinterspannung zum Sintern der Pulver,

dadurch gekennzeichnet, dass zwischen Schritt b) und Schritt c) eine Aktivierungsstromdichte, die geringer ist als die Sinterstromdichte, bei einer Aktivierungsspannung, die höher ist als die Sinterspannung, während einer Aktivierungszeit, die kürzer ist als die Sinterzeit, an die Pulver angelegt wird, um den elektrischen Widerstand der Pulver zu verringern, wobei die Aktivierungsstromdichte und die Sinterstromdichte konstant sind.

2. Verfahren nach Anspruch 1, wobei die Aktivierungsstromdichte größer als 0,5 kA/cm^2 ist, die Aktivierungsspannung größer als 10 V ist und die Aktivierungszeit kürzer als 300 ms ist.

3. Verfahren nach einem der vorhergehenden Ansprüche, wobei:

- das Anlegen der Sinterstromdichte und der Sinterspannung unter Anwendung eines ersten Netzanschlussgeräts (2) durchgeführt wird;
- das Anlegen der Aktivierungsstromdichte und der Aktivierungsspannung unter Anwendung eines zweiten Netzanschlussgeräts (3) durchgeführt wird;
- das erste und das zweite Netzanschlussgerät (2, 3) unabhängig voneinander arbeiten.

4. Verfahren nach einem der vorhergehenden Ansprüche, wobei:

- die Aktivierungsstromdichte im Bereich von 0,5 bis 5 kA/cm^2 liegt;
- die Aktivierungsspannung im Bereich von 10 bis 100 V liegt;
- die Aktivierungszeit im Bereich von 50 bis 300 ms liegt;
- die Sinterstromdichte liegt im Bereich von 3 bis 15 kA/cm^2;
- die Sinterspannung unter 15 V liegt;
- die Sinterzeit im Bereich von 500 bis 1500 ms liegt,

wobei die Aktivierungsstromdichte geriger als die Sinterstromdichte ist und die Aktivierungsspannung größer als die Sinterspannung ist.

5. Verfahren nach den Ansprüchen 1 bis 4, wobei:

- die Pulver WC-6Co- oder WC-10Co-Pulver sind;
- die Aktivierungsstromdichte im Bereich von 2 bis 4 kA/cm^2 liegt;
- die Aktivierungsspannung im Bereich von 15 bis 50 V liegt;
- die Aktivierungszeit im Bereich von 100 bis 200 ms liegt.
- die Sinterstromdichte im Bereich von 6 bis 10 kA/cm^2 liegt;
- die Sinterspannung unter 10 V liegt;
- die Sinterzeit im Bereich von 500 bis 1000 ms liegt.

6. Verfahren nach den Ansprüchen 1 bis 4, wobei:

- die Pulver Titanpulver sind;
- die Aktivierungsstromdichte im Bereich von 1 bis 3 KA/cm^2 liegt;
- die Aktivierungsspannung im Bereich von 10 bis 50 V liegt;
- die Aktivierungszeit im Bereich von 90 bis 110 ms liegt.
- die Sinterstromdichte im Bereich von 4 bis 7 kA/cm^2 liegt;
- die Sinterspannung unter 10 V liegt;
- die Sinterzeit im Bereich von 500 bis 1000 ms liegt.

7. Verfahren nach den Ansprüchen 1 bis 4, wobei:

- die Pulver eine Mischung aus TiC-25Ni-Pulvern und TiC-25Fe-Pulvern sind;
- die Aktivierungsstromdichte im Bereich von 1 bis 3 kA/cm^2 liegt;
- die Aktivierungsspannung im Bereich von 15 bis 50 V liegt;
- die Aktivierungszeit im Bereich von 100 bis 200 ms liegt.
- die Sinterstromdichte im Bereich von 6 bis 9 kA/cm^2 liegt;
- die Sinterspannung unter 10 V liegt;
- die Sinterzeit im Bereich von 500 bis 1000 ms liegt.

8. Verfahren nach den Ansprüchen 1 bis 4, wobei:

- die Pulver Aluminiumpulver sind;
- die Aktivierungsstromdichte im Bereich von 0,5 bis 2 kA/cm2 liegt;
- die Aktivierungsspannung im Bereich von 30 bis 80 V liegt;
- die Aktivierungszeit im Bereich von 100 bis 200 ms liegt.
- die Sinterstromdichte im Bereich von 3 bis 4 kA/cm^2 liegt;
- die Sinterspannung unter 10 V liegt;
- die Sinterzeit im Bereich von 500 bis 1000 ms liegt.

Revendications

1. Procédé de frittage de poudres électriquement conductrices dans une atmosphère d'air pour obtenir un produit fritté, comprenant la séquence d'étapes suivante consistant à :

   a) placer les poudres dans un moule électriquement isolant,
   b) appliquer aux poudres une pression comprise entre 100 et 500 MPa,
   c) appliquer aux poudres un courant de frittage à une tension de frittage pendant un temps de frittage, pour fritter les poudres,

   caractérisé par le fait d'appliquer aux poudres, entre l'étape b) et l'étape c), une densité de courant d'activation inférieure à la densité de courant de frittage à une tension d'activation supérieure à la tension de frittage pendant un temps d'activation inférieur au temps de frittage, afin de réduire la résistance électrique des poudres, la densité de courant d'activation et la densité de courant frittage étant constantes.

2. Procédé selon la revendication 1, dans lequel la densité de courant d'activation est supérieure à 0,5 kA/cm^2, la tension d'activation est supérieure à 10 V et le temps d'activation est inférieur à 300 ms.

3. Procédé selon l'une quelconque des revendications précédentes, dans lequel :

   - l'application de la densité de courant de frittage et de la tension de frittage est réalisée en utilisant une première unité de puissance électrique (2) ;
   - l'application de la densité de courant d'activation et de la tension d'activation est réalisée en utilisant une deuxième unité de puissance électrique (3) ;
   - la première et la deuxième unité de puisse électrique (2, 3) fonctionnent indépendamment l'une de l'autre.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel :

   - la densité de courant d'activation est comprise dans la fourchette allant de 0,5 à 5 kA/cm^2 ;
   - la tension d'activation est comprise dans la fourchette allant de 10 à 100 V ;
   - le temps d'activation est compris dans la fourchette allant de 50 à 300 ms ;
   - la densité de courant de frittage est comprise dans la fourchette allant de 3 à 15 kA/cm^2 ;
   - la tension de frittage est inférieure à 15 V ;
   - le temps de frittage est compris dans la fourchette allant de 500 à 1 500 ms, la densité de courant d'activation étant inférieure à la densité de courant de frittage et la tension d'activation étant supérieure à la tension de frittage.

5. Procédé selon les revendications 1 à 4, dans lequel :

   - les poudres sont des poudres WC-6Co ou WC-10Co ;
   - la densité de courant d'activation est comprise dans la fourchette allant de 2 à 4 kA/cm^2 ;
   - la tension d'activation est comprise dans la fourchette allant de 15 à 50 V ;
   - le temps d'activation est compris dans la fourchette allant de 100 à 200 ms ;
   - la densité de courant de frittage est comprise dans la fourchette allant de 6 à 10 kA/cm^2 ;
   - la tension de frittage est inférieure à 10 V ;
   - le temps de frittage est compris dans la fourchette allant de 500 à 1 000 ms.

6. Procédé selon les revendications 1 à 4, dans lequel :

   - les poudres sont des poudres de titane ;
   - la densité de courant d'activation est comprise dans la fourchette allant de 1 à 3 KA/cm^2 ;
   - la tension d'activation est comprise dans la fourchette allant de 10 à 50 V ;
   - le temps d'activation est compris dans la fourchette allant de 90 à 110 ms ;
   - la densité de courant de frittage est comprise dans la fourchette allant de 4 à 7 kA/cm^2 ;
   - la tension de frittage est inférieure à 10 V ;
   - le temps de frittage est compris dans la fourchette allant de 500 à 1 000 ms.

7. Procédé selon les revendications 1 à 4, dans lequel :

   - les poudres sont un mélange de poudres de TiC-25Ni et de poudres de TiC-25Fe ;
   - la densité de courant d'activation est comprise dans la fourchette allant de 1 à 3 kA/cm^2 ;
   - la tension d'activation est comprise dans la fourchette allant de 15 à 50 V ;
   - le temps d'activation est compris dans la fourchette allant de 100 à 200 ms ;
   - la densité de courant de frittage est comprise dans la fourchette allant de 6 à 9 kA/cm^2 ;
   - la tension de frittage est inférieure à 10 V ;
   - le temps de frittage est compris dans la fourchette allant de 500 à 1 000 ms.

8. Procédé selon les revendications 1 à 4, dans lequel :

   - les poudres sont des poudres d'aluminium ;
   - la densité de courant d'activation est comprise dans la fourchette allant de 0,5 à 2 kA/cm^2 ;
   - la tension d'activation est comprise dans la

fourchette allant de 30 à 80 V ;
- le temps d'activation est compris dans la fourchette allant de 100 à 200 ms ;
- la densité de courant de frittage est comprise dans la fourchette allant de 3 à 4 kA/cm^2 ;
- la tension de frittage est inférieure à 10 V ;
- le temps de frittage est compris dans la fourchette allant de 500 à 1 000 ms.

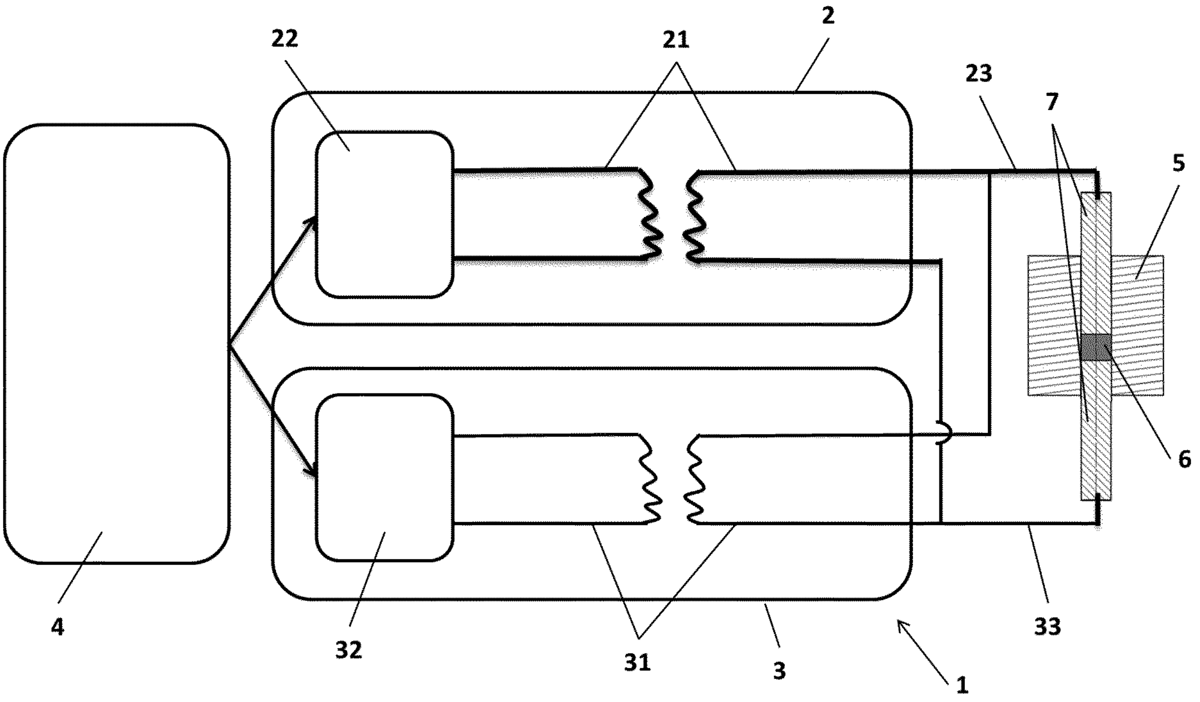
22
21
2
23
7
5
6
4
32
31
3
1
33

FIG.1

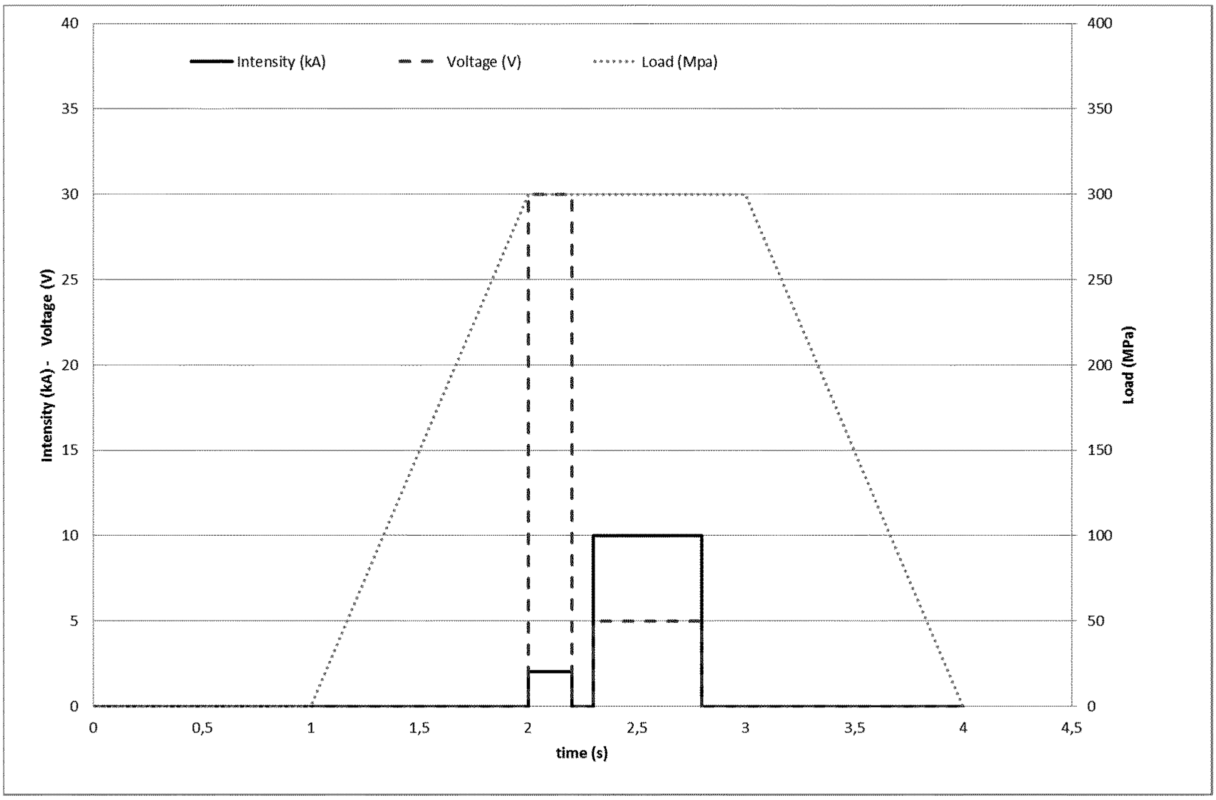
Intensity (kA)
Voltage (V)
Load (Mpa)
Intensity (kA) - Voltage (V)
Load (MPa)
time (s)
0
0,5
1
1,5
2
2,5
3
3,5
4
4,5
5
10
15
20
25
30
35
40
50
100
150
200
250
300
350
400

FIG.2

REFERENCES CITED IN THE DESCRIPTION

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