(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
1 September 2016 (01.09.2016)

WIPO | PCT

(10) International Publication Number
WO 2016/135187 A1

(51) International Patent Classification:
B22F 3/08 (2006.01) ***C22C 38/02*** (2006.01)
B22F 3/16 (2006.01) *B30B 11/00* (2006.01)
B30B 11/02 (2006.01) *B22F 9/08* (2006.01)
C22C 38/00 (2006.01) *C21D 3/04* (2006.01)
B22F 3/087 (2006.01) *C23C 8/20* (2006.01)
B22F 3/10 (2006.01) *C23C 8/64* (2006.01)

(21) International Application Number:
PCT/EP2016/053852

(22) International Filing Date:
24 February 2016 (24.02.2016)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
1550209-9 25 February 2015 (25.02.2015) SE

(71) Applicant: **HYP UTHYRNING AB** [SE/SE]; Projektilgatan 2, 691 50 Karlskoga (SE).

(72) Inventor: **ÅSLUND, Christer**; Lilla gatan 8, 644 31 Torshälla (SE).

(74) Agent: **BERGENSTÅHLE GROUP AB**; P.O. Box 177 04, 118 93 Stockholm (SE).

(81) **Designated States** *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) **Designated States** *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report (Art. 21(3))*

WO 2016/135187 A1

(54) Title: COMPACTING OF GAS ATOMIZED METAL POWDER TO A PART

(57) **Abstract**: A process for making full dense components of a carbon-containing steel, comprises the steps of: a) making a powder of the carbon-containing steel by gas atomization wherein the carbon content is low, less than 0.15 wt%, b) agglomerating the powder from step a) with at least one hydrocolloid and elemental carbon, c) compacting the agglomerated powder from step b) to a density of at least 80%of theoretical density, with the proviso that the compacted agglomerated powder still is porous allowing transport of gas to and from its interior, and d) sintering the compacted powder to a density of more than 98% of theoretical density, preferably more than 99 % of theoretical density, wherein a gas comprising carbon is added during sintering and finally subjecting the component to HVC. Advantages include that it is possible to manufacture a dense component of powders which otherwise are difficult to compact.

COMPACTING OF GAS ATOMIZED METAL POWDER TO A PART

Technical field

[0001] The present invention relates generally to a process for compacting carbon containing hardenable steels. It further relates to a part manufactured by such a process.

Background

[0002]Certain powder processes in the art use gas atomized powder of pre-alloyed steel alloys unlike conventional powder technology and where the spherical gas atomized powder to obtain good green strength agglomerated to a press powder before pressing / sintering. An example of the first mentioned process is described under the trade name Scanpac® MMS.

[0003]When using gas atomized powder of conventional carbon steel and other hardenable steels, those are hardened during the atomization process itself because during the atomization process the cooling occurs so rapidly that the powder becomes hardened. The hardness is of course determined by the alloy itself.

[0004]It is well known that the carbon content determines the hardness of a typical carbon steel and certain other alloys, e.g. martensitic, ferritic stainless chromium steel. Typically the carbon content of commercial carbon steel is from approximately 0.2wt% (wt) and up to about 1.3wt%.

[0005]The hardenability of ferrous alloys, i.e. steels, is a function of the carbon content and other alloying elements and the grain size of the austenite. The hardenability of a ferrous alloy is measured by a Jominy test: a round metal bar of standard size (indicated in the top image) is transformed to 100% austenite through heat treatment, and is then quenched on one end with room-temperature water. The cooling rate will be highest at the end being quenched, and will decrease as distance from the end increases. Subsequent to cooling a flat surface is ground on the test piece and the hardenability is then found by measuring the hardness along the bar. The farther away from the quenched end that the hardness extends, the higher the hardenability.

[0006]Direct pressing of gas atomized powder of pre-alloyed steel alloys gives a hard powder with very low densities of the pressed component after the initial pressing. This is obviously a disadvantage and adversely affects the subsequent process steps; sintering, re-compaction (restrike) and final sintering or capsular-free HIP, because the goal is to reach as high a density as possible. The goal is to reach a final density of over 99% of the theoretical density.

[0007]One approach in the art is to soft anneal the metal powder before the agglomeration. This is however not straightforward, since the metal powder will sinter to some extent during the soft annealing. After such a soft annealing the pieces of sintered powder will have to be crushed and sieved to the correct particles size before use. This procedure is expensive, complicated and time consuming.

[0008]Thus it is a problem in the prior art how to provide a method which in an easier, faster and less expensive way achieves a final density over 99% (or at least over 98%) of the theoretical density for hardenable steels, i.e. for alloys comprising carbon in particular carbon steel.

Summary

[0009]It is an object of the present invention to obviate at least some of the disadvantages in the prior art and provide an improved method to manufacture components of metal powder.

[0010] In a first aspect there is provided a process for making full dense components with a density of more than 98%, preferably more than 99% of the theoretical density of a material selected from the group consisting of a carbon-containing steel and a carbon containing alloy, said process comprising the steps of:

a) making a powder of the carbon-containing steel by gas atomization, wherein the carbon content is less than 0.15wt%,

b) agglomerating the powder from step a) with at least one hydrocolloid, wherein carbon in elemental form is added during the agglomeration,

c) compacting the agglomerated powder from step b) in a first compacting step to a density of at least 80% of theoretical density, with the proviso that the compacted agglomerated powder still is porous allowing transport of gas to and from its interior,

d) further compacting the compacted agglomerated powder from step c) to a density of more than 98% of theoretical density using at least sintering followed by HVC, high velocity compacting with a ram speed exceeding 5 m/s, wherein a gas comprising carbon is added during sintering.

[0011]In a second aspect there is provided a full dense component manufactured according to the procedure above, with a density of more than 98% of the theoretical density, wherein the component comprises a carbon-containing steel.

[0012]Further aspects and embodiments are defined in the appended claims.

[0013]One advantage is that it is possible to manufacture components with theoretical density (i.e. more than 98% of theoretical density, preferably more than 99% of theoretical density) of agglomerated particles of carbon steel which is otherwise difficult and/or at least expensive.

Detailed description

[0014]Before the invention is disclosed and described in detail, it is to be understood that this invention is not limited to particular compounds, configurations, method steps, substrates, and materials disclosed herein as such compounds, configurations, method steps, substrates, and materials may vary somewhat. It is also to be understood that the terminology employed herein is used for the purpose of describing particular embodiments only and is not intended to be limiting since the scope of the present invention is limited only by the appended claims and equivalents thereof.

[0015]As used throughout the text a steel denotes an alloy of iron and other elements, primarily carbon.

[0016]Theoretical density denotes the maximum density of a material which is possible to achieve in theory, i.e. for a perfect material without any defects.

[0017]It must be noted that, as used in this specification and the appended claims, the singular forms “a”, “an” and “the” include plural referents unless the context clearly dictates otherwise.

[0018]If nothing else is defined, any terms and scientific terminology used herein are intended to have the meanings commonly understood by those of skill in the art to which this invention pertains.

[0019] In a first aspect there is provided a process for making full dense components with a density of more than 98%, preferably more than 99% of the theoretical density of a material selected from the group consisting of a carbon-containing steel and a carbon containing alloy, said process comprising the steps of:

a) making a powder of the carbon-containing steel by gas atomization, wherein the carbon content is less than 0.15wt%,

b) agglomerating the powder from step a) with at least one hydrocolloid, wherein carbon in elemental form is added during the agglomeration,

c) compacting the agglomerated powder from step b) in a first compacting step to a density of at least 80% of theoretical density, with the proviso that the compacted agglomerated powder still is porous allowing transport of gas to and from its interior,

d) further compacting the compacted agglomerated powder from step c) to a density of more than 98% of theoretical density using at least sintering followed by HVC, high velocity compacting with a ram speed exceeding 5 m/s, wherein a gas comprising carbon is added during sintering.

[0020]In many embodiments the carbon-containing steel becomes hardenable after the steps a) to c) above, i.e. the type of alloy (steel) and the carbon content after addition of the carbon in step b) is selected so that the material becomes possible to harden. In one embodiments the carbon-containing steel is hardenable after step c). The carbon-containing steel prior to gas atomization in step a) is in one embodiment treated so that the carbon content is reduced to a carbon content less than 0.15wt%. This reduction in carbon content makes it possible to manufacture a powder using gas atomization which is easier than many other methods, the gas atomization can thus be used even for steels which have a high carbon content and are hardenable. Carbon in elemental form is added during the agglomeration in step b) so that the carbon content is increased to the desired value. In one embodiment the carbon content is selected so that the steel (or alloy) is hardenable. “Hardenability” or hardenable is the ability of an alloy comprising Fe-C to be hardened by the forming martensite as a result of a given heat treatment.

[0021]All sintering atmospheres have a certain amount of oxygen. The amount of oxygen is usually measured with a so called dew point meter which measures the dew point. The dew point level is a measure of amount of oxygen in the sintering atmosphere at a given temperature.

-5°C corresponds to 0.4 % H_2O

-10°C corresponds to 0.27 % H_2O

-20°C corresponds to 0.1 % H_2O

[0022]The actual oxygen atom in the water combines with carbon and forms CO which is a more stable compound.

[0023]If nothing is done to compensate and/or hinder this reaction the part will be depleted of carbon and the material is not fulfilling its analysis, i.e. the desired amount of carbon in the material will not be reached.

[0024]Even if the furnace is started with a low dew point, when the temperature in the furnace is raised, oxygen/H_2O is released from the insulation, the powder, the binder itself and so on. A typical value of dew point during sintering is -20°C.

[0025]Methane CH_4 is a stable compound up to high temperature but reacts with H_2O at high temperature and form CO and free hydrogen. You can therefore use a gas comprising carbon such as but not limited to methane as a “sacrifice” compound in order to prevent the loss of carbon in the pressed powder part during sintering in hydrogen.

[0026]By using a routine experiment the skilled person is able to decide the exact amount of CH_4 to be added in order to stabilize and create a “neutral” atmosphere during sintering to prevent the decarburization of the part. It is desired to create such an atmosphere in order to reach the desired carbon content in the final product. The below example(s) give a guide for this, for instance example 6 below. The skilled person can in the light of this description perform a routine experiment to determine the exact amount of methane to be added. The exact concentration needed depends on the furnace and on the amount of material in the furnace etc. A skilled person can repeat the experiment in example 6 below and similarly determine a suitable methane concentration to use.

[0027]The finished part has a carbon content higher than the initially provided powder in step a) due to additions of carbon in elemental form (in step b) and the gas comprising carbon (in step c).

[0028]The gas comprising carbon is able to reach into the part after step c) since it is still porous. After the sintering in step d) the part is not porous anymore. After step c) the density is at least 80 % of the theoretical density but not too dense so that it is still porous with a percolated structure so that gas can pass to and from the inner regions of the part. In one embodiment the density after step c) is in the interval 80-90 % of theoretical density. In an alternative embodiment the density after step c) is in the interval 85-90 % of theoretical density. In yet another embodiment the density after step c) is in the interval 80-92% of theoretical density. For most materials it is not suitable to use higher densities than 92% of theoretical density, since the powder will not be porous anymore and would thus not allow transportation of gases to and from the interior of the part. For some embodiments and materials it is also possible to compact to a density a bit lower than 80 % of the theoretical density in step c). Thus in one embodiment the first compacting step c) is to a density of at least 75% of the theoretical density. The term “% of theoretical density” denotes the density of the part as a percentage of the theoretically highest density for the material. The density is calculated as weight per unit volume.

[0029]In one embodiment the carbon in elemental form added during the agglomeration in step b) is added in an amount corresponding to the difference between the carbon content in the carbon-containing steel in step a) and the desired carbon content of the finished steel after step d). It should be noted that the carbon content of the carbon-containing steel in step a) could be very low.

[0030]In one embodiment the carbon in elemental form in step b) is added in the form of graphite. In one embodiment the carbon in elemental form in step b) is added in the form of a carbon powder. In one embodiment the carbon in elemental form in step b) is added in the form of particles with an average diameter in the interval 0.1-10 µm. This is an advantage since the small particles give a large surface which can react quicker with the surrounding materia. Carbon in any form can be added as small particles, including but not limited to graphite.

[0031]In one embodiment the carbon in elemental form in step b) is added in the form of a colloid of insoluble particles comprising carbon suspended throughout a liquid. The carbon in elemental form in a colloidal suspension can be any type of particles comprising carbon, including but not limited to elemental carbon and graphite. The addition of carbon in colloidal form has the advantage of ensuring an even distribution of the carbon particles. The colloidal suspension of solid particles in a liquid is also referred to as a sol. Preferably the sol is stable and the force originating from the Brownian motion of the particles is of the same order of magnitude or greater than the gravity force so that the sol does not settle by gravity. This ensures an even distribution of the carbon in the part. The stability of the sol is in one embodiment maintained by adding a dispersing agent.

[0032]When sintering with carbon containing steels in reducing atmospheres it is important that the carbon content is controlled.

[0033]With the new process where pure carbon can be added in form of superfine graphite it is clear that the carbon is even more easily removed due to the fact that the carbon is in form of small particles on the existing powder grains and therefore extremely easy to reduce as the part/material is porous. The porosity is typically 10-15 % (corresponding to a density of 85-90% of the theoretical density) allowing free flow of the gases/atmosphere at sintering.

[0034]In one embodiment the carbon-containing steel prior to gas atomization in step a) is treated so that the carbon content is reduced to a carbon content less than 0.10wt%, preferably less than 0.050wt%. The lower carbon content prevents the metal particles from being hardened during gas atomization.

[0035]The compacted agglomerated powder from step c) is sintered. A gas comprising carbon atoms is added during sintering in order to prevent carburizing or decarburization. The gas is in one embodiment methane. It is also possible to conduct the method with other carbon bearing gases including but not limited to propane and gasoline, but experiments performed so far show that methane gives the best accuracy and the best results, at least in the setup used in the experiments.

[0036]During step d) the compacted agglomerated powder from step c) is both sintered and subjected to HVC and optionally further compacting steps. The HVC is a high velocity compacting with a ram speed of 5 m/s or more. In step d) it is conceived that the material is first sintered with the gas and then subjected to HVC. The compacting in step d) can thus be separated in time, i.e. the material can first be sintered with the gas and after some time it can be subjected to HVC.

[0037]In one embodiment the carbon-containing steel prior to gas atomization in step a) is treated with the process argon oxygen decarburization. Argon oxygen decarburization (AOD) is a process used for making stainless steel and other high grade alloys with oxidizable elements such as chromium and aluminum. After initial melting the metal is then transferred to an AOD vessel where it will be subjected to three steps of refining; decarburization, reduction and desulphurization.

[0038]In one embodiment the carbon-containing steel prior to gas atomization in step a) is treated in a vacuum furnace.

[0039]In a second aspect there is provided a full dense component manufactured according to the procedure above, with a density of more than 98% of the theoretical density, wherein the component comprises a carbon-containing steel. In one embodiment the density is more than 99% of the theoretical density.

[0040]In another aspect there is provided a process for making full dense components (i.e. a density of more than 98wt%, preferably more than 99wt% of the theoretical density) of a carbon-containing steel, said process comprising the steps of:

a) Making a powder of the carbon-containing steel by gas atomization,

b) Agglomerating the powder from step a) with at least one hydrocolloid, *c) compacting the agglomerated powder from step b) in a first compacting step to a density of at least 80wt% of theoretical density,*

wherein the carbon-containing steel is hardeneble by the steps a) to c) above, wherein the carbon-containing steel prior to gas atomization in step a) is treated so that the carbon content is reduced to a carbon content less than 0.15wt% and that carbon in elemental form is added during the agglomeration in step b).

[0041]In one embodiment the carbon in elemental form added during the agglomeration in step b) is added in an amount corresponding to the difference between the carbon content in the carbon-containing steel in step a) and the desired carbon content of the finished steel.

[0042]In one embodiment the carbon in elemental form in step b) is added in the form of graphite. In one embodiment the carbon in elemental form in step b) is added in the form of a carbon powder.

[0043]In one embodiment the carbon-containing steel prior to gas atomization in step a) is treated so that the carbon content is reduced to a carbon content less than 0.10wt%, preferably less than 0.050wt%.

[0044]In one embodiment the compacted agglomerated powder from step c) is sintered. In one embodiment a gas comprising carbon atoms is added during sintering in order to prevent carburizing or decarburization. In one embodiment the gas is methane.

[0045]In one embodiment the compacted agglomerated powder from step c) is subjected to at least one treatment selected from the group consisting of sintering, HVC (high velocity compaction, i.e. compaction with a ram speed exceeding 5 m/s)

[0046]In one embodiment the carbon-containing steel prior to gas atomization in step a) is treated with the process argon oxygen decarburization. In one

embodiment the carbon-containing steel prior to gas atomization in step a) is treated in a vacuum furnace.

[0047]All the described alternative embodiments above or parts of an embodiment can be freely combined without departing from the inventive idea as long as the combination is not contradictory.

[0048]Other features and uses of the invention and their associated advantages will be evident to a person skilled in the art upon reading the description and the examples.

[0049]It is to be understood that this invention is not limited to the particular embodiments shown here. The embodiments are provided for illustrative purposes and are not intended to limit the scope of the invention since the scope of the present invention is limited only by the appended claims and equivalents thereof.

[0050]The following gives some examples of the new process.

Examples

Example 1, comparative

[0051]A 42CrMo4 steel type with a carbon content of about 0.4wt% was atomized and sieved to a powder with a maximum particle size of 150 µm. The powder was agglomerated and then pressed in a so-called HVC (High Velocity Compaction) press. The density of the compacted component was measured to be about 74% of the nominal density (theoretical density). This is a very low value. By comparison a standard stainless steel type 316 L (1.4404) was pressed to a density of 90% with approximately the same parameters. It is clear that even if the steel is optimally processed further, it is very difficult, if not impossible, to reach final densities of 98-99% which is necessary so that the finished component shall get sufficient properties compared with the same forged materials.

Example 2

[0052]There was another experiment with the same steel grade as in example 1. In this case the steel was passed through a so-called AOD (Argon Oxygen Decarburizer) where the carbon content in the steel was reduced down to a carbon content of under 0.1wt%, specifically 0.08wt%. After AOD treatment the steel was atomized to powder as described above and agglomerated. After pressing with the same parameters there was obtained a density of the pressed component of 86% of theoretical density. To ensure that the component after the pressing had a proper carbon content there was made an addition to the agglomeration mixture of finely divided graphite, which compensated for the carbon removed with the AOD. The atmosphere in the subsequent sintering was hydrogen, with a small addition of methane with such a concentration that no decarburization occurred during sintering. The added graphite was absorbed by the frit and there was obtained an even distribution of the carbon content throughout the component. After sintering at 1250°C and subsequent annealing and again HVC as well as final sintering there was obtained a final density of 99.2% of theoretical density. The finished component displayed normal structure and normal expected mechanical values.

Example 3

[0053]The same experiment as in example 2 was repeated, but the carbon was reduced to 0.012wt% carbon. At HVC pressing a density of 91% of theoretical density was reached. The final density was in this case 99.6% following the above steps.

Example 4, comparative

[0054]In another example, a steel normally used in the manufacture of ball bearings was manufactured. The steel is called 100Cr6 (1.3505). The steel has a carbon content of about 1wt% and is relatively "hard" when it is hardened. The density after direct compression was only 72% of the theoretical density and thus the final density did not reach more than 94% of theoretical density.

Example 5

[0055]The same process as in example 4 was repeated but where the carbon content was reduced down to 0.009wt% and made according to the invention. It resulted in a density of the pressed component of 90%. The final density was 99.2%, which gave good properties of the finished component. The final density was obtained after sintering with methane and HVC.

Example 6

[0056]A powder with an analysis corresponding to a ball bearing steel 100 Cr 6/EN 1.3505 but with a carbon content of 0.05 % was agglomerated with a hydrocolloide. During the agglomeration carbon was added in form of colloidal graphite with a grain size of 1-3 µ. The amount of graphite was so calculated that the final carbon content should be 1.05 % which is within the range of the standard forged, wrought material.

[0057]The parts were pressed in a HVC press to 87 % of theoretical density.

[0058]The parts were then heated in a batch furnace of type CM with a furnace space of 0.4 m in cubic form to a temperature of 1170°C. Up to 400°C where the binder/hydrocolloide was evaporized the heating speed was 100°C/hr. and after that 200°C/hr. The gas flow of pure hydrogen was 1.6 m^3/hr.

[0059]Methane was added through a flow meter at different rates. On the scale 100 points meant a flow of Methane of 1.01 x 10^{-2} m^3 per hour. This means that about 1 wt% of the carrier gas was hydrogen.

[0060]In the first test the addition was 60 points on the scale. The final carbon analysis was 0.46 wt% carbon which meant that the addition of CH_4 was too low.

[0061]Second trial with 80 points of the scale showed a final carbon content of 0.78 wt% C.

[0062]The next time 100 points gave a carbon content of 0.98 wt%

[0063]120 points on the scale gave a final carbon content of 1.13 wt%.

[0064]The above trial showed that with a scale factor for methane between 100-110 point you got a carbon content within the limits of the standard for the above material 100Cr 6 which is 0.93-1.05 wt% carbon.

[0065]The dew point which was measured during the whole cycle was at start with unloaded furnace -60˚C but increased to approximately - 20°C during sintering.

Example 7

[0066]Similar trials as in example 6 with other carbon bearing gases as propane, gasoline etc worked but did not give the same accuracy and stability as methane in this particular setup. It seems therefore as the methane has a stable behavior and can suitably be used industrially to control the final carbon content when sintering above materials.

[0067]Sintering tests up to 1250° C confirmed this behavior.

CLAIMS

1. A process for making full dense components with a density of more than 98%, preferably more than 99% of the theoretical density of a material selected from the group consisting of a carbon-containing steel and a carbon containing alloy, said process comprising the steps of:

a) making a powder of the carbon-containing steel by gas atomization, wherein the carbon content is less than 0.15wt%,

b) agglomerating the powder from step a) with at least one hydrocolloid, wherein carbon in elemental form is added during the agglomeration,

c) compacting the agglomerated powder from step b) in a first compacting step to a density of at least 80% of theoretical density, with the proviso that the compacted agglomerated powder still is porous allowing transport of gas to and from its interior,

d) further compacting the compacted agglomerated powder from step c) to a density of more than 98% of theoretical density using at least sintering followed by HVC, high velocity compacting with a ram speed exceeding 5 m/s, wherein a gas comprising carbon is added during sintering.

2. The process according to claim 1, wherein the carbon in elemental form added during the agglomeration in step b) is added in an amount corresponding to the difference between the carbon content in the carbon-containing steel in step a) and the desired carbon content of the finished steel after step d).

3. The process according to any one of claims 1-2, wherein the carbon in elemental form in step b) is added in the form of graphite.

4. The process according to any one of claims 1-3, wherein the carbon in elemental form in step b) is added in the form of a carbon powder.

5. The process according to any one of claims 1-4, wherein the carbon in elemental form in step b) is added in the form of particles with an average size in the interval 0.1-10 µm.

6. The process according to any one of claims 1-5, wherein the carbon in elemental form in step b) is added in the form of a colloid of insoluble particles comprising carbon suspended throughout a liquid.

7. The process according to any one of claims 1-6, wherein the carbon-containing steel prior to gas atomization in step a) is treated so that the carbon content is reduced to a carbon content less than 0.10wt%.

8. The process according to any one of claims 1-7, wherein the carbon-containing steel prior to gas atomization in step a) is treated so that the carbon content is reduced to a carbon content less than 0.050wt%.

9. The process according to any one of claims 1-8, wherein the carbon-containing steel prior to gas atomization in step a) is treated with argon oxygen decarburization.

10. The process according to any one of claims 1-9, wherein the carbon-containing steel prior to gas atomization in step a) is treated in a vacuum furnace.

11. The process according to any one of claims 1-10, wherein the carbon-containing steel is hardeneble after step c).

12. The process according to any one of claims 1-11, wherein the gas comprising carbon is methane.

13. A full dense component manufactured according to any one of claims 1-12, with a density of more than 98% of the theoretical density, wherein the component comprises a carbon-containing steel.

14. The full dense component according to claim 13, wherein the density is more than 99% of the theoretical density.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2016/053852

A. CLASSIFICATION OF SUBJECT MATTER

INV. B22F3/08 B22F3/16 B30B11/02 C22C38/00 B22F3/087
B22F3/10 C22C38/02

ADD. B30B11/00 B22F9/08 C21D3/04 C23C8/20 C23C8/64

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B22F B30B C22C C23C C21D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2010/080064 A1 (METEC POWDER METAL AB [SE]; AASLUND CHRISTER [SE]) 15 July 2010 (2010-07-15)	13,14
Y	paragraphs [0013], [0079], [0087]; claims 1-18; example 3 -----	1-12
X	US 2011/256015 A1 (SLUND CHRISTER [SE]) 20 October 2011 (2011-10-20) paragraphs [0003], [0061], [0069], [0071] - [0072]; claims 1-3,6; examples 2,8 paragraphs [0084] - [0086], [0100] -----	13,14
X	US 4 632 702 A (DIGIAMBATTISTA VINCENT N [US]) 30 December 1986 (1986-12-30) claims 1-13 ----- -/--	13,14

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search 10 May 2016	Date of mailing of the international search report 18/05/2016
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Aliouane, Nadir

3

International application No
PCT/EP2016/053852

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2008/202651 A1 (UENOSONO SATOSHI [JP] ET AL) 28 August 2008 (2008-08-28) paragraphs [0079] - [0080], [0089] - [0090], [0094], [0101] - [0109], [0134]; claims 12,17,21 abstract -----	1-12
Y	EP 1 049 553 A1 (HOEGANAES AB [SE]) 8 November 2000 (2000-11-08) paragraphs [0001], [0008], [0012], [0020], [0023]; claims 1,4,5,8 -----	9

3

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2016/053852

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 2010080064 A1	15-07-2010	EP 2376247 A1	19-10-2011
		US 2011262763 A1	27-10-2011
		US 2015239045 A1	27-08-2015
		WO 2010080064 A1	15-07-2010
US 2011256015 A1	20-10-2011	CN 102271841 A	07-12-2011
		EP 2376248 A1	19-10-2011
		JP 5697604 B2	08-04-2015
		JP 2012515258 A	05-07-2012
		SE 0950007 A1	13-07-2010
		US 2011256015 A1	20-10-2011
		WO 2010080063 A1	15-07-2010
US 4632702 A	30-12-1986	NONE	
US 2008202651 A1	28-08-2008	US 2008202651 A1	28-08-2008
		WO 2006057434 A1	01-06-2006
EP 1049553 A1	08-11-2000	AU 2446799 A	09-08-1999
		BR 9907146 A	24-10-2000
		CA 2318214 A1	29-07-1999
		DE 69909966 D1	04-09-2003
		DE 69909966 T2	29-01-2004
		EP 1049553 A1	08-11-2000
		ES 2199545 T3	16-02-2004
		JP 2002501123 A	15-01-2002
		US 6355087 B1	12-03-2002
		WO 9937425 A1	29-07-1999