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(54) **Method of making a cemented carbide or cermet powder by using a resonant acoustic mixer**

Verfahren zur Herstellung von zementiertem Karbid oder Cermet-Pulver durch Verwendung eines akustischen Resonanzmischers

Procédé de fabrication de carbure cimenté ou poudre de cermet par un mélangeur acoustique résonnant

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Description

[0001] The present relates to a method of making a cemented carbide or cermet powder where the powder constituents are subjected to a non-milling mixing operation by using an acoustic mixer.

Background

[0002] Cemented carbide and cermet powders used for making sintered bodies for e.g. cutting tools, wear parts etc. are usually made by first forming a slurry by milling the powder constituents together with binder metal powders, organic binder (e.g. polyethylene glycol) and a milling liquid in either a ball mill or an attritor mill for several hours. The slurry is then usually subjected to a spray drying operation to form granulated cemented carbide or cermet powders which can be used to press green parts that are subsequently sintered.

[0003] The main purpose of the milling operation is to obtain a good binder phase distribution and good wettability between the hard constituent grains and the binder phase powder. A good binder phase distribution and good wettability is essential for achieving cemented carbide and cermet materials of high quality. If the phase distribution or wettability is poor, pores and cracks will be formed in the final sintered body which is detrimental for the material. However, obtaining a good binder phase distribution and wettability is very difficult for these types of materials and requires a high input of energy, i.e. quite long milling times, usually 10-40 hours depending on the type of mill used and/or the grade produced.

[0004] Ball mills and attritor mills both provide good, homogenous mixing of the powder constituents, binder metal powders and the organic binder. These processes provides a large energy input that can overcome the static friction and binding forces that is required to obtain a good binder phase distribution and good wettability. However, such mills will subject the powders to a milling operation. Hence, the powders, both hard constituent powders and binder metal powders, will partly be grinded so that a fine fraction will be formed. This fine fraction can cause uncontrolled grain growth during the subsequent sintering. Hence, narrow sized raw material can be destroyed by milling.

[0005] It is difficult to produce well controlled narrow grain size microstructures since the milling produce a fine fraction that contribute to an uncontrolled grain growth during sintering.

[0006] Several attempts have been done to solve this problem. One method designed to obtain a powder comprising a coarse grained WC with a good binder phase distribution, is to deposit a salt, e.g. cobalt acetate, onto the WC-particles, then subjecting the coated WC grains to an elevated temperature thus reducing the cobalt acetate to cobalt. By doing this prior to milling, a good cobalt distribution can be obtained at a reduced grinding time. These types of processes are quite complicated and time consuming. One example of this type of process is described in EP752921B1.

[0007] Other types of non-milling mixing methods have also been tested with the aim to avoid the grinding of the powders and thus maintaining properties like grain size of the raw materials.

[0008] EP 1 900 421 A1 discloses a process where the slurry is homogenized in a mixer comprising a rotor, a dispersing device and means to circulate the slurry. The dispersion device contains moving parts.

[0009] WO 02/06545 discloses superhard filler hardmetal having a porosity rating of substantially A06, B00, C00 or better. The superhard filler hardmetal is formed by mechanically mixing (including ultrasonic mixing), shaping the mixture into a green body and consolidating the green body at a preselected temperature, superatmospheric pressure and time at temperature and time at superatmospheric pressure sufficient to form the superhard filler hardmetal.

[0010] EP 1231288 discloses a method of manufacturing composite material. The method comprises a step of ultrasonic mixing.

[0011] One object of the present invention is to obtain a homogenous powder blend without milling.

[0012] Another object of the present invention is to obtain a powder blend where the grain size distribution of the raw materials can be maintained while still obtaining a homogenous powder blend.

[0013] Another object of the present invention is to obtain a powder blend using a mixing process that does not contain any moving parts and is subjected to a minimum amount of wear.

Detailed description of the present invention

[0014] The present invention relates to the method defined in claim 1. This is a method of making a cemented carbide or cermet agglomerated powder without milling where the powder constituents are subjected to a non-milling operation, comprising the steps of first forming a slurry of one or more powders forming hard constituents, wherein said one or more powders forming the hard constituents is selected from borides, carbides, nitrides or carbonitrides of metals from groups 4, 5 and 6 of the periodic table metal binder powders and a mixing liquid. Then the slurry is mixed and dried to form an agglomerated powder. The mixing is done in a non-contact mixer wherein acoustic waves achieving resonance conditions are utilized. Those types of mixers are usually called resonant acoustic mixers.

[0015] Acoustic mixers are known in the art, see e.g. WO 2008/088321 and US 7,188,993. Such mixers use low-frequency, high intensity sound energy for mixing. They have shown good results when mixing fragile organic compounds

but also other types of materials have been mixed. Acoustic mixers are non-contact mixers, i.e. they do not contain any mechanical means for mixing such as stirrers, baffles or impellers. Instead, the mixing is performed by creating micro-mixing zones throughout the entire mixing vessel by mechanical resonance applied to the materials to be mixed by the propagation of an acoustic pressure wave in the mixing vessel.

5 **[0016]** In the method according to the present invention, the grain size of the powders forming hard constituents depends on the application for the alloy and is preferably from 0.2 to 30 μm . If not otherwise specified, all amounts in wt% given herein are the wt% of the total dry weight of the dry powder constituents.

10 **[0017]** The binder metal powders can either be in a powder of one single binder metal, or a powder blend of two or more metals, or a powder of an alloy of two or more metals. The binder metals are selected from Cr, Mo, Fe, Co or Ni, preferably from Co, Cr or Ni. The grain size of the added binder metal powders is suitably between 0.5 to 3 μm , preferably between 0.5 to 1.5 μm .

[0018] When the method according to the present invention relates to making a cemented carbide powder, it is herein meant that cemented carbide is a WC-Co based powder, which also can contain, in addition to WC and Co, additions such as grain growth inhibitors, cubic carbides etc. commonly used in the art of making cemented carbides.

15 **[0019]** In one embodiment of the present invention, a cemented carbide powder is made of hard constituents suitably comprising WC with a grain size of between 0.5 to 2 μm , preferably between 0.5 to 0.9 μm . The binder metal content is suitably between 3 to 17 wt%, preferably 5 to 15 wt% of the total dry weight of the dry powder constituents. Cemented carbides made from these powders are commonly used in cutting tools such as inserts, drills end-mills etc.

20 **[0020]** In one embodiment of the present invention, a cemented carbide powder is made of hard constituents suitably comprising WC having a grain size between 1 to 8 μm , preferably between 1.5 to 4 μm . The binder metal content is suitably between 3 to 30 wt%, preferably 5 to 20 wt% of the total dry weight of the dry powder constituents. Cemented carbides made from these powders are commonly used in tool forming tools and wear parts, e.g. buttons for drillbits mining or asphalt milling hot rolls, parts for mining applications, wire drawing etc.

25 **[0021]** In one embodiment of the present invention, a cemented carbide powder is made of hard constituents suitably comprising WC having a grain size between 4 to 25 μm , preferably between 4.5 to 20 μm . The binder metal content is suitably between 3 to 30 wt%, preferably 6 to 30 wt% of the total dry weight of the dry powder constituents. Cemented carbides made from these powders are commonly used in buttons for drillbits, mining or asphalt milling, hot rolls.

30 **[0022]** In another embodiment of the present invention the method relates to making a cermet powder. By cermet powder is herein meant a powder where the hard constituents comprising large amounts of TiCN and/or TiC. Cermets comprise carbonitride or carbide hard constituents embedded in a metallic binder phase. In addition to titanium, group VIa elements, such as Mo, W and sometimes Cr, are added to facilitate wetting between binder and hard constituents and to strengthen the binder by means of solution hardening. Group IVa and/or Va elements, i.e., Zr, Hf, V, Nb and Ta, can also be added in commercial alloys available today. All these additional elements are usually added as carbides, nitrides and/or carbonitrides. The grain size of the powders forming hard constituents is usually $<2 \mu\text{m}$.

35 **[0023]** An organic binder is also optionally added to the slurry in order to facilitate the granulation during the following spray drying operation but also to function as a pressing agent for any following pressing and sintering operations. The organic binder can be any binder commonly used in the art. The organic binder can e.g. be paraffin, polyethylene glycol (PEG), long chain fatty acids etc. The amount of organic binder is suitably between 15 and 25 vol% based on the total dry powder volume, the amount of organic binder is not included in the total dry powder volume.

40 **[0024]** To form a slurry a mixing liquid is required. Any liquid commonly used as a milling liquid in conventional cemented carbide manufacturing can be used. The milling liquid is preferably water, alcohol or an organic solvent, more preferably water or a water and alcohol mixture and most preferably a water and ethanol mixture. The properties of the slurry are dependent on amount of grinding liquid added. Since the drying of the slurry requires energy, the amount of liquid should be minimized in order to keep costs down. However, enough liquid need to be added in order to achieve a pumpable slurry and avoid clogging of the system.

45 **[0025]** Also, other compounds commonly known in the art can be added to the slurry e.g. dispersion agents, pH-adjusters etc.

50 **[0026]** Drying of the slurry is preferably done according to known techniques, in particular spray-drying. The slurry containing the powdered materials mixed with the organic liquid and possibly the organic binder is atomized through an appropriate nozzle in the drying tower where the small drops are instantaneously dried by a stream of hot gas, for instance in a stream of nitrogen, to form agglomerated granules. The formation of granules is necessary in particular for the automatic feeding of compacting tools used in the subsequent stage. For small scale experiments, other drying methods can also be used, like pan drying.

55 **[0027]** Green bodies are subsequently formed from the dried powders/granules. Any kind of forming operation known in the art can be used, e.g. injection molding, extrusion, uniaxial pressing, multiaxial pressing etc. If injection moulding or extrusion is used, additional organic binders are also added to the powder mixture.

[0028] The green bodies formed from the powders/granules made according to the present invention, is subsequently sintered according to any conventional sintering methods e.g. vacuum sintering, Sinter HIP, plasma sintering etc.. The

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sintering technique used for each specific slurry composition is preferably the technique that would have been used for that slurry composition when the slurry was made according to conventional methods, i.e. ball milling or attritor milling.

Example 1

[0029] Different slurries of cemented carbide were prepared by blending powders of hard constituents like WC and Cr_3C_2 , Co and PEG with a liquid with an ethanol/water ratio of 90/10 by weight. The WC grain size and the Co grain size given is the Fisher grain size (FSSS). The composition of the dry constituents of the slurries and the properties of the raw material are shown in Table 1. The amount of Co, WC and Cr_3C_2 given in wt% are based on the total dry powder constituents in the slurry. The amount of PEG is based on the total dry powder constituents of the slurry, where the amount of PEG is not included into the dry powder constituents of the slurry.

Table 1

Slurry	Co (wt%)	Co (μm)	Cr_3C_2 (wt%)	WC (μm)	PEG wt%
Composition 1	10.0	0.5	0.5	0.8	2
Composition 2a	6.0	0.5	-	2.5	2
Composition 2b	6.0	0.5	-	5	2
Composition 3a	6.3	0.9	-	5	2
Composition 3b	6.0*	0.9	-	5*	2

* Approximately 2 wt% of the cobalt originates from the WC powder which has been coated with Co by sol-gel technique as described in EP752921B1.

Example 2

[0030] The slurry with Composition 1 from Example 1 were then subjected to a mixing operation either using a Resodyn Acoustic Mixer (LabRAM) according to the invention or a conventional paint shaker (Natalie de Lux), the slurries were then pan dried at 90°C. The mixing conditions are displayed in Table 2.

Table 2

Powders	Composition	Mixer	Mixing time (s)	Energy (G)
Invention 1	Composition 1	RAM	300	95
Comparison 1	Composition 1	Natalie	300	N/A

[0031] The powders were then first subjected to a conventional uniaxial pressing operation forming a green body which is subsequently subjected to a Sinter HIP operation at a sintering temperature of 1410°C.

[0032] The properties of the sintered material made from the powders are displayed in Table 3. As an additional comparison a slurry with Composition 1 made according to conventional techniques is included as Reference 1. The Reference 1 sample has been made according by first making a slurry through ball milling for 56 hours and then subjecting them to a spray drying operation. The powder was then pressed and sintered in the same way as the other samples. The average grain size for fine grained WC is not that affected by the ball milling. Where two values have been given, those represent measurements done on two different pieces from the same sintering batch.

Table 3

Powders	Density (g/cm^3)	Com	Hc (kA/m)	Porosity	HV3
Invention 1	14.47/14.46	8.06/8.03	18.76/18.77	A00,B00,C00	1676/1706
Comparison 1	14.11/14.32	8.30/7.69	18.97/18.50	A00,B00,C00 Co pools	1643/1701
Reference 1	14.48	8.5	20.4	A00,B00,C00	1650

[0033] As can be seen in Table 3, the cemented carbide made according to the invention obtains about the same properties as the Comparison 1 and the Reference 1 samples.

Example 3

[0034] The slurry with Composition 2a from Example 1 were subjected to a mixing operation either using a Resodyn Acoustic Mixer (LabRAM) or a conventional paint shaker (Natalie de Lux), the slurries were then pan dried at 90°C. The mixing conditions are displayed in Table 4.

Table 4

Powders	Composition	Mixer	Mixing time (s)	Energy (G)
Invention 2	Composition 2a	RAM	300	95
Comparison 2	Composition 2a	Natalie	300	N/A

[0035] The powders were then pressed and sintered in the same way as the samples in Example 2.

[0036] The properties of the sintered material made from the powders are displayed in Table 5. As a comparison a slurry with Composition 2b is included as Reference 2. The Reference 2 sample has been made from Composition 2b according to conventional techniques, i.e. ball milling for 20 hours and then subjecting them to a spray drying operation. The powder was then pressed and sintered in the same way as the other samples. The WC grain size prior to the ball milling step is 5 μm. The WC grain size is then drastically reduced by the milling operation. After the sintering step the WC grain size is approx. 2.7 μm. All values given herein on the WC grain size as measured on the sintered material is estimated from the Hc value.

Table 5

Powders	Density (g/cm ³)	Com	Hc (kA/m)	porosity	HV3
Invention 2	15.00/14.98	5.30/5.36	9.90/9.81	A00,B00,C00	1408/1536
Comparison 2	14.79/14.77	5.36/5.34	9.76/9.77	A00,B00,C00 Co pools	1419/1502
Reference 2	14.95	5.7	11.7	N/A	1430

[0037] As can be seen in Table 5, the cemented carbide made according to the invention obtains about the same properties as the Comparison 2 and Reference 2 samples. Also, for Invention 2 the narrow WC grain size distribution of the WC raw material is maintained in the sintered structure. This can be seen in Fig. 1 which shows a SEM-image (Scanning Electron Microscope) of Invention 1. Figure 2 is showing a LOM-image (Light Optic Microscope) of the Reference 2 sample which clearly is affected by the milling which can be seen by the presence of a number of larger grains originating from the grain growth of the fine fraction of WC grains.

Example 4

[0038] The slurry with composition 3a from Example 1 were subjected to a mixing operation either using a Resodyn Acoustic Mixer (LabRAM) the slurry were then pan dried at 90°C. The mixing conditions are displayed in Table 6.

Table 6

Powders	Composition	Mixer	Mixing time (s)	Energy (G)
Invention 3	Composition 3a	RAM	300	95

[0039] The powders were then pressed and sintered in the same way as the samples in Example 2 and 3.

[0040] The properties of the sintered material made from the powders are displayed in Table 7. As a comparison, a slurry with composition 3b is included as Reference 3. The Reference 3 sample has been made by wet mixing the powders and then subjecting them to a spray drying operation. The powder was then pressed and sintered in the same way as the other samples.

Table 7

Powders	Density (g/cm ³)	Com	Hc (kA/m)	porosity	HV30
Invention 3	14.97	5.72	5.65	A02,B00,C00	1240

(continued)

Powders	Density (g/cm ³)	Com	Hc (kA/m)	porosity	HV30
Reference 3	14.95	5.7	6.8	<A02	1280

[0041] As can be seen in Table 7, the cemented carbide made according to the invention obtains about the same properties as the Comparison 3 and Reference 3 samples. Also, it can be seen that about the same properties can be obtained for the Invention 3 where the WC is uncoated compared to Reference 3, where the WC has been coated with Co with use of the complex and expensive sol-gel process.

[0042] As a conclusion, the Examples show that the method according to the present invention can lead to products having the same properties as products been produced with conventional methods. Hence, considerable shorter milling times can be achieved leading to a decrease in energy consumption. Also, the complex sol-gel process commonly used for can be avoided.

Claims

1. A method of making a cemented carbide or cermet agglomerated powder without milling, where the powder constituents are subjected to a non-milling mixing operation, comprising the steps of:

forming a slurry of one or more powders forming hard constituents, metal binder powders and a mixing liquid, wherein said one or more powders forming the hard constituents is selected from borides, carbides, nitrides or carbonitrides of metals from groups 4, 5 and 6 of the periodic table;

mixing and drying said slurry, to form an agglomerated powder, wherein the mixing is done in a resonant acoustic mixer, being a non-contact mixer, wherein acoustic waves achieving resonance conditions are utilized.

2. The method according to claim 1 wherein the slurry contains an organic binder.

3. The method according to any of the preceding claims wherein the drying is performed by spray drying.

4. The method according to any of the preceding claims wherein the metal binder powders is one or more selected from Cr, Mo, Fe, Co or Ni.

5. The method according to any of the preceding claims wherein a cemented carbide powder is made.

6. The method according to claim 5 wherein the hard constituents comprises WC with a grain size of between 0.5 to 2 μm , and a binder metal content between 3 to 17 wt%.

7. The method according to claim 5 wherein the hard constituents comprises WC having a grain size between 1 to 8 μm and a binder metal content between 3 to 30 wt%.

8. The method according to claim 5 wherein the hard constituents comprises WC having a grain size between 4 to 25 μm and a binder metal content between 3 to 30 wt%.

9. The method according to any of claims 1-4 wherein a cermet powder is made.

Patentansprüche

1. Verfahren zum Herstellen einer Pulveragglomeration aus Hartmetall (Sintercarbide) oder Cermet ohne Mahlen, wobei die Pulverbestandteile einem Mischvorgang ohne mahlen unterzogen werden, mit den Schritten:

Bilden einer Aufschlämmung aus einem oder mehreren Pulvern, welche die harten Bestandteile bilden, Metallbinderpulvern und einer Mischflüssigkeit, wobei die eine oder mehreren Pulver, welche die harten Bestandteile bilden, aus Boriden, Carbiden, Nitriden oder Carbonitriden aus Metallen der Gruppen 4, 5 und 6 des Periodensystems ausgewählt werden,

Mischen und Trocknen der Aufschlämmung, um eine Pulveragglomeration zu bilden, wobei die Mischung durch

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einen akustischen Resonanzmischer, der ein kontaktfreier Mischer ist, erfolgt, wobei akustische Wellen, die einen Resonanzzustand einnehmen, verwendet werden.

2. Verfahren nach Anspruch 1, wobei die Aufschlammung ein organisches Bindemittel aufweist.
3. Verfahren nach einem der vorstehenden Ansprüche, wobei das Trocknen durch Sprühtrocknung erfolgt.
4. Verfahren nach einem der vorstehenden Ansprüche, wobei die Pulver eines metallischen Binders eines oder mehrere Pulver ausgewählt aus Cr, Mo, Fe, Co oder Ni ist beziehungsweise sind.
5. Verfahren nach einem der vorstehenden Ansprüche, wobei ein Sintercarbidgepulver hergestellt wird.
6. Verfahren nach Anspruch 5, wobei die harten Bestandteile WC mit einer Korngröße zwischen 0,5 und 2 μm und einen Bindermetallgehalt zwischen 3 und 17 Gewichtsprozent aufweisen.
7. Verfahren nach Anspruch 5, wobei die harten Bestandteile WC eine Korngröße zwischen 1 und 8 μm und einen Bindermetallgehalt zwischen 3 und 30 Gewichtsprozent aufweisen.
8. Verfahren nach Anspruch 5, wobei die harten Bestandteile WC mit einer Korngröße zwischen 4 und 25 μm und mit einem Bindermetallgehalt zwischen 3 und 30 Gewichtsprozent aufweisen.
9. Verfahren nach einem der Ansprüche 1 bis 4, wobei ein Cermet-Pulver hergestellt wird.

Revendications

1. Procédé de fabrication d'une poudre agglomérée de carbure cémenté ou de cermet sans broyage, dans lequel les constituants de la poudre sont soumis à une opération de mélange sans broyage, comprenant les étapes consistant à :

former une bouillie d'une ou plusieurs poudres formant des constituants durs, des poudres de liants métalliques et un liquide de mélange, dans lequel lesdites une ou plusieurs poudres formant les constituants durs sont sélectionnés à partir des borures, des carbures, des nitrures ou des carbonitrides de métaux des groupes 4, 5 et 6 du tableau périodique,
mélanger et sécher ladite bouillie, pour former une poudre agglomérée, dans lequel le mélange est réalisé dans un mélangeur acoustique à résonance, qui est un mélangeur sans contact, dans lequel des ondes acoustiques atteignant des conditions de résonance sont utilisées.
2. Procédé selon la revendication 1 dans lequel la bouillie contient un liant organique.
3. Procédé selon l'une quelconque des revendications précédentes dans lequel le séchage est réalisé par séchage par pulvérisation.
4. Procédé selon l'une quelconque des revendications précédentes dans lequel les poudres de liants métalliques sont une ou plusieurs poudres sélectionnées parmi Cr, Mo, Fe, Co ou Ni.
5. Procédé selon l'une quelconque des revendications précédentes dans lequel une poudre de carbure cémenté est réalisée.
6. Procédé selon la revendication 5 dans lequel les constituants durs comprennent du carbure de tungstène WC avec une taille de grain entre 0,5 et 2 μm , et une teneur en métal de liant entre 3 et 17 % en poids.
7. Procédé selon la revendication 5 dans lequel les constituants durs comprennent du carbure de tungstène WC avec une taille de grain entre 1 et 8 μm , et une teneur en métal de liant entre 3 et 30 % en poids.
8. Procédé selon la revendication 5 dans lequel les constituants durs comprennent du carbure de tungstène WC avec une taille de grain entre 4 et 25 μm , et une teneur en métal de liant entre 3 et 30 % en poids.

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9. Procédé selon l'une quelconque des revendications 1 à 4 dans lequel une poudre de cermet est réalisée.

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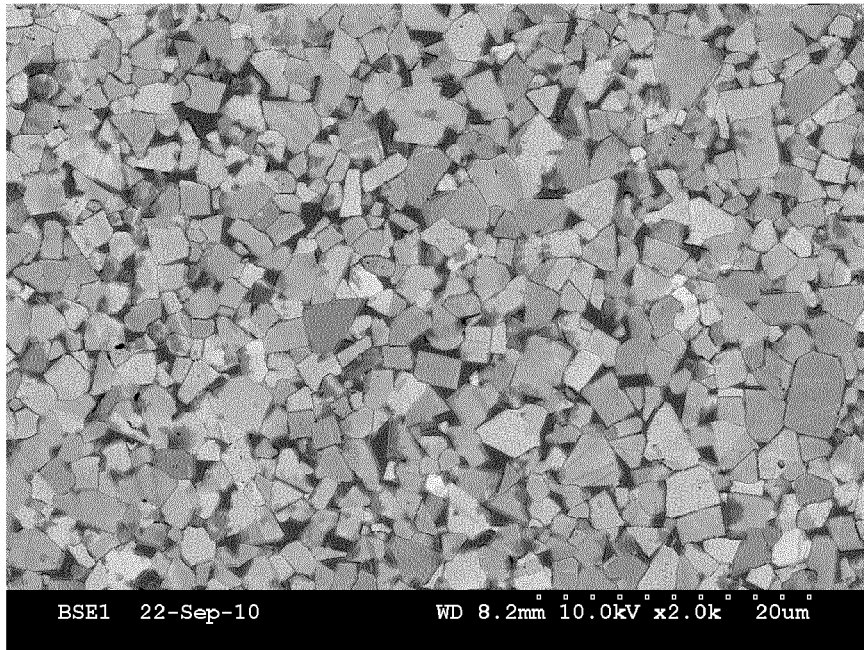
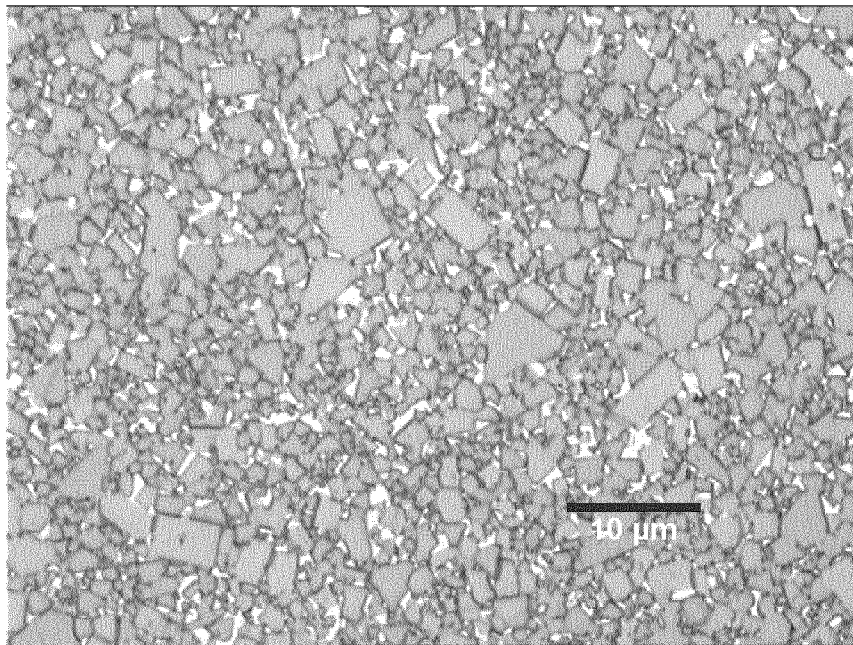


Fig. 2



REFERENCES CITED IN THE DESCRIPTION

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