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(72) Inventors:
• **Fais, Alessandro**
10129 Torino (TO) (IT)
• **Bonetti, Serena**
10129 Torino (TO) (IT)

(71) Applicant: **EPoS S.r.L.**
10129 Torino (IT)

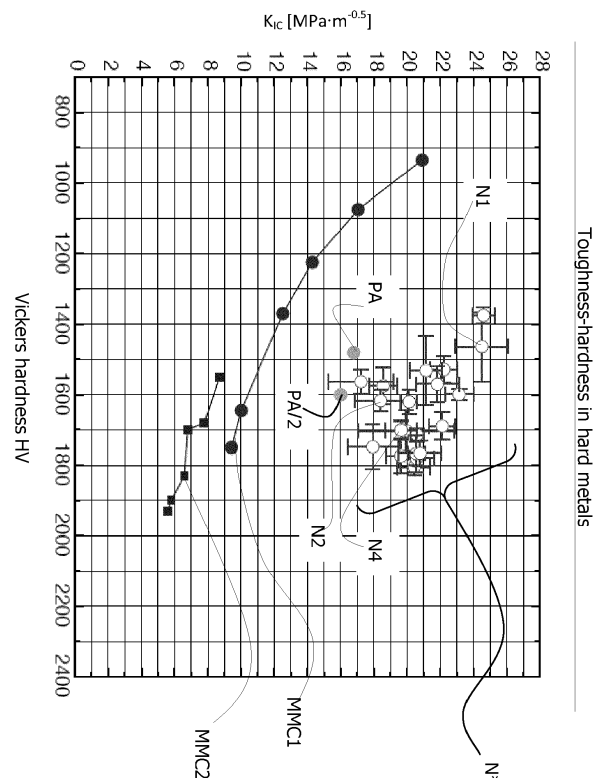
(74) Representative: **Gallarotti, Franco**
Buzzi, Notaro & Antonielli d'Oulx S.r.l.
Via Maria Vittoria, 18
10123 Torino (IT)

(54) **A method for sintering metal matrix composite materials**

(57) Described herein is a method for sintering a composite material including at least one metal phase and at least one ceramic phase. The method comprises the following steps:

- providing the at least one metal phase in the form of powders of pre-alloyed metal material;
 - providing the at least one ceramic phase in the form of powders of ceramic material;
 - mixing the powders of the at least one metal phase and of the at least one ceramic phase; and
 - sintering the mixed powders for a time interval of between 10^{-4} s and 60 s,
- wherein one or more metal phases have a coefficient of thermal expansion of between 0 K^{-1} and $6 \cdot 10^{-6}\text{ K}^{-1}$.

FIG. 4



Description

Field of the invention

[0001] The present invention relates to a method for sintering metal-matrix composite materials and to the corresponding materials that can be obtained by the aforesaid method.

Prior art

[0002] Metal-matrix composite (MMC) materials are very widespread in all applications that are subject to phenomena of abrasion, high temperatures, erosion, and impact, such as for example cutting tools and tools for die-forming of metal materials.

[0003] This is due to their particular combination of properties, in particular:

- hardness values measured according to ISO standard 3878:1983 ranging from 800 to 1900 HV measured at 30 kgf (HV30); and
- toughness values measured according to ISO standard 28079:2009 ranging from 5 to 25 MPa·M^{-0.5}.

[0004] The combination of hardness and toughness is obtained thanks to the combination of the properties of a well-distributed metal phase and of a ceramic phase or even a combination of ceramic phases.

[0005] Traditionally, by reducing the amount of metal within a composite material it is possible to increase the hardness thereof (hence losing toughness), whereas by increasing the amount of metal it is possible to increase the toughness of the composite material (hence losing hardness). An example of said behaviour is clearly visible in Figure 4, wherein the references MMC1 and MMC2 refer to sets of data representative of the performance of materials of a known type as the volume percentages of the metal phase and of the ceramic phase vary.

[0006] High speed steels, which historically have been used up to the advent of cemented carbides as materials for cutting tools, are still today competitive in industrial applications thanks to their high values of toughness combined with a hardness sufficient for a vast number of uses.

[0007] Practically all technical metals and/or transition metals have been used and tested as metal matrices, even though modern-day industry is oriented substantially towards the use of cobalt (thanks to its excellent wettability with the ceramic phase), nickel (thanks to its wettability and resistance to corrosion) or nickel alloys (thanks to their improved wettability with complex ceramic mixtures).

[0008] The ceramic phase usually comprises tungsten carbide (WC) as single component, but also carbides of titanium (TiC), tantalum (TaC), vanadium (VC), molybdenum (Mo₂C), chromium (Cr₃C₂), and hafnium (HfC) are used both as single component or in different com-

binations and proportions thereof.

[0009] Nitrides of transition metals have also found application, especially in combination with carbides, in so-called CERMET materials, such as titanium carbonitride (TiCN).

[0010] Currently, it is known that the best combination of hardness and toughness for metal-matrix composite materials comprises a material having a cobalt matrix and a ceramic reinforcement basically constituted by grains of tungsten carbide that have been stabilised with other carbides or additives, which thus constitute an additional phase.

[0011] This is due to the high intrinsic toughness of tungsten carbide as compared to that of other carbides or nitrides.

[0012] Industrial standards for methods for sintering metal-matrix composite materials for cutting tools entail the following steps:

- i) wet powder milling with process control agents such as polymeric binders and/or lubricants, followed, if necessary, by granulation of the powders themselves;
- ii) pressing or extrusion of the powder to provide a semifinished product (also known as "green");
- iii) de-waxing/pre-sintering and, if necessary, machining of the semifinished product ("green"); and
- iv) sintering at temperatures close to or slightly above the liquidus temperature and, if necessary, hot isostatic pressing (HIP) to remove or at least reduce residual porosities.

[0013] This method is otherwise known as "liquid-phase sintering" and the complete thermal cycle (steps iii) to iv)) may last, including cooling, up to 20 hours in order to reduce or at least prevent the thermal stresses inside the sintered components.

[0014] The presence of a liquid phase is required in known sintering methods for improving the wettability and the diffusion kinetics of the elements that constitute the ceramic phase, thus reducing the cycle time to the bare minimum.

[0015] As a consequence of liquid-phase sintering, the ceramic phase grows in size and assumes a peculiar faceted shape due to the high orientation dependent free energy of the surfaces.

[0016] Alternative methods to the liquid-phase sintering comprise, for example, electric-current assisted sintering (ECAS). Said methods are known for enabling sintering of a high volumic fraction of metal-matrix composite materials in a very short time and with low temperatures thanks to the effect of the electromagnetic field on the diffusivity of the elements.

[0017] In order to achieve a contained sintering time, the ECAS sintering apparatus used must be rapidly heated and cooled progressively, but as fast as possible.

[0018] This causes internal stresses and cracks within the material if this has a traditional composition, i.e., of

the type described previously, not enabling a stable and reliable solution for the industrial production of sintered components using this methodology.

[0019] The increase in the performance of the cutting tools has historically been sought with different means:

- decrease in carbide grain size by addition of grain growth inhibitors;
- decrease of the grain size by controlling the processing conditions;
- decrease of the grain size by a variation of the sintering technique;
- functionalization of the surface gradient so as to have a lower percentage of cobalt on the surface of the cutting tools and a higher percentage of cobalt inside; and
- addition of transformation toughening mechanisms in order to increase the value of the parameter K_{IC} , indicating Palmquist toughness.

[0020] The reduction of the production cost and of the impact on the health of workers (see, for example, the document "Report on carcinogens, background document for Cobalt-Tungsten Carbide: powders and hard metals" - published in 2009) for the production of cemented carbides has been sought through the at least partial substitution of cobalt with iron and nickel. Outside of the tight window within which the content of carbon needs to be controlled in conventional sintering technologies in order to achieve a structure of the type comprising a simple metal matrix and tungsten carbide, the inferior mechanical properties of the materials obtained have never attracted producers and users.

Object of the invention

[0021] The object of the present invention is to overcome the technical problems described previously; namely:

- low toughness with high hardness in metal-matrix composite materials with high volumic fraction of ceramic phase;
- short service life of traditional/conventional metal-matrix composite materials with high ceramic volume fraction in applications for machining with chip removal;
- search for an alternative to cobalt as metal matrix in the production of hard materials for containing the fluctuations of price and the problems of toxicity in the production of cutting tools;
- grain coarsening of the ceramic phase during oven or conduction sintering of metal-matrix composite materials with high ceramic volume fraction; and
- poor repeatability of the methods for high-speed electric-current assisted sintering of metal-matrix composite materials with high ceramic volume fraction.

Summary of the invention

[0022] The objects of the present invention are achieved by a method for the production of metal-matrix composite materials having the features forming the subject of one or more of the ensuing claims, which form an integral part of the technical disclosure provided herein in relation to the invention.

[0023] In particular, the objects of the invention are achieved by a method for sintering a composite material including at least one metal phase and at least one ceramic phase, the method comprising the steps of:

- providing said at least one metal phase in the form of powders of pre-alloyed metal material;
- providing said at least one ceramic phase in the form of powders of ceramic material;
- mixing the powders of said at least one metal phase and of said at least one ceramic phase; and
- sintering the mixed powders for a time interval of between 10^{-4} s and 60 s,

wherein one or more metal phases have a coefficient of thermal expansion of between 0 K^{-1} and $6 \cdot 10^{-6}\text{ K}^{-1}$ at room temperature.

Brief description of the figures

[0024] The invention will now be described with reference to the annexed drawings, which are provided purely by way of non-limiting example and in which:

- Figure 1 illustrates a pair of comparative diagrams of the performance of a cutting tool obtained from a known material and sintered in a known way and of a cutting tool made of a first composite material sintered using the method according to the invention, where each of Figures 1A, 1B refers to a specific cutting speed;
- Figure 2 illustrates a comparative diagram of the performance of a cutting tool obtained from a known material and sintered in a known way and of cutting tools obtained from, respectively, a second and a third composite material sintered using the method according to the invention;
- Figure 3 illustrates a comparative diagram of the performance of a cutting tool obtained from a known material and sintered in a known way and of cutting tools obtained from, respectively, the third material and a fourth material, which are sintered using the method according to the invention; and
- Figure 4 illustrates a comparative diagram of the values of Vickers hardness and of Palmquist toughness of various materials of a known type and of the materials that can be obtained using the method according to the invention.

Detailed description of the invention

[0025] The steps that make up a method for sintering metal-matrix composite materials according to a preferred embodiment of the invention will now be described. The composite material that can be obtained using the method according to the invention comprises at least one metal phase and at least one ceramic phase. A monophase metal, a multiphase metal, a single metal element or a metal alloy can be used as metal phase.

[0026] Providing the at least one metal phase in the form of powders of pre-alloyed metal material (or materials) and providing the at least one ceramic phase, which is also in the form of powders of ceramic material (or materials) are initial steps. By providing the pre-alloyed metal material, the possibility of formation of the final metal phase from a mixture of metal elements during sintering is excluded. This is necessary since, as will be seen hereinafter, on account of the high velocities of the sintering process, it is not possible to form the alloy during sintering.

[0027] The powders of metal material are provided in amounts sufficient to obtain a composite material in which the metal phase has a volume percentage of between 2 vol% and 40 vol%.

[0028] The size of the particles of the powders of the (at least one) metal phase can range from 1 nm to 500 μm and can be mono-modal or multi-modal. It is in general preferable to have all the particles with an average size of less than 50 μm .

[0029] Similar considerations apply to the average size of the particles of the powders of the (at least one) ceramic phase: it can range from 1 nm to 500 μm and can be mono-modal or multi-modal, but it is preferable to have an average size of the particles of less than 5 μm .

[0030] A further constraint concerning the metal phase exists, namely, the coefficient of thermal expansion. The method according to the invention envisages that one or more metal phases have a coefficient of thermal expansion of between 0 K^{-1} and $6 \cdot 10^{-6}\text{ K}^{-1}$ at room temperature. The method according to the invention yields optimal results when each metal phase (in particular, in the case of multiphase metal matrix) has a coefficient of thermal expansion of between 0 K^{-1} and $6 \cdot 10^{-6}\text{ K}^{-1}$ at room temperature. In addition, on the basis of what has been said above, in the case where the metal matrix comprises a single metal phase it is envisaged that this has a coefficient of thermal expansion of between 0 K^{-1} and $6 \cdot 10^{-6}\text{ K}^{-1}$ at room temperature.

[0031] An example of metal phase that satisfies said condition is that of an Fe-Ni alloy with a weight percentage of nickel of between 32 wt% and 42 wt%.

[0032] After preparation of the powders of the metal phase and of the ceramic phase the operations proceed with mixing of the aforesaid powders (of the metal and ceramic phases), possibly combined with a dry or wet grinding operation, which can be performed at the same time as the mixing step or even prior to this. Grinding

becomes necessary in the case where the powders have an average size excessive for the final characteristics that it is intended to bestow on the sintered component.

[0033] In general, the mixing and/or milling operation can be carried out with the contribution of a mixing agent that is in the liquid phase at room temperature and pressure and that is preferably chosen in a group comprising ethanol, alkanes - preferably heptane, hexane - cyclohexane, water, or polymeric mixing agents, for example silicone or silicone-based compounds. In this eventuality, there is envisaged a further step of elimination of the liquid mixing agent from the slurry that is extracted from the milling/mixing apparatus.

[0034] The inventors have further observed that downstream of the step of elimination of the liquid agent a residual percentage of additive of around 1% can be tolerated. In an experiment conducted with the use of glycol and heptane as liquid mixing additives, it has been observed that, by tolerating a residual percentage of 0.5 wt% of glycol and 1 wt% of heptane, the impact on the properties of the sintered material is almost negligible.

[0035] The method according to the invention yields optimal results in the case where the steps of mixing of the powders and the possible steps of milling are carried out in the absence of any process-control polymeric agent that at room temperature is in the solid state, such as wax and paraffin.

[0036] The mixed powders of the (at least one) metal phase and of the (at least one) ceramic phase are then inserted in a mould of sintering equipment. The method according to the invention yields optimal results if the sintering is performed using an electric-current-assisted sintering (ECAS) apparatus.

[0037] The powders within the mould are then subjected to sintering with a duration of the cycle, comprising a heating step and a cooling step, of between 10^{-4} s and 60 s. The result is that of a composite material with a density of between 90% and 100% of the theoretical density (an ECAS method is described in the document No. WO-A-2010/070623 filed in the name of the present applicant). The method according to the invention yields optimal results in the case where the sintering operation is performed in the absence of protective atmosphere (which for these applications is typically a gaseous protective atmosphere).

[0038] Moreover, no further methods of treatment downstream of the ones just mentioned are necessary. The sintered material thus obtained has unprecedented values of toughness with high hardness and presents a better performance in the applications of machining with chip removal as compared to known materials.

[0039] No grain growth inhibitor has been added to the mixture of powders, nor do the compounds obtained using the method according to the invention have ceramic grains of a size equal to that of the starting powders. This is basically due to the short time interval in which sintering develops as compared to the known methods (which are much slower). This moreover leads to the advantage of

preventing formation of undesirable phases due generally to the reaction between the ceramic phase and the metal phase in the case of long sintering times.

[0040] The inventors have moreover found that very satisfactory results in terms of mechanical properties of the sintered composite material are obtained by conducting the heating step in a time range of between 1 ms and 500 ms. Preferably, however, the time interval concerned by the heating step is chosen between 1 ms and 100 ms because the effects just mentioned are further amplified.

[0041] By way of example, it is possible to use as ceramic phase a single phase, for example tungsten carbide (WC), titanium carbide (TiC), titanium nitride (TiN), or titanium carbo-nitride (TiCN), or alternatively a mixture of two or more ceramic phases in different percentages can be provided.

[0042] Generalizing, the usable ceramic phases can comprise at least one carbide of a metal chosen in the group comprising tungsten (WC), titanium (TiC), tantalum (TaC), hafnium (HfC), molybdenum (Mo_2C), niobium (NbC and Nb_2C), zirconium (ZrC), vanadium (VC), chromium (Cr_3C_2).

[0043] In addition, the aforesaid ceramic phases can alternatively comprise also the family of the nitrides of the same metals, i.e., tungsten (WN and WN_2), titanium (TiN), tantalum (TaN), hafnium (HfN), molybdenum (Mo_2N), niobium (NbN), zirconium (ZrN), vanadium (VN), chromium (CrN), or carbo-nitrides $\text{TiC}(1-x)\text{N}_x$ where x is comprised between 0 and 1.

[0044] A first example of mixture of ceramic phases is a mixture comprising 70 vol% of tungsten carbide (WC) and 30 vol% of titanium carbide. A second example is a mixture comprising 50 vol% of tungsten carbide (WC) and 50 vol% of titanium carbo-nitride (TiCN). It is also possible to use a mixture of more than two ceramic phases, such as, for example, a mixture with 50 vol% of tungsten carbide (WC), 30 vol% of titanium carbide (TiC), and 20 vol% of titanium carbo-nitride (TiCN).

[0045] Other examples of mixtures of ceramic phases comprise:

- 50 vol% of tungsten carbide (WC) and 50 vol% of titanium carbide (TiC);
- 30 vol% of tungsten carbide (WC) and 70 vol% of titanium carbide (TiC);
- 30 vol% of tungsten carbide (WC) and 70 vol% of titanium carbo-nitride (TiCN); and
- 70 vol% of tungsten carbide (WC) and 30 vol% of titanium carbo-nitride (TiCN).

[0046] With reference to what has been described above, the method according to the invention will be further illustrated via the description of specific examples of sintering of metal-matrix composite materials, illustrating also the superior performance of said materials as compared to materials obtained with known methods.

EXAMPLE 1

[0047] Metal particles constituted by an Fe-Ni alloy with 42 wt% of nickel and a coefficient of thermal expansion at room temperature of $6 \cdot 10^{-6} \text{ K}^{-1}$ obtained by a process of mechanical alloying and sifted through a 150- μm sieve (102 mesh) are mixed without polymer additives in an industrial mixer with powder of tungsten carbide (WC) and titanium carbide (TiC). The size of the ceramic powders is determined according to the ASTM standard B330-07 and corresponds to FSSS = 1.03 μm for the tungsten carbide and FSSS = 1.9 μm for the titanium carbide.

[0048] The weight percentages of each powder are the following: 9.77 wt% of Fe-Ni alloy with 42 wt% of nickel, 79.53 wt% of tungsten carbide, and 10.7 wt% of titanium carbide.

[0049] 2.90 g of mixture are poured into a mould for an ECAS apparatus of the type described in WO-A-2010/070623 filed in the name of the present Applicant.

[0050] By applying a single 30-ms electromagnetic pulse, cylinders of sintered material with a diameter of 10 mm, a height of $3.26 \pm 0.07 \text{ mm}$, and a density of $11.33 \pm 0.09 \text{ g/cm}^3$ are produced.

[0051] After an accurate preparation of the samples, the Vickers hardness at 30 kgf and the Palmquist toughness are respectively: $1464 \pm 98 \text{ HV30}$ and $24.49 \pm 1.58 \text{ MPa} \cdot \text{m}^{-0.5}$. The grain size of the titanium-carbide phase in the composite material is approximately 2 μm , whilst the grain size of the tungsten-carbide phase is approximately 1 μm .

[0052] The cylinders were ground and polished in order to create a cutting insert with a nose radius of 0.8 mm and an end relief angle of 5° that enables the metal chip to break off during machining.

[0053] By way of illustration, Figure 1A presents a comparison with a similar cutting insert obtained starting from a commercial tool of degree P20 (ISO standard) with a value of Vickers hardness at 30 kgf of $1481 \pm 18 \text{ HV30}$ and a value of Palmquist toughness of $16.78 \pm 0.5 \text{ MPa} \cdot \text{m}^{-0.5}$. The insert was brazed on a tool holder and used on a lathe for machining normalized C40 steel with a Brinell hardness of 210 HB. The cutting speed considered was 120 m/min. The abscissae represent the cutting time expressed in minutes, and the ordinates represent the parameter of wear of the tool VB_B according to ISO 3685, expressed in millimetres. The set of data designated by the reference N1 corresponds to a tool using a composite material corresponding to the one described in this example (which will at times be referred to hereinafter for brevity as "tool N1"), whilst the set of data designated by PA refers to the tool of degree P20 mentioned above (which will at times be referred to hereinafter for brevity as "tool PA"). From the comparison, it is immediately evident that already at low cutting speeds the reduction of wear of the cutting edge is appreciable, with obvious benefits in terms of costs and of reduction of

machine downtime for replacing the tool. Even more eloquent is the subsequent Figure 1B, which illustrates the same comparison between the tool N1 and the tool PA but performed with a cutting speed increased to 140 m/min. It may be noted how, upon overstepping of a cutting time of three minutes, the tool PA shows a wear that is approximately twice that of the cutting edge of the tool N1 made of the material of Example 1. The increase in wear is moreover rather contained as compared to the wear observable on the tool PA, where, assuming as reference the cutting instants corresponding to three minutes and six minutes, there is noted an increase of the wear up to approximately six times the initial value, whilst with the tool N1 the wear presents an increase that reaches approximately three times the initial value.

EXAMPLE 2

[0054] Metal particles constituted by an Fe-Ni alloy with 42 wt% of nickel, with a coefficient of thermal expansion at room temperature of $6 \times 10^{-6} \text{ K}^{-1}$ obtained by a process of mechanical alloying and sifted through a 150- μm sieve (102 mesh) are mixed without polymer additives in an industrial mixer with powder of tungsten carbide (WC) and titanium carbide (TiC). The size of the ceramic powders is determined according to the ASTM standard B330-07 and corresponds to FSSS = 1.9 μm for titanium carbide.

[0055] The weight percentages of each powder are the following: 18.65 wt% of Fe-Ni alloy with 42 wt% of nickel and 81.35 wt% of titanium carbide.

[0056] 1.38 g of mixture are poured into a mould for an ECAS apparatus of the type described in WO-A-2010/070623.

[0057] By applying a single electromagnetic pulse with a heating time of approximately 30 ms, cylinders of sintered material are obtained with a diameter of 10 mm, a height of $3.56 \pm 0.04 \text{ mm}$, and a density of $4.93 \pm 0.04 \text{ g/cm}^3$.

[0058] After an accurate preparation of the specimen, the Vickers hardness at 30 kgf and the Palmquist toughness are $1613 \pm 48 \text{ HV30}$ and $18.82 \pm 3.02 \text{ MPa} \cdot \text{m}^{-0.5}$ respectively. The grain size of the titanium-carbide phase in the composite material is approximately 2 μm .

[0059] Figure 2 is similar to Figures 1A and 1B but comprises in just one diagram four sets of data, namely:

- a first set PA obtained at a cutting speed of 140 m/min, which corresponds to the performance of the tool of a known type of Example 1 at the cutting speed of 140 m/min;
- a second set N1 obtained at a cutting speed of 140 m/min, which corresponds to the performance of the tool N1 at the cutting speed of 140 m/min;
- a third set N2 obtained at a cutting speed of 140 m/min, which corresponds to the performance of a tool using a cutting insert made of a material sintered according to the method of the invention (which will

at times be referred to hereinafter for brevity as "tool N2") and corresponding to the one described in this example (Example 2) at the cutting speed of 140 m/min; and

- a fourth set N2 obtained at a cutting speed of 210 m/min corresponding to the performance of the same tool of the set N2 obtained at a cutting speed of 140 m/min but with a cutting speed brought up to 210 m/min.

The improvement as compared to the tool PA is highly significant, as likewise it is as compared to the tool N1. It should be noted how, even after a long cutting time, the wear parameter VB_B for the tool N2 remains, with both cutting speeds, abundantly below 0.2 mm, whilst the same parameter is well over 0.6 mm already after three minutes and with a cutting speed of 140 m/min for the tool PA.

EXAMPLE 3

[0060] Metal particles constituted by Fe-Ni alloy with 42 wt% of nickel with a coefficient of thermal expansion at room temperature of $6 \times 10^{-6} \text{ K}^{-1}$ obtained by a process of mechanical alloying and sifted through a 150- μm sieve (102 mesh) are mixed without polymer additives in an industrial mixer with powders of tungsten carbide and titanium carbide. The size of the ceramic powders is determined according to the ASTM standard B330-07 and is equal to FSSS = 1.03 μm for the tungsten carbide and FSSS = 1.9 μm for the titanium carbide.

[0061] The weight percentages for each powder are the following: 11.81 wt% of Fe-Ni alloy with 42 wt% of nickel, 67.11 wt% of tungsten carbide, and 21.07 wt% of titanium carbide.

[0062] 3.23 g of mixture are put in a mould for an ECAS apparatus of the type described in WO-A-2010/070623. With a single electromagnetic pulse that heats the powders in approximately 30 ms cylinders of sintered material are obtained with a diameter of 10 mm, a height of $4.09 \pm 0.02 \text{ mm}$, and a density of $9.87 \pm 0.04 \text{ g/cm}^3$.

[0063] The grain size of the tungsten-carbide phase in the composite material is approximately 2 μm , whilst the grain size of tungsten carbide is approximately 1 μm .

EXAMPLE 4

[0064] Metal particles consisting of Fe-Ni alloy at 36 wt% of nickel with a coefficient of thermal expansion at room temperature of $2 \times 10^{-6} \text{ K}^{-1}$ are obtained by a process of atomization and with a maximum size of 45 μm .

[0065] The weight percentages for each powder are the following: $11.5 \pm 0.5 \text{ wt\%}$ of Fe-Ni alloy with 42 wt% of nickel, $67.5 \pm 0.5 \text{ wt\%}$ of tungsten carbide, and $21.5 \pm 0.5 \text{ wt\%}$ of titanium carbide.

[0066] 3.23 g of mixture are put into a mould for an ECAS apparatus of the type described in WO-A-2010/070623. With a single electromagnetic

pulse that heats the powders for approximately 30 ms, cylinders of sintered material are obtained with a diameter of 10 mm, a height of 4.09 ± 0.04 mm, and a density of 9.72 ± 0.07 g/cm³.

[0067] After accurate preparation of the specimen, the Vickers hardness at 30 kgf and the Palmquist toughness are, respectively, 1716 ± 27 HV30 and 20.03 ± 0.64 MPa·m^{-0.5}. The grain size of the tungsten-carbide phase in the composite material is approximately 2 μm, whilst the grain size of tungsten carbide is approximately 1 μm.

[0068] Figure 3 illustrates a comparison of the performance of the tool PA with tools having a cutting insert made of the material described in Example 3 and Example 4, respectively.

[0069] Consequently, there are four sets of data, namely:

- a first set PA obtained at a cutting speed of 140 m/min associated to the tool made of known material and referring to the cutting speed of 140 m/min;
- a second set N3 obtained at a cutting speed of 140 m/min associated to a tool with cutting insert made of the material described in Example 3 (which will at times be referred to hereinafter for brevity as "tool N3") and referring to the cutting speed of 140 m/min;
- a third set N3 obtained at a cutting speed of 210 m/min associated to a tool with cutting insert made of the material described in Example 3 and referring to the cutting speed of 210 m/min; and
- a fourth set N4 obtained at a cutting speed of 140 m/min associated to a tool with cutting insert made of the material described in Example 4 (which will at times be referred to hereinafter for brevity as "tool N4") and referring to the cutting speed of 140 m/min.

[0070] The difference between the material of Example 3 and that of Example 4 lies in the use of atomized powders instead of powders obtained by mechanical milling.

[0071] The values of the wear parameter VB_B settle substantially on values that are close to each other in the case of the tools N3 and N4, in a way somewhat independent of the cutting speed. Even more evident is the gap with respect to the tools obtained with known materials.

[0072] Finally, Figure 4 illustrates a comparative diagram where the abscissae represent the values of Vickers hardness (HV30) and the ordinates represent the values of Palmquist toughness (K_{IC}), the latter being expressed in MPa·m^{-0.5}, for different materials, namely:

- the reference MMC1 designates a set of data corresponding to a first class of metal-matrix composite materials of a known type made up of tungsten and cobalt carbide with variable percentages of cobalt ranging from 13 vol% to 25 vol%;
- the reference MMC2 designates a set of data corresponding to a second class of commercially available metal-matrix composite materials of a known type;

- the reference PA designates a datum corresponding to the pair of values of toughness/hardness for the material for cutting tools of degree P20 used as term of comparison in Examples 1, 2, 3 and 4, described previously;
- the reference PA/2 designates a datum corresponding to the pair of values of toughness/hardness for a further material for cutting tools of a known type; and
- the reference N* designates a cloud of points (with corresponding bands of uncertainty of measurement) corresponding to pairs of values of toughness/hardness of metal-matrix composite materials obtained by means of the method according to the invention and including points corresponding to the materials of Examples 1, 2, 3 and 4 and to other sintered materials not explicitly mentioned herein. In particular, the dots corresponding to Examples 1, 2 and 4 are designated by the references N1, N2 and N4.

[0073] It may be noted how the method according to the invention enables materials to be obtained, the properties of which in terms of combination of toughness and hardness fall in an area of the diagram substantially without data regarding known materials obtained with known methods and where there coexist values of toughness and hardness that are unattainable in combination by the aforesaid known sintered materials using known methods.

[0074] Of course, the details of construction and the embodiments may vary widely with respect to what has been described and illustrated herein purely by way of non-limiting example, without thereby departing from the scope of the invention, as defined in the annexed claims.

Claims

1. A method for sintering a composite material including at least one metal phase and at least one ceramic phase, the method comprising the steps of:
 - providing said at least one metal phase in the form of powders of pre-alloyed metal material;
 - providing said at least one ceramic phase in the form of powders of ceramic material;
 - mixing the powders of said at least one metal phase and of said at least one ceramic phase; and
 - sintering the mixed powders for a time interval of between 10^{-4} s and 60 s,
 wherein one or more metal phases have a coefficient of thermal expansion of between 0 K^{-1} and $6 \cdot 10^{-6} \text{ K}^{-1}$ at room temperature.
2. The method according to Claim 1, wherein each met-

al phase has a coefficient of thermal expansion of between 0 K^{-1} and $6\cdot 10^{-6}\text{ K}^{-1}$ at room temperature.

3. The method according to Claim 1 or Claim 2, wherein said step of mixing the powders of said at least one metal phase and of said at least one ceramic phase is performed in the absence of polymer additives that are in the solid state at room temperature.
4. The method according to any one of the preceding claims, wherein said sintering step is performed by means of an electric-current assisted sintering (ECAS) apparatus.
5. The method according to Claim 1, wherein the sintering step is performed in the absence of a protective atmosphere.
6. The method according to any one of the preceding claims, wherein said sintering step comprises a heating step that is performed for a time interval of between 1 ms and 500 ms, preferably between 1 ms and 100 ms.
7. The method according to any one of Claims 1 to 3, wherein said step of mixing the powders of said at least one metal phase and of said at least one ceramic phase comprises a further step of milling of the powders of said at least one metal phase and of said at least one ceramic phase, said further grinding step being performed at the same time as or prior to said step of mixing the powders of said at least one metal phase and of said at least one ceramic phase.
8. The method according to Claim 7, wherein milling is of the wet type and is performed by means of the contribution of a mixing agent that is in the liquid state at room temperature and pressure and that is preferably chosen in a group comprising ethanol, alkanes, cyclohexane, water, silicone and silicone-based compounds, wherein said alkanes preferably comprise heptane and hexane.
9. The method according to Claim 8, comprising a further step of elimination of said mixing agent performed prior to said step of sintering of said mixed powders, preferably by means of drying.
10. The method according to any one of the preceding claims, wherein said at least one ceramic phase comprises at least one carbide of a metal chosen in a group comprising tungsten, titanium, tantalum, hafnium, molybdenum, niobium, zirconium, vanadium, chromium.
11. The method according to any one of the preceding claims, wherein said at least one ceramic phase comprises at least one nitride of a metal chosen in

a group comprising tungsten, titanium, tantalum, hafnium, molybdenum, niobium, zirconium, vanadium, chromium.

12. The method according to any one of the preceding claims, wherein said at least one ceramic phase comprises titanium carbo-nitride.
13. The method according to Claim 10, wherein said at least one ceramic phase comprises a mixture of a first tungsten-carbide phase and of a second titanium-carbide phase, said first and second phases being present in said mixture preferably in one of the following proportions, alternatively:
 - 70 vol% of tungsten carbide and 30 vol% of titanium carbide;
 - 50 vol% of tungsten carbide and 50 vol% of titanium carbide; and
 - 30 vol% of tungsten carbide and 70 vol% of titanium carbide, respectively.
14. The method according to Claim 12, wherein said at least one ceramic phase comprises a mixture of a first tungsten-carbide phase and of a second titanium-carbo-nitride phase, each of said first and second phases being present in said mixture preferably in one of the following proportions, alternatively:
 - 70 vol% of tungsten carbide and 30 vol% of titanium carbo-nitride;
 - 50 vol% of tungsten carbide and 50 vol% of titanium carbo-nitride;
 - 30 vol% of tungsten carbide and 70 vol% of titanium carbo-nitride;
15. The method according to any one of the preceding claims, wherein the powders of said at least one metal phase has an average size of the particles of between 1 nm and 500 μm , preferably less than 50 μm .
16. The method according to any one of the preceding claims, wherein the powders of said at least one ceramic phase have an average size of the particles of between 1 nm and 500 μm , preferably less than 5 μm .
17. A metal-matrix composite material that can be obtained using a method according to one or more of the preceding claims.

FIG.1A

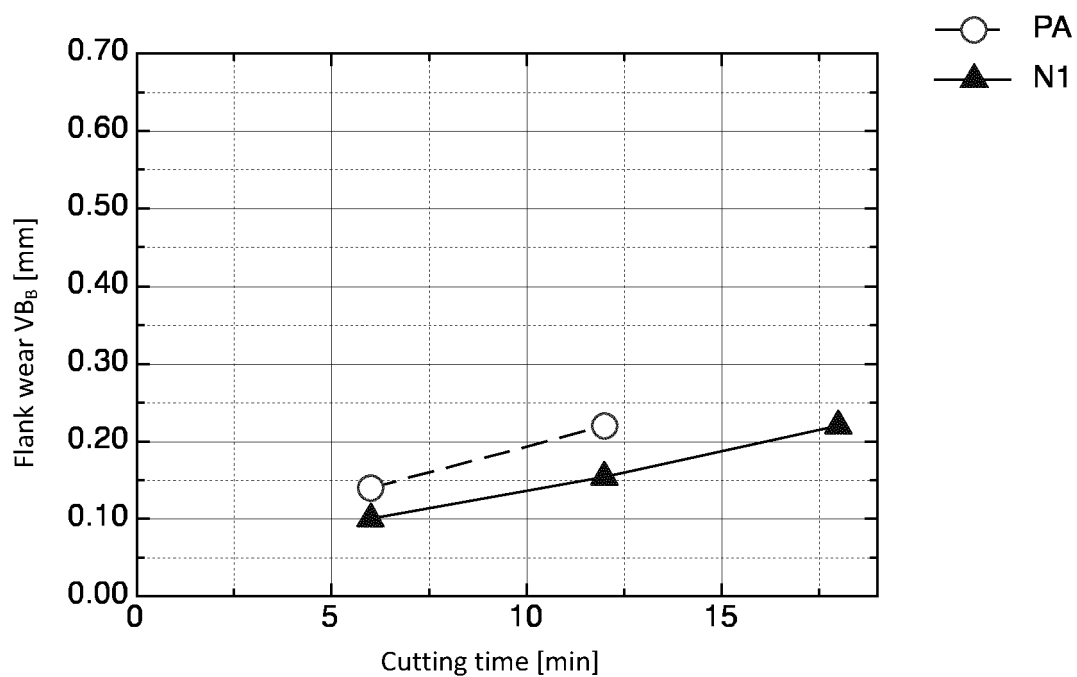


FIG. 1B

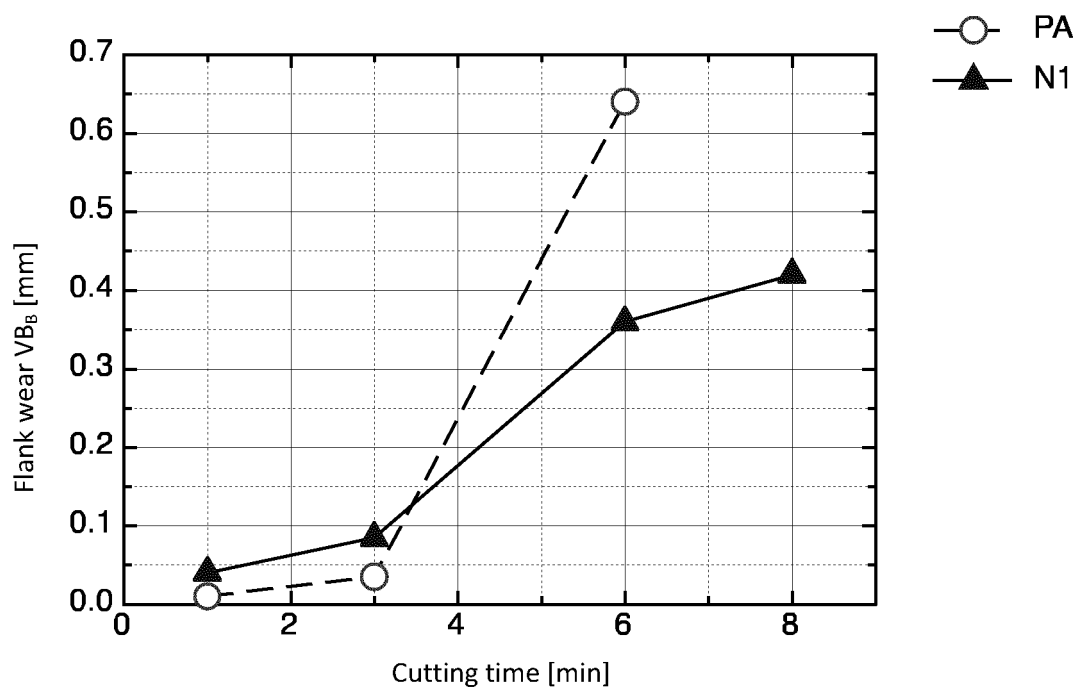


FIG. 2

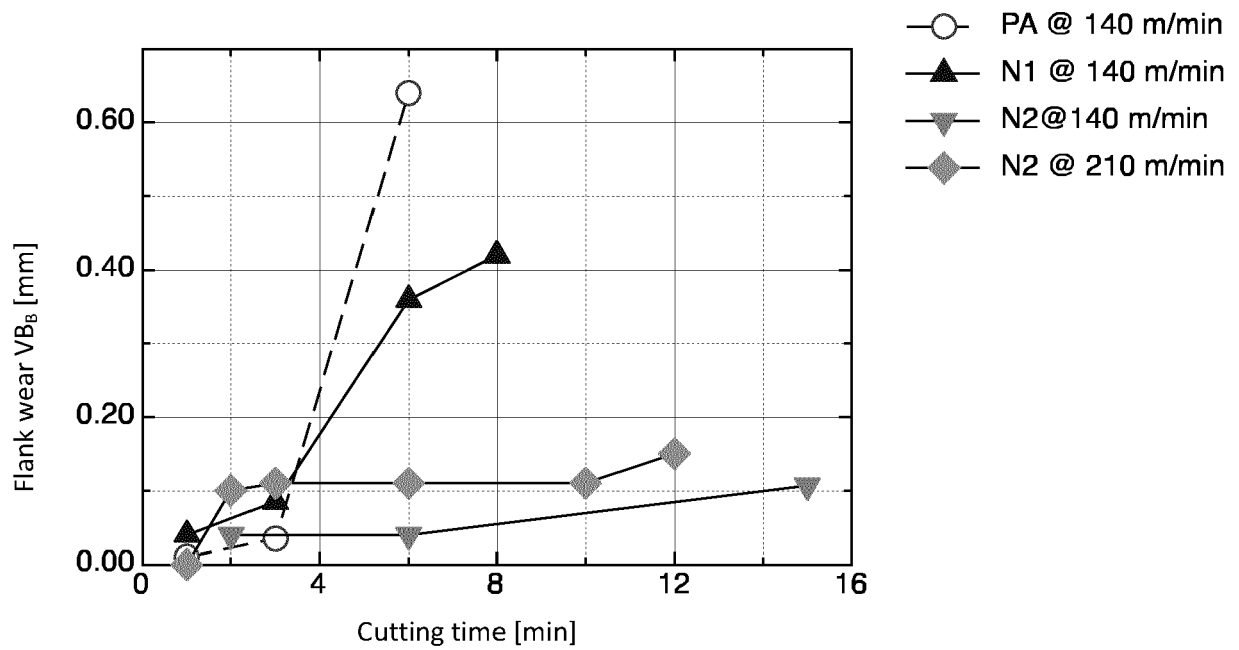


FIG. 3

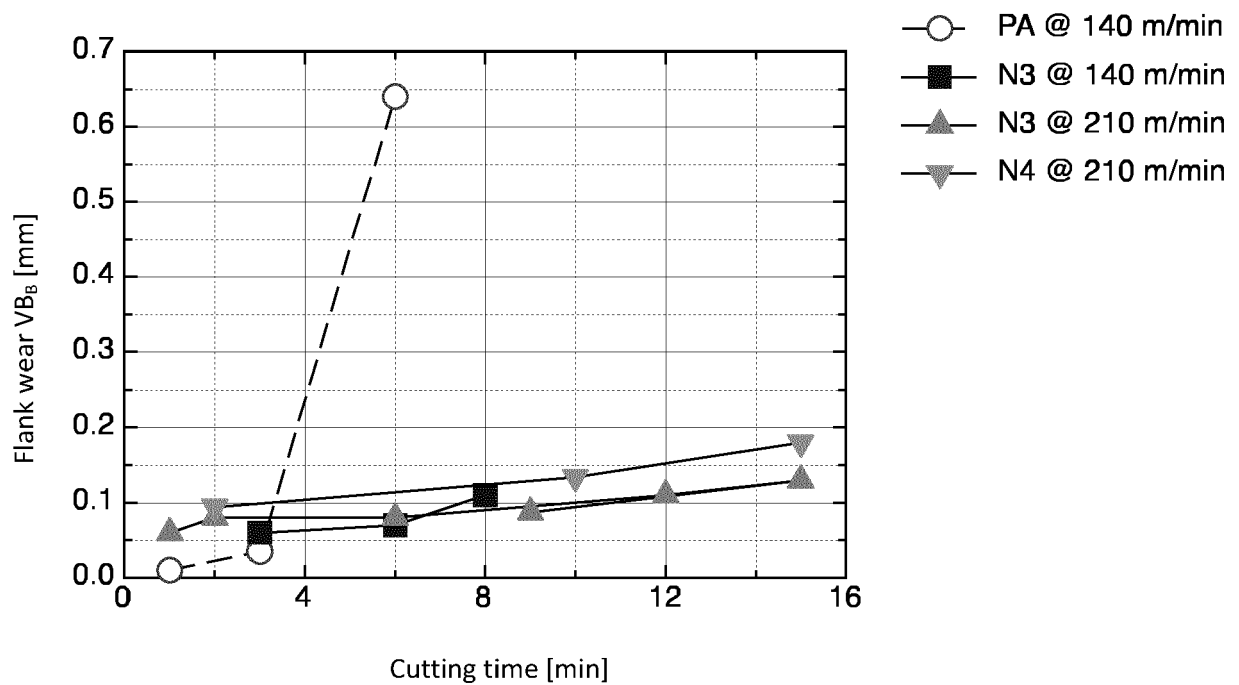
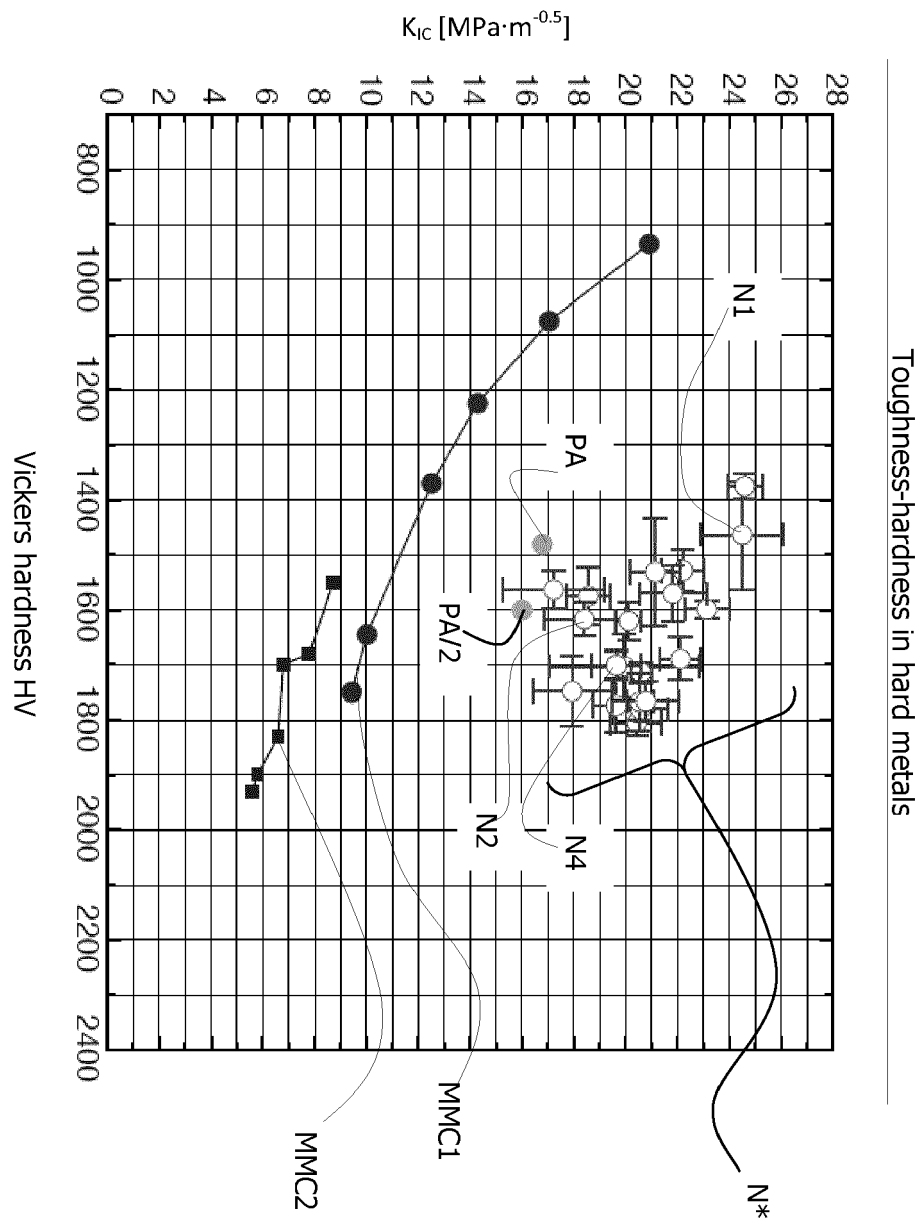


FIG. 4





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Application Number
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Place of search Munich		Date of completion of the search 22 May 2012	Examiner Fortunati, Taddiano
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