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(54) **METHOD FOR PREPARING NANO-SIO₂
REINFORCED ALUMINUM MATRIX
COMPOSITES**

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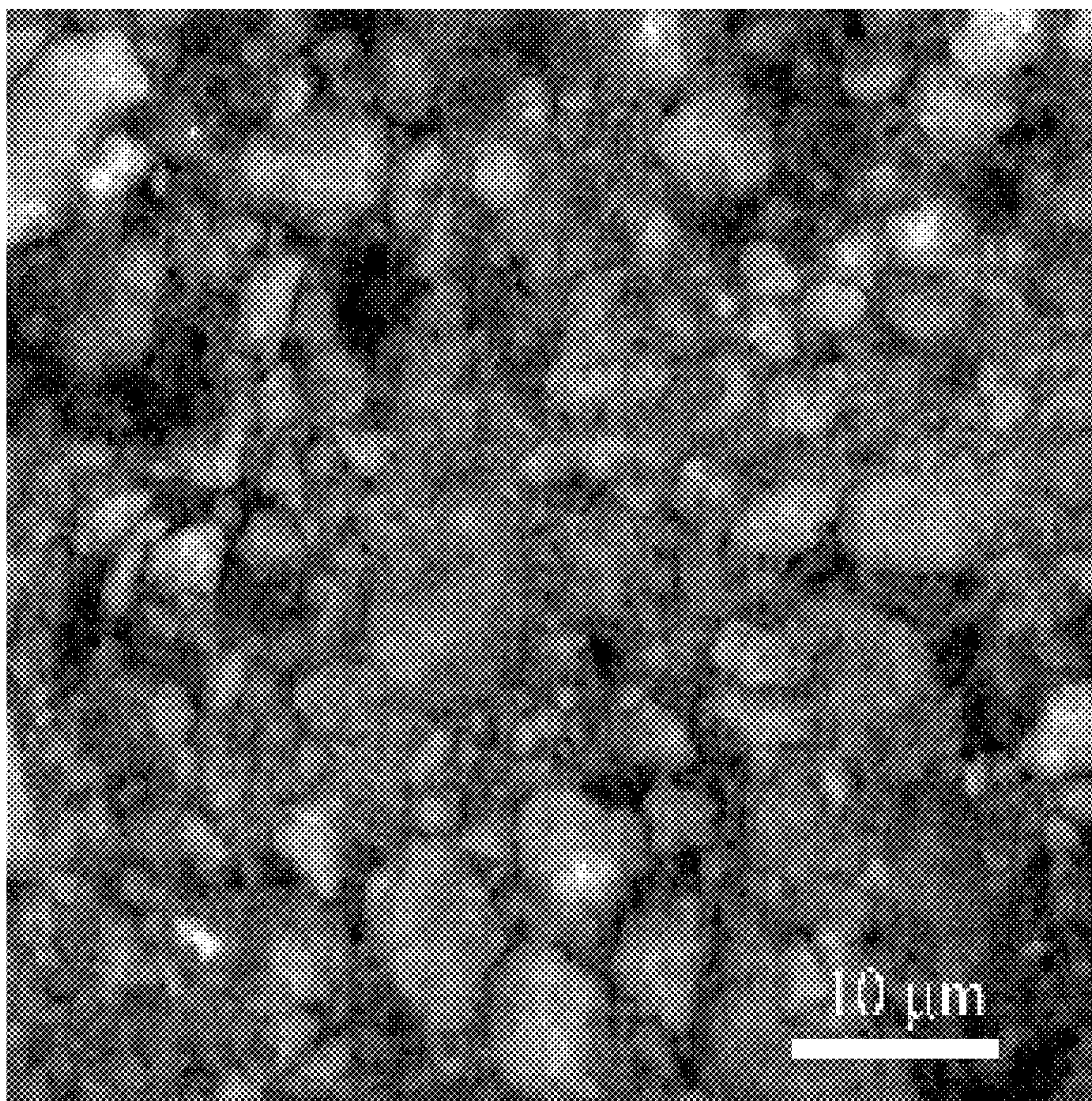
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(57) **ABSTRACT**

A method for preparing nano-SiO₂ reinforced aluminum matrix composites, includes the following: Step-1, powder mixing: mixing aluminum matrix powder with nano-SiO₂ powder to obtain raw material powder, wherein the aluminum matrix powder has an average particle size between 30 μm to 100 μm, the nano-SiO₂ powder has an average particle size between 5 nm to 145 nm, mass percentage of nano-SiO₂ in the raw material powder is 0.01% to 5% and the remaining raw material powder is the aluminum matrix powder; Step-2, shaping: press shaping the powder obtained in the Step-1 to obtain base bodies; Step-3, sintering: sintering the base bodies obtained in the Step-2 in an atmosphere of N₂ at 550° C. to 660° C., preserving the temperature for a period of 5 min to 60 min, and cooling in a furnace at end of the period under protection of N₂ for 0.5 h to 3 h; and Step-4, heat treatment.



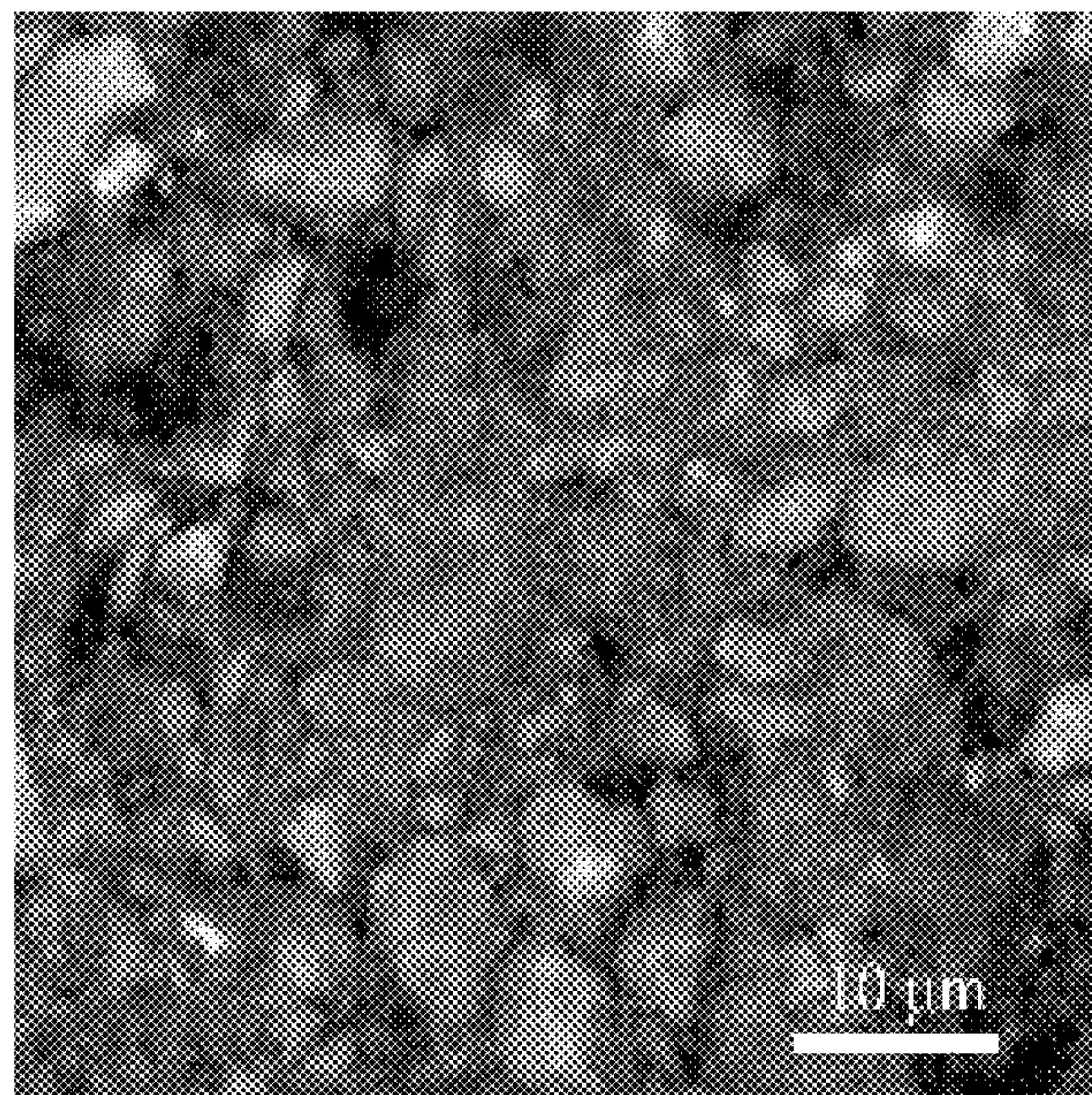


Fig.1

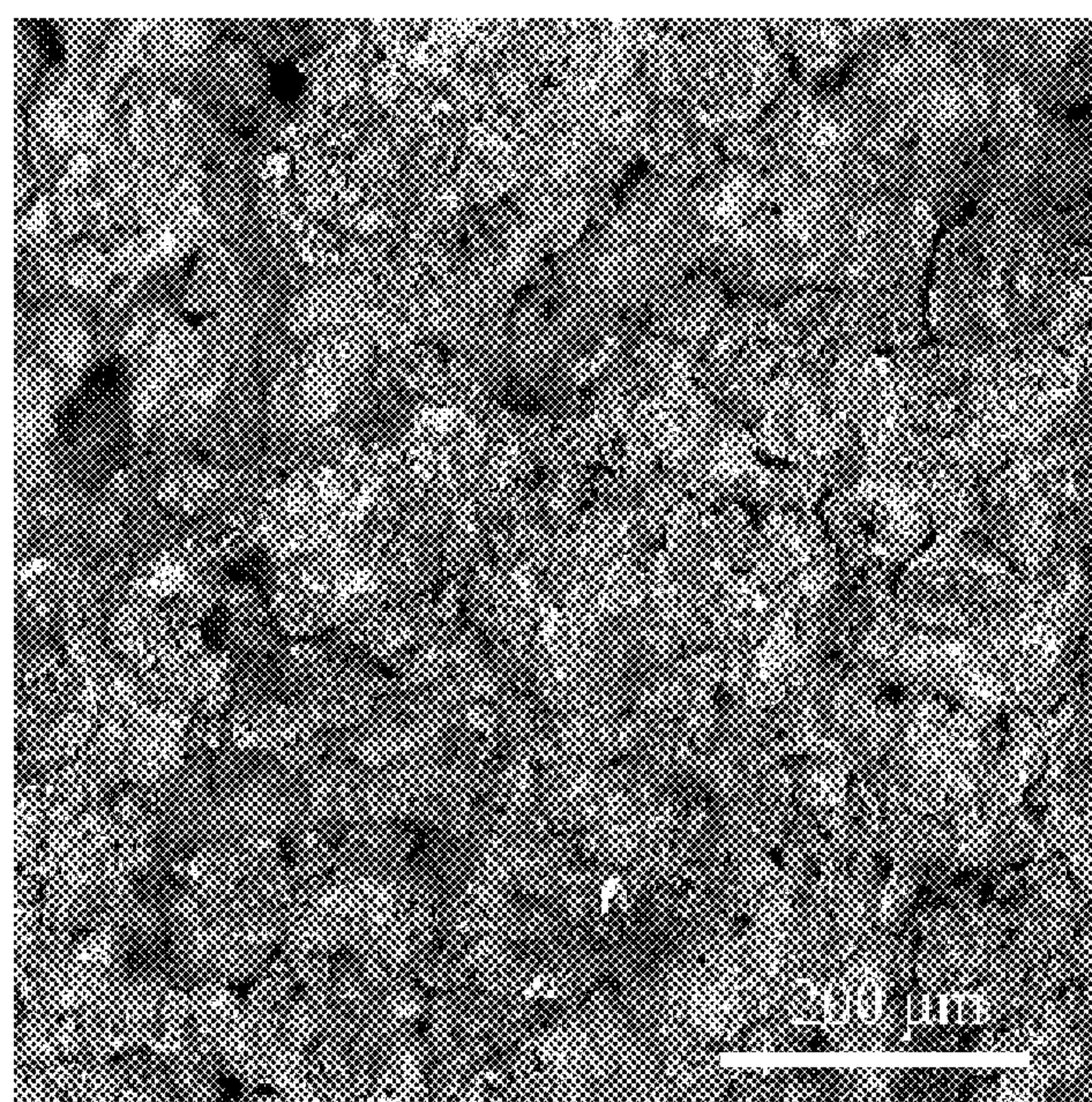


Fig.2

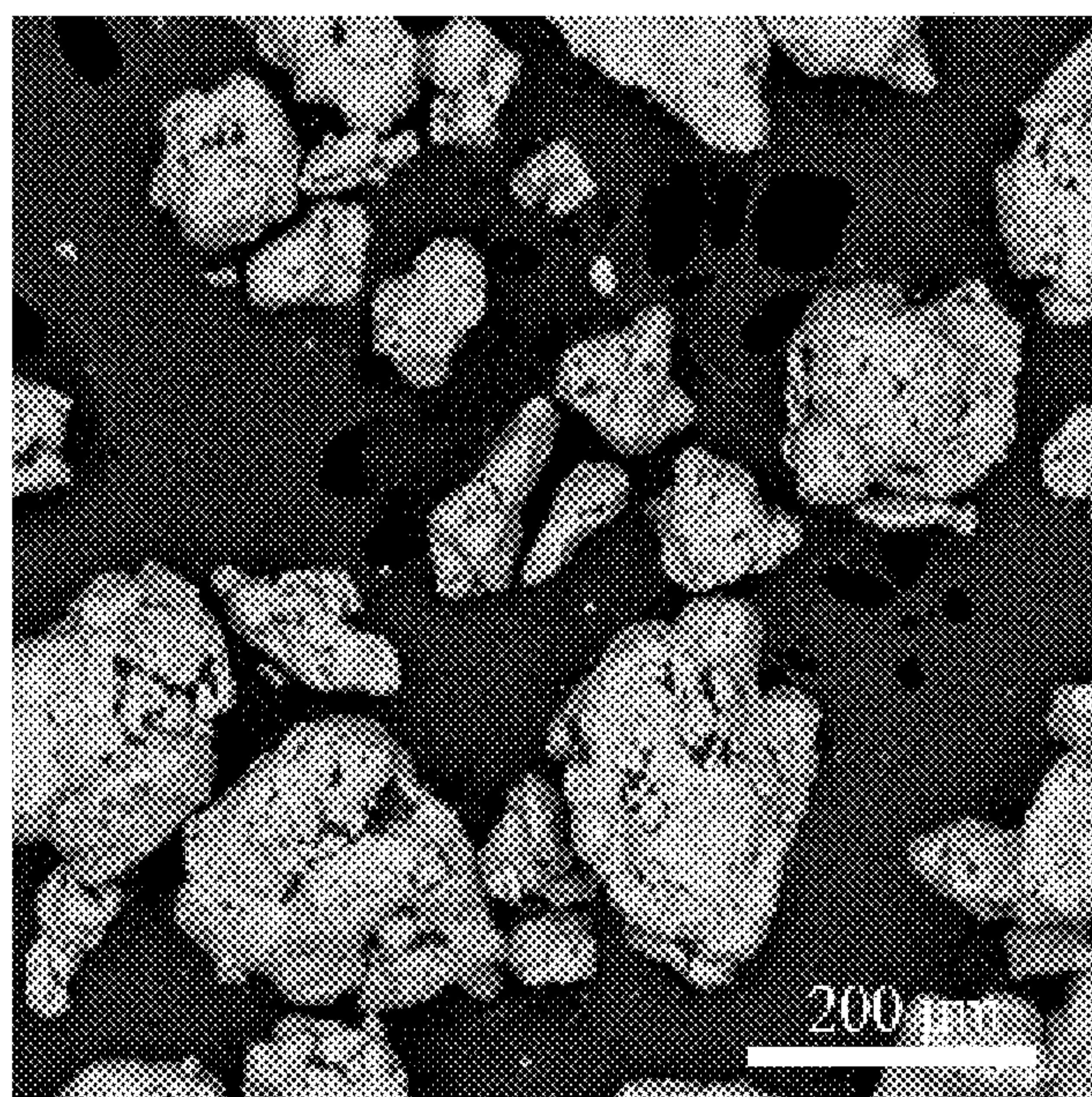


Fig.3

METHOD FOR PREPARING NANO-SiO₂ REINFORCED ALUMINUM MATRIX COMPOSITES

CROSS REFERENCE TO RELATED PATENT APPLICATION

[0001] The present application claims the priority of the Chinese patent application No. 201711342990.7 filed on Dec. 14, 2017 which is incorporated herein by reference.

TECHNICAL FIELD OF THE INVENTION

[0002] The present invention relates to the technical field of preparation of aluminum matrix composites, and in particular to a method for preparing nano-SiO₂ reinforced aluminum matrix composites.

BACKGROUND OF THE INVENTION

[0003] Aluminum alloys are lightweight material with low density and high specific strength. The replacement of iron-based or copper-based pieces with aluminum alloys is advantageous for weight reduction, energy conservation and emission reduction. Therefore, aluminum alloys have been widely applied in various fields such as aerospace, automobile, machine manufacturing, shipping and chemical industry. For example, aluminum alloys are widely applied in aluminum can bodies, crossbeams for supersonic fighters, high-pressure cylinders, automotive cantilever members, blades for engines, machine enclosures, cylinder sleeves and the like. However, in many working environments which require light weight and also high wear resistance, low coefficient of thermal expansion and excellent high-temperature mechanical properties, conventional aluminum alloy material cannot satisfy practical application requirements. In order to solve this problem, a ceramic phase is used to enhance the performance of aluminum matrix composites. The ceramic phase reinforced aluminum matrix composites have advantages of both the aluminum alloys and the ceramic reinforcing phase and can maintain high performance and high reliability under severe conditions such as high speed, vibration, humidity, no lubrication, less lubrication, demand for light weight, and corrosive atmosphere, so the ceramic phase reinforced aluminum matrix composites have a very promising industrial prospect. For example, in a Chinese patent CN1182063A entitled METHOD FOR PREPARING CERAMIC PARTICLE REINFORCED ALUMINUM MATRIX COMPOSITES, the combination of ceramic particles and aluminum powder greatly improves the performance of aluminum matrix composites.

[0004] There are many methods for compounding a ceramic phase with an aluminum matrix, for example, diffusion bonding, infiltration, dispersive mixing, spray atomization, in-situ synthesis and powder metallurgy. Composites prepared by powder metallurgy have a uniform reinforced phase and high performance, and the preparation process is an energy-saving and material-saving environmentally-friendly manufacturing technology and suitable for industrial applications. The ceramic phase powder used during the powder metallurgy can be preferably nanopowder. The nano-material has special advantages of surface effect and small size effect, and is obviously different from the conventional material in physical and chemical performances. By the good composition effect of nanoparticles and

an aluminum alloy matrix and fully utilizing respective performance advantages of the matrix and the reinforced phase, aluminum matrix composites with excellent performance may be obtained. A Chinese patent CN104498752A entitled METHOD FOR PREPARING MICRO/NANO-PARTICLE REINFORCED ALUMINUM MATRIX COMPOSITES disclosed a preparation method in which aluminum alloy powder is mixed with reinforced phase powder by high-energy ball-milling and then activated, sintered and densified. However, this method is relatively complicated in process steps and high in cost. In addition, a Chinese patent CN105039793A entitled METHOD FOR PREPARING MICRO/NANO-PARTICLE REINFORCED ALUMINUM MATRIX COMPOSITES disclosed a preparation method in which the aluminum matrix composites are reinforced by steps of mixing, ball-milling and hot pressing aluminum powder as a matrix and nano-silicon carbide particles as reinforced phase particles. This preparation method is simple in steps, but nano-silicon carbide particles are used as a ceramic phase and the performance of the prepared aluminum matrix composite needs to be further enhanced. At present, there is no preparation method in which aluminum matrix composites are reinforced by directly compounding nano-SiO₂ powder with aluminum alloy matrix powder by conventional powder metallurgy.

SUMMARY OF THE INVENTION

[0005] A technical problem to be solved by the present invention is, in view of the prior art, to provide a method for preparing nano-SiO₂ reinforced aluminum matrix composites, which is simple in process and good in performance of the prepared aluminum matrix composites.

[0006] To solve the above technical problem, the method for preparing nano-SiO₂ reinforced aluminum matrix composites comprises the following steps of:

[0007] Step-1, powder mixing: mixing aluminum matrix powder with nano-SiO₂ powder to obtain raw material powder, the raw material powder being uniformly mixed, wherein the aluminum matrix powder has an average particle size between 30 μm to 100 μm, the nano-SiO₂ powder has an average particle size between 5 nm to 145 nm, mass percentage of nano-SiO₂ in the raw material powder is 0.01% to 5% and remaining of the raw material powder is the aluminum matrix powder, and the aluminum matrix powder is elemental aluminum powder, aluminum alloy powder, or aluminum matrix composite powder;

[0008] Step-2, shaping: press shaping the powder obtained in the Step-1 to obtain base bodies;

[0009] Step-3, sintering: sintering the base bodies obtained in the Step-2 in an atmosphere of N₂ at 550° C. to 660° C., preserving the temperature for a period of 5 min to 60 min, and cooling in a furnace at end of the period under protection of N₂ for 0.5 h to 3 h; and

[0010] Step-4: successively performing solid solution and artificial aging heat treatment on the sintered blanks of the Step-3, where the temperature for the solid solution is 450° C. to 580° C., the time for the solid solution is 0.5 h to 6 h, the temperature for the artificial aging is 100° C. to 200° C. and the time for the artificial aging is 3 h to 24 h.

[0011] Preferably, in the Step-1, the average particle size of the aluminum matrix powder is 60 μm to 80 μm, the average particle size of the nano-SiO₂ powder is 10 nm to 50 nm, the mass percentage of the nano-SiO₂ powder in the raw material powder is 0.1% to 1.5%.

[0012] Preferably, the powder mixing in the Step-1 is done by a ball mill, a rotation speed of the ball mill is 180 r/min to 230 r/min, and the time for ball milling is 4 h to 8 h.

[0013] Preferably, the rotation speed of the ball mill is 180 r/min to 210 r/min, and the time for ball milling is 5 h to 6 h.

[0014] Preferably, the nano-SiO₂ powder in the Step-1 is hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET)	m ² /g	110 ± 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	≤0.5
Loss on ignition: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	≤2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	≥99.8

[0015] In order to make the demolding and shaping easy, in the powder mixing of the Step-1, a lubricant is added in the raw material powder, wherein the mass of the lubricant is 0.5% to 2% of the mass of the raw material powder. The lubricant may be a stearic acid lubricant or paraffin.

[0016] Preferably, the lubricant is added manually or by a mixer, wherein the mixer is any one of a ball mill, a V-type mixer, a conical mixer, a barrel mixer and a helical mixer.

[0017] In order to prevent the lubricant from influencing the composites, the base bodies are dewaxed for 20 min to 50 min at 350° C. to 450° C. before the sintering in the Step-3.

[0018] Preferably, in the Step-2, the press shaping method of the raw material powder is die shaping, and the pressure for shaping is 150 MPa to 500 MPa.

[0019] Additionally, the Step-4 will not be performed when the sinter is an elemental aluminum matrix or a non-heat-treatable aluminum alloy matrix.

[0020] Compared with the prior art, in the present invention, a nanometer reinforcing phase is directly added in an aluminum matrix, the process is simple, and the required equipment is common powder metallurgy equipment. Since the process is simple, the present invention is suitable for the powder metallurgy of nano-SiO₂ reinforced multi-component aluminum matrix parts, high in universality and advantageous for popularization and large-scale production.

[0021] By adding second-phase nano-SiO₂ particles in the aluminum matrix, due to different states of the nano-SiO₂ particles and the matrix, lattice distortion will occur at an interface of the particles with the matrix to form a complicated strain state, so that the matrix is reinforced significantly. Meanwhile, the dislocation movement is hindered due to the presence of the nano-SiO₂ particles, so that a driving force required for the dislocation movement in the aluminum matrix is increased greatly and the strength is improved. In addition, due to a great difference in coefficient of thermal expansion between the nano-SiO₂ particles and the matrix, lots of dislocations will be inevitably caused by different strains resulted from the change in temperature during the sintering and heat treatment processes, and the matrix is also partially reinforced. Either the nano-SiO₂

particles or a flow rate enhancer may improve the liquidity and filling ability of the raw material powder, and it is advantageous to improve the uniformity of the shaping density and the strength. Therefore, the prepared aluminum matrix composites are good in performance.

BRIEF DESCRIPTION OF THE DRAWINGS

[0022] FIG. 1 is a micro-topography of nano-SiO₂ particles according to Embodiment 2 of the present invention;

[0023] FIG. 2 is a fracture topography of a sinter according to Embodiment 2 of the present invention; and

[0024] FIG. 3 is a micro-topography of mixed powder according to Embodiment 7 of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

[0025] The specific implementations of the present invention will be further described in detail by embodiments with reference to the accompanying drawings.

Embodiment 1

[0026] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a pure aluminum matrix as example. The method specifically includes the following steps.

[0027] Step-1, powder mixing: elemental aluminum powder having an average particle size of 60 μm and nano-SiO₂ powder having an average particle size of 50 nm were milled by a mortar and mixed uniformly to obtain raw material powder, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 0.01%, and the remaining was the elemental aluminum powder. The nano-SiO₂ powder used in this step may preferably be hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 ± 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	≤0.5
Ignition loss: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	≤2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	≥99.8

[0028] Step-2, shaping: the powder obtained in the step (1) was die-shaped, where the pressure for pressing was 150 MPa and the pressure holding time was 15 s.

[0029] Step-3, sintering: by a tubular furnace and in the atmosphere of high-purity N₂, the shaped powder was sintered at 650° C. for 60 min, and then cooled in the furnace at the end of temperature preservation. The sintering environment in this step may preferably have an oxygen content of less than 10 ppm and a dew point of below -40° C.

[0030] By tests, the flow rate of pure aluminum powder without nano-SiO₂ powder was 110 S/50 g, and the flow rate after the addition of 0.01% of nano-SiO₂ powder was 102 S/50 g. The strength of the pure aluminum powder without

nano-SiO₂ powder after sintering was 68 MPa, and the strength of the aluminum matrix composite obtained by sintering the pure aluminum powder and 0.01% of nano-SiO₂ powder was 76 MPa.

Embodiment 2

[0031] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a 2014 aluminum alloy matrix as example. The method specifically includes the following steps.

[0032] Step-1, powder mixing: 2014 aluminum alloy powder having an average particle size of 78 μm and nano-SiO₂ powder having an average particle size of 16 nm were mixed to obtain raw material powder, and the raw material powder was ball-milled for 4 h by a planetary ball mill at a rotation speed of 180 r/min and mixed uniformly, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 1%, and the remaining was the 2014 aluminum alloy powder. The nano-SiO₂ powder used in this step was preferably hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 \pm 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	\leq 0.5
Ignition loss: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	\leq 2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	\geq 99.8

[0033] Step-2, shaping: the powder obtained in the step (1) was uniformly mixed with zinc stearate as a lubricant by a V-type mixer and die-shaped, where the mass percentage of the zinc stearate in the raw material powder was 0.5 wt %, the pressure for pressing was 400 MPa and the pressure holding time was 20 s.

[0034] Step-3, sintering: by a mesh-belt continuous furnace and in the atmosphere of high-purity N₂, the shaped powder was dewaxed at 350° C. for 30 min, then sintered at 560° C. for 40 min, and cooled in the furnace at the end of temperature preservation to the room temperature within 2 h by controlling the cooling rate, so as to obtain a sinter. The sintering environment in this step may preferably have an oxygen content of less than 10 ppm and a dew point of below -40° C. As shown in FIG. 2, there were obvious dimples on the fracture surface of the sinter of the nano-SiO₂ reinforced 2014 aluminum alloy composite. It is indicated that the sinter realizes good metallurgical bonding and has high mechanical properties.

[0035] Step-4, Heat treatment: solid solution and artificial aging heat treatment were successively performed on the sinter, where the temperature for the solid solution was 510° C., the time for the solid solution was 1 h, the temperature for the artificial aging was 180° C. and the time for the artificial aging was 15 h.

[0036] By tests, the flow rate of the 2014 aluminum alloy powder without nano-SiO₂ powder was 90 S/50 g, and the flow rate after the addition of 1% of nano-SiO₂ powder was 79 S/50 g. The strength of the 2014 aluminum alloy powder without nano-SiO₂ powder after sintering was 210 MPa, and the strength of the aluminum matrix composite obtained by sintering the 2014 aluminum alloy powder and 1% of nano-SiO₂ powder was 235 MPa.

Embodiment 3

[0037] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a 7039 aluminum alloy matrix as example. The method specifically includes the following steps.

[0038] Step-1, powder mixing: 7039 aluminum alloy powder having an average particle size of 45 μm and nano-SiO₂ powder having an average particle size of 10 nm were mixed to obtain raw material powder, and the raw material powder was ball-milled for 4.5 h by a planetary ball mill at a rotation speed of 185 r/min and mixed uniformly, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 1.5%, and the remaining was the 7039 aluminum alloy powder. The nano-SiO₂ powder used in this step was preferably hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 \pm 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	\leq 0.5
Ignition loss: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	\leq 2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	\geq 99.8

[0039] Step-2, shaping: the powder obtained in the step (1) is uniformly mixed with zinc stearate as a lubricant by a helical mixer and die-shaped, where the mass percentage of the zinc stearate in the raw material powder was 0.8%, the pressure for pressing was 180 MPa and the pressure holding time was 15 s.

[0040] Step-3, sintering: by a mesh-belt continuous furnace and in the atmosphere of high-purity N₂, the shaped powder was dewaxed at 350° C. for 22 min, then sintered at 550° C. for 5 min, and cooled in the furnace at the end of temperature preservation to the room temperature within 0.5 h by controlling the cooling rate, so as to obtain a sinter. The sintering environment in this step may preferably have an oxygen content of less than 10 ppm and a dew point of below -40° C.

[0041] Step-4, Heat treatment: solid solution and artificial aging heat treatment were successively performed on the sinter, where the temperature for the solid solution was 450° C., the time for the solid solution was 0.8 h, the temperature for the artificial aging was 150° C. and the time for the artificial aging was 3 h.

[0042] By tests, the flow rate of the 4039 aluminum alloy powder without nano-SiO₂ powder was 92 S/50 g, and the flow rate after the addition of 1.5% of nano-SiO₂ powder was 86 S/50 g. The strength of the 7039 aluminum alloy powder without nano-SiO₂ powder after sintering was 134 MPa, and the strength of the aluminum matrix composite obtained by sintering the 7039 aluminum alloy powder and 1.5% of nano-SiO₂ powder was 143 MPa.

Embodiment 4

[0043] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a 6061 aluminum alloy matrix as example. The method specifically includes the following steps.

[0044] Step-1, powder mixing: 6061 aluminum alloy powder having an average particle size of 80 μm and nano-SiO₂ powder having an average particle size of 5 nm were mixed to obtain raw material powder, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 5%, and the remaining was the 6061 aluminum alloy powder. The raw material powder was ball-milled for 5 h by a planetary ball mill at a rotation speed of 180 r/min. At 0.5 h before the end of ball milling, stearic acid having a mass percentage of 1.5% in the raw material powder was added in the raw material powder as a lubricant. At the end of ball milling, mixed powder was obtained. The nano-SiO₂ powder used in this step may be preferably hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 \pm 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	\leq 0.5
Ignition loss: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	\leq 2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	\geq 99.8

[0045] Step-2, shaping: the powder obtained in the step (1) was die-shaped, where the pressure for pressing was 200 MPa and the pressure holding time is 10 s.

[0046] Step-3, sintering: by a pusher continuous furnace, in the atmosphere of high-purity N₂ and in a sintering environment having an oxygen content of less than 10 ppm and a dew point of below -40° C., the shaped powder was dewaxed at 450° C. for 20 min, then sintered at 590° C. for 60 min, and cooled in the furnace at the end of temperature preservation to the room temperature within 3 h by controlling the cooling rate, so as to obtain a sinter. The sintering environment in this step preferably had an oxygen content of less than 10 ppm and a dew point of below -40° C.

[0047] Step-4, Heat treatment: solid solution and artificial aging heat treatment were successively performed on the sinter, where the temperature for the solid solution was 580°

C., the time for the solid solution was 0.5 h, the temperature for the artificial aging was 160° C. and the time for the artificial aging was 24 h.

[0048] By tests, the flow rate of the 6061 aluminum alloy powder without nano-SiO₂ powder was 93 S/50 g, and the flow rate after the addition of 5% of nano-SiO₂ powder was 84 S/50 g. The strength of the 6061 aluminum alloy powder without nano-SiO₂ powder after sintering was 127 MPa, and the strength of the aluminum matrix composite obtained by sintering the 6061 aluminum alloy powder and 5% of nano-SiO₂ powder was 151 MPa.

Embodiment 5

[0049] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a 6061 aluminum alloy matrix as example. The method specifically includes the following steps.

[0050] Step-1, powder mixing: 6061 aluminum alloy powder having an average particle size of 75 μm and nano-SiO₂ powder having an average particle size of 35 nm were mixed to obtain raw material powder, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 1.2%, and the remaining was the 6061 aluminum alloy powder. The raw material powder was ball-milled for 5.5 h by a planetary ball mill at a rotation speed of 185 r/min. At 0.5 h before the end of ball milling, stearic acid having a mass percentage of 1.8% in the raw material powder was added in the raw material powder as a lubricant. At the end of ball milling, mixed powder was obtained. The nano-SiO₂ powder used in this step may preferably be hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 \pm 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	\leq 0.5
Ignition loss: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	\leq 2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	\geq 99.8

[0051] Step-2, shaping: the powder obtained in the step (1) was die-shaped, where the pressure for pressing was 350 MPa and the pressure holding time is 25 s.

[0052] Step-3, sintering: by a pusher continuous furnace, in the atmosphere of high-purity N₂ and in a sintering environment having an oxygen content of less than 10 ppm and a dew point of below -40° C., the shaped powder was dewaxed at 370° C. for 45 min, then sintered at 660° C. for 55 min, and cooled in the furnace at the end of temperature preservation to the room temperature within 2.5 h by controlling the cooling rate, so as to obtain a sinter. The sintering environment in this step preferably had an oxygen content of less than 10 ppm and a dew point of below -40° C.

[0053] Step-4, Heat treatment: solid solution and artificial aging heat treatment were successively performed on the sinter, where the temperature for the solid solution was 550° C., the time for the solid solution was 3.5 h, the temperature for the artificial aging was 200° C. and the time for the artificial aging was 15 h.

[0054] By tests, the flow rate of the 6061 aluminum alloy powder without nano-SiO₂ powder was 95 S/50 g, and the flow rate after the addition of 1.2% of nano-SiO₂ powder was 82 S/50 g. The strength of the 6061 aluminum alloy powder without nano-SiO₂ powder after sintering was 193 MPa, and the strength of the aluminum matrix composite obtained by sintering the 6061 aluminum alloy powder and 1.2% of nano-SiO₂ powder was 209 MPa.

Embodiment 6

[0055] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a 7075 aluminum alloy matrix as example. The method specifically includes the following steps.

[0056] Step-1, powder mixing: 7075 aluminum alloy powder having an average particle size of 100 μm and nano-SiO₂ powder having an average particle size of 8 nm were mixed to obtain raw material powder, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 0.5%, and the remaining was the 7075 aluminum alloy powder. The raw material powder was ball-milled for 5 h by a planetary ball mill at a rotation speed of 200 r/min. At 0.5 h before the end of ball milling, paraffin having a mass percentage of 2% in the raw material powder was added in the raw material powder as a lubricant. At the end of ball milling, mixed powder was obtained. The nano-SiO₂ powder used in this step may preferably be hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 ± 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	≤0.5
Ignition loss: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	≤2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	≥99.8

[0057] Step-2, shaping: the powder obtained in the step (1) was die-shaped, where the pressure for pressing was 450 MPa and the pressure holding time is 30 s.

[0058] Step-3, sintering: by a pusher continuous furnace and in the atmosphere of high-purity N₂, the shaped powder was dewaxed at 350° C. for 50 min, then sintered at 620° C. for 30 min, and cooled in the furnace at the end of temperature preservation to the room temperature within 3 h by controlling the cooling rate, so as to obtain a sinter. The sintering environment in this step may preferably have an oxygen content of less than 10 ppm and a dew point of below -40° C.

[0059] Step-4, Heat treatment: solid solution and artificial aging heat treatment were successively performed on the sinter, where the temperature for the solid solution was 460° C., the time for the solid solution was 6 h, the temperature for the artificial aging was 100° C. and the time for the artificial aging was 8 h.

[0060] By tests, the flow rate of the 7075 aluminum alloy powder without nano-SiO₂ powder was 98 S/50 g, and the flow rate after the addition of 0.5% of nano-SiO₂ powder was 85 S/50 g. The strength of the 7075 aluminum alloy powder without nano-SiO₂ powder after sintering was 241 MPa, and the strength of the aluminum matrix composite obtained by sintering the 7075 aluminum alloy powder and 0.5% of nano-SiO₂ powder was 282 MPa.

Embodiment 7

[0061] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a high-silicon aluminum alloy 4A11 matrix as example. The method specifically includes the following steps.

[0062] Step-1, powder mixing: 4A11 aluminum alloy powder having an average particle size of 30 μm and nano-SiO₂ powder having an average particle size of 145 nm were mixed to obtain raw material powder, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 0.08%, and the remaining was the 4A11 aluminum alloy powder. The raw material powder was ball-milled for 6 h by a planetary ball mill at a rotation speed of 200 r/min. At 0.5 h before the end of ball milling, stearic acid having a mass percentage of 1.5% in the raw material powder was added in the raw material powder. At the end of ball milling, mixed powder was obtained. The nano-SiO₂ powder used in this step may preferably be hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 ± 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	≤0.5
Ignition loss: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	≤2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	≥99.8

[0063] Step-2, shaping: the powder obtained in the step (1) was die-shaped, where the pressure for pressing was 250 MPa and the pressure holding time is 10 s.

[0064] Step-3, sintering: by a tubular furnace and in the atmosphere of high-purity N₂, the shaped powder was dewaxed at 400° C. for 35 min, then sintered at 610° C. for 50 min, and cooled in the furnace at the end of temperature preservation to the room temperature within 1 h by controlling the cooling rate, so as to obtain a sinter. The sintering environment in this step may preferably have an oxygen content of less than 10 ppm and a dew point of below -40° C.

[0065] Step-4, Heat treatment: solid solution and artificial aging heat treatment were successively performed on the sinter, where the temperature for the solid solution was 525° C., the time for the solid solution was 1.5 h, the temperature for the artificial aging was 175° C. and the time for the artificial aging was 10 h.

[0066] By tests, the flow rate of the 4A11 aluminum alloy powder without nano-SiO₂ powder was 103 S/50 g, and the flow rate after the addition of 0.08% of nano-SiO₂ powder was 97 S/50 g. The strength of the 4A11 aluminum alloy powder without nano-SiO₂ powder after sintering was 186 MPa, and the strength of the aluminum matrix composite obtained by sintering the 4A11 aluminum alloy powder and 0.08% of nano-SiO₂ powder was 198 MPa.

Embodiment 8

[0067] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a high-silicon aluminum alloy Al-10Si matrix as example. The method specifically includes the following steps.

[0068] Step-1, powder mixing: Al-10Si aluminum alloy powder having an average particle size of 50 μm and nano-SiO₂ powder having an average particle size of 100 nm were mixed to obtain raw material powder, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 0.05%, and the remaining was the Al-10Si aluminum alloy powder. The raw material powder was ball-milled for 8 h by a planetary ball mill at a rotation speed of 230 r/min. At 0.5 h before the end of ball milling, paraffin having a mass percentage of 1.5% in the raw material powder was added in the raw material powder as a lubricant. At the end of ball milling, mixed powder was obtained. The nano-SiO₂ powder used in this step may preferably be hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 ± 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	≤0.5
Ignition loss: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	≤2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	≥99.8

[0069] Step-2, shaping: the powder obtained in the step (1) was die-shaped, where the pressure for pressing was 250 MPa and the pressure holding time is 10 s.

[0070] Step-3, sintering: by a mesh-belt continuous furnace and in the atmosphere of high-purity N₂, the shaped powder was dewaxed at 380° C. for 40 min, then sintered at 595° C. for 45 min, and cooled in the furnace at the end of temperature preservation to the room temperature within 3 h by controlling the cooling rate, so as to obtain a sinter. The sintering environment in this step may preferably have an oxygen content of less than 10 ppm and a dew point of below -40° C.

[0071] Step-4, Heat treatment: solid solution and artificial aging heat treatment were successively performed on the sinter, where the temperature for the solid solution was 520° C., the time for the solid solution was 2 h, the temperature for the artificial aging was 170° C. and the time for the artificial aging was 10 h.

[0072] By tests, the flow rate of the Al-10Si aluminum alloy powder without nano-SiO₂ powder was 107 S/50 g, and the flow rate after the addition of 0.05% of nano-SiO₂ powder was 95 S/50 g. The strength of the Al-10Si aluminum alloy powder without nano-SiO₂ powder after sintering was 130 MPa, and the strength of the aluminum matrix composite obtained by sintering the Al-10Si aluminum alloy powder and 0.05% of nano-SiO₂ powder was 145 MPa.

Embodiment 9

[0073] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a ceramic particle reinforced Al₂O₃-2024 aluminum matrix composite as example. The method specifically includes the following steps.

[0074] Step-1, powder mixing: Al₂O₃-2024 aluminum matrix composite powder having an average particle size of 85 μm and nano-SiO₂ powder having an average particle size of 15 nm were mixed to obtain raw material powder, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 0.1%, and the remaining was the Al₂O₃-2024 aluminum matrix composite powder. The raw material powder was ball-milled for 7 h by a planetary ball mill at a rotation speed of 210 r/min. At 0.5 h before the end of ball milling, zinc stearate having a mass percentage of 1% in the raw material powder was added in the raw material powder as a lubricant. At the end of ball milling, mixed powder was obtained. As shown in FIG. 3, it could be known that, by uniformly mixing Al₂O₃-2024 aluminum matrix composite powder with the nano-SiO₂ powder, the nano-SiO₂ particles could be well bonded with the matrix after sintering, so that the mechanical properties were significantly enhanced. The nano-SiO₂ powder used in this step may preferably be hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 ± 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	≤0.5
Ignition loss: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	≤2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	≥99.8

[0075] Step-2, shaping: the powder obtained in the step (1) was die-shaped, where the pressure for pressing was 300 MPa and the pressure holding time is 20 s.

[0076] Step-3, sintering: by a mesh-belt continuous furnace and in the atmosphere of high-purity N₂, the shaped powder was dewaxed at 440° C. for 25 min, then sintered at 585° C. for 40 min, and cooled in the furnace at the end of

temperature preservation to the room temperature within 2 h by controlling the cooling rate, so as to obtain a sinter. The sintering environment in this step may preferably have an oxygen content of less than 10 ppm and a dew point of below -40°C .

[0077] Step-4, Heat treatment: solid solution and artificial aging heat treatment were performed on the sinter, where the temperature for the solid solution was 490°C ., the time for the solid solution was 1 h, the temperature for the artificial aging was 190°C . and the time for the artificial aging was 20 h.

[0078] By tests, the flow rate of the Al₂O₃-2024 aluminum matrix composite powder without nano-SiO₂ powder was 94 S/50 g, and the flow rate after the addition of 0.1% of nano-SiO₂ powder was 81 S/50 g. The strength of the Al₂O₃-2024 aluminum matrix composite powder without nano-SiO₂ powder after sintering was 226 MPa, and the strength of the aluminum matrix composite obtained by sintering the Al₂O₃-2024 aluminum matrix composite powder and 0.1% of nano-SiO₂ powder was 257 MPa.

Embodiment 10

[0079] In this embodiment, the method for preparing nano-SiO₂ reinforced aluminum matrix composites is described by taking a non-heat-treatable 3003 aluminum alloy matrix as example. The method specifically includes the following steps.

[0080] Step-1, powder mixing: 3003 aluminum alloy powder having an average particle size of 70 μm and nano-SiO₂ powder having an average particle size of 70 nm were mixed to obtain raw material powder, where the mass percentage of the nano-SiO₂ powder in the raw material powder was 0.4%, and the remaining was the 3003 aluminum alloy powder. The raw material powder was ball-milled for 4 h by a planetary ball mill at a rotation speed of 190 r/min. At 0.5 h before the end of ball milling, paraffin having a mass percentage of 1.5% in the raw material powder was added in the raw material powder as a lubricant. At the end of ball milling, mixed powder was obtained. The nano-SiO₂ powder used in this step may preferably be hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET method)	m ² /g	110 \pm 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon content	Wt. %	0.6-1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105°C .	Wt. %	≤ 0.5
Ignition loss: the material dried for 2 h at 105°C . is burnt for 2 h at 1000°C .	Wt. %	≤ 2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	≥ 99.8

[0081] Step-2, shaping: the powder obtained in the step (1) was die-shaped, where the pressure for pressing was 500 MPa and the pressure holding time is 10 s.

[0082] Step-3, sintering: by a pusher continuous furnace and in the atmosphere of high-purity N₂, the shaped powder was dewaxed at 400°C . for 30 min, then sintered at 570°C . for 40 min, and cooled in the furnace at the end of temperature preservation to the room temperature within 3 h by controlling the cooling rate, so as to obtain a sinter. The sintering environment in this step may preferably have an oxygen content of less than 10 ppm and a dew point of below -40°C .

[0083] By tests, the flow rate of the 3003 aluminum alloy powder without nano-SiO₂ powder was 91 S/50 g, and the flow rate after the addition of 0.4% of nano-SiO₂ powder was 78 S/50 g. The strength of the 3003 aluminum alloy powder without nano-SiO₂ powder after sintering was 112 MPa, and the strength of the aluminum matrix composite obtained by sintering the 3003 aluminum alloy powder and 0.4% of nano-SiO₂ powder was 140 MPa.

1. A method for preparing nano-SiO₂ reinforced aluminum matrix composites, comprising the following steps of:

Step-1, powder mixing: mixing aluminum matrix powder with nano-SiO₂ powder to obtain raw material powder, the raw material powder being uniformly mixed, wherein the aluminum matrix powder has an average particle size between 30 μm to 100 μm , the nano-SiO₂ powder has an average particle size between 5 nm to 145 nm, mass percentage of nano-SiO₂ in the raw material powder is 0.01% to 5% and remaining of the raw material powder is the aluminum matrix powder, and the aluminum matrix powder is elemental aluminum powder, aluminum alloy powder, or aluminum matrix composite powder;

Step-2, shaping: press shaping the powder obtained in the Step-1 to obtain base bodies;

Step-3, sintering: sintering the base bodies obtained in the Step-2 in an atmosphere of N₂ at 550°C . to 660°C ., preserving the temperature for a period of 5 min to 60 min, and cooling in a furnace at end of the period under protection of N₂ for 0.5 h to 3 h; and

Step-4: successively performing solid solution and artificial aging heat treatment on the sintered blanks of the Step-3, where the temperature for the solid solution is 450°C . to 580°C ., the time for the solid solution is 0.5 h to 6 h, the temperature for the artificial aging is 100°C . to 200°C . and the time for the artificial aging is 3 h to 24 h.

2. The method of claim 1, wherein in the Step-1, the average particle size of the aluminum matrix powder is 60 μm to 80 μm , the average particle size of the nano-SiO₂ powder is 10 nm to 50 nm, the mass percentage of the nano-SiO₂ powder in the raw material powder is 0.1% to 1.5%.

3. The method of claim 1, wherein the powder mixing in the Step-1 is done by a ball mill, a rotation speed of the ball mill is 180 r/min to 230 r/min, and the time for ball milling is 4 h to 8 h.

4. The method of claim 3, wherein the rotation speed of the ball mill is 180 r/min to 210 r/min, and the time for ball milling is 5 h to 6 h.

5. The method of claim 1, wherein the nano-SiO₂ powder in the Step-1 is hydrophobic gas-phase silicon dioxide having main parameters shown in the following table:

Property	Unit	Typical value
Specific surface area (BET)	m ² /g	110 ± 20
Bulk density*	g/l	50
in accordance with DIN EN ISO 787/11, August 1983		
Carbon	Wt. %	0.6~1.2
Average particle size of primary particles	nm	16
Moisture content: dried for 2 h at 105° C.	Wt. %	≤0.5
Loss on ignition: the material dried for 2 h at 105° C. is burnt for 2 h at 1000° C.	Wt. %	≤2.0
pH value: in a 4% dispersoid	Wt. %	3.6-4.4
SiO ₂ content: for the burnt material	Wt. %	≥99.8

6. The method of claim 1, wherein in the powder mixing of the Step-1, a lubricant is added in the raw material powder, wherein the mass of the lubricant is 0.5% to 2% of the mass of the raw material powder.

7. The method of claim 6, wherein the lubricant is added manually or by a mixer, wherein the mixer is any one of a ball mill, a V-type mixer, a conical mixer, a barrel mixer and a helical mixer.

8. The method of claim 6 or claim 7, wherein the base bodies are dewaxed for 20 min to 50 min at 350° C. to 450° C. before the sintering in the Step-3.

9. The method of claim 1, wherein in the Step-2, the press shaping method of the raw material powder is die shaping, and the pressure for shaping is 150 MPa to 500 MPa.

10. The method of claim 1, wherein the Step-4 will not be performed when the sinter is an elemental aluminum matrix or a non-heat-treatable aluminum alloy matrix.

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