PARTICULATE ALUMINIUM MATRIX NANO-COMPOSITES AND A PROCESS FOR PRODUCING THE SAME

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ABSTRACT

The present invention provides a process for reinforced aluminum matrix composite. The aluminum matrix composite is reinforced with compound selected from the group consisting of Titanium carbide, Titanium boride, Vanadium and Zirconium compounds. The process is carried out pneumatically using pressurized carrier gas. The pressurized carrier gas also provides efficient stirring during the process which leads to uniform dispersion of the particulate in the aluminum matrix.
FIGURE 2
FIGURE 4
PARTICULATE ALUMINUM MATRIX NANO-COMPOSITES AND A PROCESS FOR PRODUCING THE SAME

CROSS-REFERENCE TO RELATED APPLICATIONS


FIELD OF INVENTION

[0003] The present disclosure relates to metal matrix composites.

[0004] Particularly, the present disclosure envisages a reinforced Aluminum composites and a process for producing the same.

DEFINITION OF THE TERM USED IN THE SPECIFICATION

[0005] The term “Pneumatically” used in the specification means a process (function/operation) carried out using air, gas such as a carrier/ inert gas or a gaseous mixture.

BACKGROUND OF INVENTION

[0006] A metal matrix composite (MMC) is a composite material with at least two constituent parts, one being a metal and the other material being a different non metallic material such as a ceramic or inorganic compound.

[0007] Metal matrix composites (MMC) are tailor made material comprising a reinforcing material dispersed in a matrix metal. The reinforcing material can be synthesized externally and added to the metal matrix or else prepared in-situ in the metal.

[0008] One particular class of MMC that have gained more interest in the recent times is particulate reinforced aluminum matrix composites, prepared by in-situ techniques. These composites have superior mechanical properties compared to the aluminum matrix and have applications in transportation, electronics and recreational products.

[0009] U.S. Pat. No. 4,772,452 discloses a process for TiC reinforced aluminum matrix composites wherein the aluminum metal, titanium bearing compound and the carbide, all provided in the powder form are pre-mixed, compacted and further heated at a reaction temperature approximating melting point of the aluminum to produce the composite.

[0010] U.S. Pat. No. 6,843,865, discloses a process for TiC reinforced aluminum matrix composites wherein the mixture of aluminum and titanium metals in its molten form is reacted with a halide of carbon to produce the composite. The reaction is carried out under vigorous mechanical stirring.

[0011] U.S. Pat. No. 4,748,001 discloses a process for TiC reinforced aluminum matrix composites wherein the carbon powder preheated to 7000 C is added to the molten mixture of aluminum and titanium metals and the melt is stirred vigorously at high temperature and additional processing is carried out at a very high temperature (1100 to 1400 C) to produce the desired composite. The melt is agitated by mechanical stirring.

[0012] One severe limitation of the above techniques is heterogeneous distribution of the reinforced particulate, leading to variation in the properties within the sample and batch to batch. Also other parameters such as additional processing at a very high temperature (1100 to 1400 C), preheating of precursors to allow wetting of the powders to the melt and controlled powder size within tight specifications to enable good mixing and wetting leads to high processing cost. Some of the process as disclosed above are used to make composites with up to 5% particulate reinforcement, beyond which the mixing is very poor.

[0013] Therefore, there is felt a need to develop a composite having higher amount of particulate reinforcement and uniform homogenous distribution of the particulate reinforcement for superior mechanical properties.

OBJECTS

[0014] Main object of the present disclosure is to prepare aluminum composites with fine and uniform distribution of the particulate.

[0015] Another object of the present disclosure is to provide aluminum composites with improved mechanical properties.

[0016] Yet another object of the disclosure is to provide a cost effective process for preparing aluminum composites.

SUMMARY OF INVENTION

[0017] A process for preparing particulate aluminum matrix nano-composites, said process comprising the following steps:

[0018] a) injecting a mixture comprising (i) at least one metal bearing compound selected from the group consisting of titanium compounds, vanadium compounds and zirconium compounds, and (ii) at least one non-metal bearing compound selected from the group containing carbon bearing compounds, boron bearing compounds and oxygen bearing compounds into molten aluminum metal maintained at a temperature in the range of 7500 C to 12000 C to obtain a melt;

[0019] c) agitating the melt for a period of 5 to 60 minutes to obtain molten composite; and

[0020] d) casting and solidifying the molten composite.

[0021] In a preferred embodiment of the present disclosure the injecting step is carried out such that at least one of the compounds in the mixture is injected pneumatically. Typically, the injecting step is carried out pneumatically using pressurized carrier gas.

[0022] Typically, at least one of the compounds in the mixture in step a) is pneumatically injected into the molten aluminum through a feeder attached to a submersible lance, said lance being immersed in the molten aluminum metal.

[0023] In a preferred embodiment of the present disclosure the melt is agitated with a carrier gas. Typically, the melt is agitated with the carrier gas over a period of 5 to 20 minutes.

[0024] Typically, the carrier gas is selected from the group consisting of argon and nitrogen.
Typically, the temperature in the step a) to step b) is maintained in the range of 8500 C to 10000 C.

In a preferred embodiment of the present disclosure the compound in step a) is selected from the group consisting of potassium titanium fluoride, titanium oxide, titanium diboride.

Typically, the compound is titanium compound selected from the group consisting of potassium titanium fluoride and titanium oxide. Typically, the titanium compound is in the powder form.

In a preferred embodiment of the present disclosure, the carbon is selected from the group consisting of graphite powder, carbon-dioxide and methane gas.

In a preferred embodiment of the present disclosure, the oxygen is selected from the group consisting of oxygen gas, silica, alumina, ozone and cuprous oxide.

In a preferred embodiment of the present disclosure, the particulate aluminum matrix nano-composite as formed contains up to 15% titanium carbide composite.

In another aspect of the present disclosure, the particulate aluminum matrix nano-composite further comprises at least one alloying metal selected from the group consisting of magnesium, copper, zinc and silicon.

BRIEF DESCRIPTION OF DRAWINGS

FIG. 1 represents XRD overlay of samples prepared according to present disclosure and the conventional route.

FIG. 2 represents Scanning electron micrographs of prepared according to present disclosure and the conventional route.

FIG. 3 represents Tensile curve of cast aluminum sample and composite made by present disclosure.

FIG. 4 represents Photographs of samples produced by (a) method of present disclosure and (b) conventional stir casting method.

FIG. 5 represents Optical micrograph of sample prepared by method of present disclosure employing carbon dioxide as source of carbon.

FIG. 6 represents Aging curve for composite sample prepared by method of this disclosure.

FIG. 7 represents Extruded composite samples.

FIG. 8 represents Forged composite samples.

DETAILED DESCRIPTION

Metal matrix composites (MMC) are tailor made material consisting a reinforcing material dispersed in a metal matrix. The matrix is a monolithic material into which the reinforcement is embedded. The reinforcement is provided to improve physical properties such as wear resistance, friction coefficient and thermal conductivity of the metal.

Various methods are used to prepare MMC such as i) Solid state method, where powdered metal and reinforcement material are mixed and then bonded through a process of compaction, degassing, and thermo-mechanical treatment. ii) Liquid state method wherein reinforcement material is stirred into the molten metal and allowed to solidify. iii) A chemical reaction between the reactants to form reinforcement material in-situ in metal matrix. iv) Vapor deposition wherein the fiber is passed through a thick cloud of vaporized metal, coating it.

Aluminum Matrix Composites are manufactured by the fabrication methods such as Powder metallurgy (sintering), Stir casting and Infiltration. Usually the reinforcement of Aluminum Matrix Composites results in high strength, high stiffness (modulus of elasticity), low density, high thermal conductivity and excellent abrasion resistance of the reinforced metal compared to properties of pure metal.

Aluminum Matrix Composites (AMC) are used for manufacturing automotive parts (pistons, pushrods, brake components), brake rotors for high speed trains, bicycles, golf clubs, electronic substrates, cars for high voltage electrical cables.

The present disclosure provides a process for in-situ reinforced aluminum matrix composite. The aluminum matrix composite is reinforced with at least one compound obtained by the reaction of a metal bearing compound selected from the group consisting of Titanium compounds, Vanadium compounds, Zirconium compounds, and a non-metal bearing compound selected from the group consisting of carbon bearing compounds, boron bearing compounds and oxygen bearing compounds, wherein the preferred reinforcing compound is Titanium carbide. TiC particulate is prepared in-situ by injecting titanium bearing compound and a carbon bearing compound into the molten aluminum. The Titanium compound is selected from the group consisting of Potassium titanium fluoride, titanium boride and titanium oxide. A pressurized powder injection lance is used to inject the ingredients in the molten aluminum melt. The Titanium bearing compound (e.g. Potassium titanium fluoride, titanium oxide) in powder form is injected pneumatically into the molten aluminum through a submerged lance placed in the bottom of the bath. Carbon can be added either as graphite powder mixed with titanium bearing salt or in the form of CO2/methane gas. An inert or reactive gas acts as the powder carrier and disperses the powder within the melt. The gas also agitates the melt to ensure intimate mixing, which enhances the reaction kinetics and lowers the processing temperature (750-1200°C) and time (5 to 60 min). The process thus avoids mechanical stirring which may lead to irregular particle size improvement is also observed in the homogeneity of mechanical properties, e.g. hardness variation is <5% within the casting. The present disclosure allows higher amount of reinforcement to be introduced in the melt (up to 15%), without compromising casting integrity. Composites prepared by this process have a finer and more uniform distribution compared to those prepared by conventional route of mechanical stirring. Therefore for the same volume fraction of particles, composites according to the disclosure have superior mechanical properties.

The disclosure will now be described with respect to the following examples which do not limit the disclosure in any way and only exemplifies the disclosure.

Example 1

462 gms of aluminum metal was melt in a graphite crucible at 900° C. A mixture of Potassium titanium fluoride and carbon powder (97.3 g of K2TiF6 and 7.5 g of C) was added to the molten aluminum using a screw feeder attached to an alumina lance immersed in the molten melt using argon as the carrier gas. After feeding the additives for 8 minutes, the screw feeder was switched off and the melt was mixed with argon stirring for additional 5 minutes. The amount of addition corresponds to a nominal addition of 5% TiC by volume. At the end of stirring, the crucible was removed from the furnace, the dress was skimmed off from the melt. Composite sample, generally represented by numeral 101 was cast in cast iron moulds.
[0047] FIG. 1 shows the X-ray diffraction pattern of the composite with peaks corresponding to aluminum, TiC and minor Al3Ti phases.

[0048] FIG. 2a shows Scanning Electron Microscope analysis showing a very fine and uniform distribution of equiaxed particles of Al4C3 and TiC. A few plates of Al3Ti are also found to be present.

[0049] To compare the homogeneity of the casting produced by the present disclosure and the conventional method, samples prepared by stir casting (mechanical stirring), generally represented by numeral 102 and Sample prepared by method of present disclosure, 101 were produced under identical conditions i.e. 9000 C and 30 minutes reaction time. About 15 hardness readings were taken on the longitudinal section of the casting and analyzed statistically. For the casting made using submerged lance and argon carrier gas in accordance with the present disclosure, Vicker’s hardness was measured to be 60.5+/−1.1, while for the casting made using graphite stirrer the hardness was 65.1+/−1.7. This shows that that casting made from the present disclosure leads to more homogenous casting compared to stir casting method.

Example 2

[0050] A number of casting were made by the two different methods as described in example 1 under different process parameters as tabulated in Table 1. XRD analysis of the composites shows that the present disclosure allows composites with large volume fraction of TiC precipitates to be prepared at lower temperatures, in shorter cycle times and without the need for pre-hosting the precursor material. The presence of TiC precipitates lead to enhancement of yield strength, tensile strength and Young’s modulus as shown in FIG. 3. Due to the equiaxed and well distributed nature of TiC precipitates, the ductility of the composites is not sacrificed.

Example 3

[0051] Metal matrix composites were made as described in example 1. One

[0052] Sample was prepared using coarse K2TiF6 powder having d90 of 300 micron while another sample was prepared using ground and sieved K2TiF6 powder having d90 of 68 micron. Hardness of both the samples was measured to be 51 Hv.

Example 4

[0053] Composite sample, generally represented by numeral 103 was prepared by the method of the present disclosure as described in Example 1. 12 kg of aluminum was melt to 9000 C and to the molten aluminum, a mixture of K2TiF6 and carbon powder was added through a screw feeder using argon as the carrier gas. The total addition corresponds to nominal addition of 10% of TiC volume fraction. The total batch time for the reaction was 20 minutes. After completion of the reaction, dross was removed from the crucible and skimmed melt was poured into a sand mould to produce billets. FIG. 4a shows the photograph of the defect free cast billet.

[0054] Another sample was prepared by the conventional stir casting method by melting 500 gms of aluminum to 9000 C. A mixture of 495 g of K2TiF6 and 22 g of carbon powders were mixed and added to the melt while stirring with graphite stirrer. The reaction was completed in 20 minutes. Due to high viscosity of the melt, skimming operation could not be carried out properly and some dross was left within the melt. The melt was poured in a cast iron mould. FIG. 4b shows a photograph of the casting.

Example 5

[0055] Aluminum composite sample, generally represented by numeral 104 was prepared by melting 530 gms of aluminum in a SiC crucible at 950C. 113 g of K2TiF6 powder was added to the melt and stirred manually using an alumina rod. CO2 gas was bubbled through the molten mixture for 10 minutes through an alumina lance submerged in the melt. The crucible was then removed from the furnace, dross was skimmed from the top of the melt and melt poured in cast iron moulds. XRD analysis revealed the formation of TiC precipitates in the casting and hardness was measured to be 48.2 Hv. Optical micrograph of the sample is shown in FIG. 5.

Example 6

[0056] A melt comprising of aluminum and K2TiF6 was bubbled with a mixture of CO2/N2 to produce Al—AlN—TiC composites. Alternatively, air was used as carrier gas instead of argon in Example 1 to produce Al—AlN—TiC composites.

Example 7

[0057] A Composite sample, generally represented by numeral 105 was prepared using the method described in example 1. Additional alloying additions of 0.5% Mg and 0.8% Si were made before pouring the composite in moulds. Sample test samples were prepared from the cast composite. The test samples were solutionized at 5500 C for 1 h and quenched in water. Solutionized samples were then heat treated at 1700 C for different duration and hardness taken. The aging curve is shown in FIG. 6.

Example 8

[0058] A number of composite samples were prepared by the method described in example 1. The cast samples were machined into billet form and extruded in the temperature range of 400 to 550 C in a die to form extruded sections like rod and 1-beam. FIG. 7 shows the extruded products without any visual surface defects. Some of the other samples were forged after preheating to 450 C and are shown in FIG. 8.

<table>
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<tr>
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<th>Carbon (g)</th>
<th>Process</th>
<th>Reaction temp</th>
<th>Batch time</th>
<th>Hardness</th>
<th>Precipitates</th>
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<td>Carbon (g)</td>
<td>Process</td>
<td>Reaction temp</td>
<td>Batch time</td>
<td>Hardness</td>
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<td>40.1</td>
<td>ALTiSiC</td>
</tr>
</tbody>
</table>

[0059] Analysis of the composites as tabulated in Table 1 shows that the present disclosure allows composites with large volume fraction of TiC precipitates to be prepared at lower temperatures, in shorter cycle times and without the need for pre-heating the precursor material.

[0060] Hardness of the composite prepared in accordance with the present disclosure was found to be 59 Hv5 compared to 44 Hv5 for Al—Si matrix using conventional technique. Hardness of composites prepared by mechanical stirring under similar conditions have hardness of 30 Hv5. Elastic modulus of the cast composite was measured from tensile tests to be 90 GPa compared to 69 GPa for pure aluminum.

[0061] Small pieces were cut from the cast ingot and hot forged at 450°C. These samples showed no signs of cracking during hot forging.

[0062] Sliding wear tests show significantly improved wear performance; 1.14 mm/3km for the composite compared to 2.27 mm/3km for pure aluminum.

[0063] While considerable emphasis has been placed herein on the specific steps of the preferred process, it will be appreciated that additional steps can be made and that many changes can be made in the preferred steps without departing from the principles of the disclosure. These and other changes in the preferred steps of the disclosure will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

What is claimed is:

1. A process for preparing particulate aluminum matrix nano-composites, said process comprising the following steps:
   a. injecting a mixture comprising (i) at least one metal bearing compound selected from the group consisting of titanium compounds, vanadium compounds and zirconium compounds, and (ii) at least one non-metal bearing compound selected from the group containing carbon bearing compounds, boron bearing compounds and oxygen bearing compounds, into molten aluminum metal maintained at a temperature in the range of 7500°C to 12000°C to obtain a melt;
   b. agitating the melt for a period of 5 to 60 minutes to obtain molten alloy; and
   c. casting and solidifying the molten alloy.

2. The process as claimed in claim 1, wherein the injecting step is carried out such that at least one of the compounds in the mixture is injected pneumatically.

3. The process as claimed in claim 1, wherein the injecting step is carried out pneumatically using pressurized carrier gas.

4. The process as claimed in claim 1, wherein at least one of the compounds in the mixture in step a) is pneumatically injected into the molten aluminum through a feeder attached to a submersible lance, said lance being immersed in the molten aluminum metal.

5. The process as claimed in claim 1, wherein the melt is agitated with a carrier gas.

6. The process as claimed in claim 1, wherein the melt is agitated with the carrier gas over a period of 5 to 60 minutes.

7. The process as claimed in claim 1, wherein the carrier gas is selected from the group consisting of argon and nitrogen.

8. The process as claimed in claim 1, wherein the compound in step a) is selected from the group consisting of potassium titanium fluoride, titanium oxide, titanium diboride, silica, alumina, zinc oxide and cuprous oxide.

9. The process as claimed in claim 1, wherein the compound in step a) is a titanium compound selected from the group consisting of potassium titanium fluoride and titanium oxide.

10. The process as claimed in claim 1, wherein the metal bearing compound is in powder form.

11. The process as claimed in claim 1, wherein the non metal bearing compound is selected from carbon bearing compounds and is further selected from the group consisting of graphite powder, carbon-dioxide and methane gas.

12. The process as claimed in claim 1, wherein the metal bearing compound selected is a compound of titanium and the non metal bearing compound is selected from carbon bearing compounds and the particulate aluminum matrix nano-composite so formed contains up to 15% titanium carbide composite.

13. The process as claimed in claim 1, wherein the particulate aluminum matrix nano-composite further comprises at least one alloying metal selected from the group consisting of magnesium, copper, zinc and silicon.

14. An aluminum matrix nano-composite as prepared by the process claimed in claim 1.