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Review Article

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Production of Metal Matrix Composites by *In situ* Techniques

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Abstract Composite, consisting of matrix and reinforcement phases is the material obtained by joining of at least two engineering materials. Reinforcement components are generally supplied into the liquid matrix externally. This method is termed '*ex situ'*. There are some disadvantages of this method on microstructures of composites. Agglomeration of the reinforcement components, non-homogenous microstructure and risk of breakage of ceramic particles with high hardness are some of them. However, the reinforcement elements can be synthesized through chemical reactions that occur within the molten matrix. Higher strength composites can be obtained by technique called as '*in situ*'. This synthesis technique include exothermic dispersion (XD), mechanical alloying (MA) and reactive hot pressing (RHP). In the current study, it is mentioned properties of metal matrix composites (MMCs) produced by different methods.

Keywords *In situ,* Metal matrix composites, Reinforcement elements

Introduction

Reinforcement elements forms the composites can be produced through chemical reactions within solid or liquid matrix. Composites produced by this way (*in situ*) have many excellent advantages, such as clean reinforcement-matrix interface, fine and thermodynamically stable reinforcements, good compatibility and high bond strength between reinforcements-matrix, and low fabrication costs [1]. The chemical reactions between the reactans, which are keys to process the *in situ* MMCs, are extremely important [2]. Al_2O_3, TiB_2, TiC are most widely used as reinforcement and they does not react with matrix [3]. Because theyare stable components. Through *in situ* process, composites will have a more uniform microstructure.

In situ MMCs have some characteristics, such ashigh wear and corrosion resistance (thanks to ceramic phases), high spesific modul, thermodynamically stable reinforcements, and low sensitivity to thermal shocks and temperature change. There are some parameters that affect mechanical properties of the *in situ* composites produced byliquid and solidstate powder processing technique: weight percentages of reactants, reaction time, sintering temperature, pressure of hot-cold pressing, heating-cooling rates.The change of these parameters can change the mentioned above wear and corrosion properties ofcomposites, including hardness first.

In this study, the MMCs are described produced by different methods, such as exothermic dispersion (XD), mechanical alloying (MA), reactive hot pressing (RHP) and general principles of the production methods are defined.

2. *In situ* Production Methods

2.1. Mechanical alloying (MA)

This process consists of repeated welding–fracturing–rewelding of a mixture of powder particles in a high energy ball mill [4]. It is a solid state powder processing technique. The process avoids many problems associated with conventional melting and solidification [5-6].

Arik *et al*. (2006) were alloyed mechanically Al and C powders with those parameters: in total 50 g powder (48,5 g Al-1,5 g C), ball to powder ratio 6:1, 450 rpm rotor speed, milling time 20 hand argon atmosphere. They compacted the powders at 1000 MPa pressure to produce blanks with ø10 x 25 mm in size. Blanks were put in graphite boat which was placed in an atmosphere controlled tube furnace and heated to 650 ^{o}C in a flowing argon atmosphere and then cooled to room temperature at 5 ^{o}C/min. Sintering process was performed for 20 h. After sintering they testedt he composites to wear test using a pin on-discwear tester under dry sliding condition. Tests were carried out at 1.41 m/ssliding velocity and at applied loads of 20, 50 and 80 N. Sliding distance is 2538 m, track diameter 90 mm and sliding time is 30 min [7].

According to their results; in the case of low applying force, Fe based stable mechanical mixed layer was formed on the pin surface and which decreased the wear rate. However, high applied force resulted in the formation of mechanical deformed layer on the surface of samples which facilities the wear rate.

Shuguang *et al*. (2012) prepared $W_{14}Al_{86}$ powders in advance using MA technique alloying was performed on high energy ball mill with a rotational speed of 580 rpm. The ball-to-powder weight ratio was 15:1. Various contents of Al–$W_{14}Al_{86}$ powders were then blended with 0.2 vol % pure alcohol, as a process control agent, in a stainless steel vial filled with argon atmosphere for 1 h respectively. All the handling was performed under argon atmosphere in a glove box for avoiding oxidation of powders during MA treatment. The blended powders were then cold-pressed in a steel die under a pressure of 250 MPa to form green samples with a dimension of 40 mm×20 mm×10 mm. A vacuum hot-press machine was used to consolidate the green samples that have been enclosed in assembling graphite dice under argon atmosphere. The pressure in the die was kept at 25 MPa and the vacuum is 80 Pa in the furnace. Sintering temperature varied from 450 ^{o}C to 690 ^{o}C and meanwhile, sintering duration changed from 5 min to 30 min accordingly [8].

According to their results; *in situ* WAl_{12} intermetallic particle reinforced Al-based MMC was fabricated from Al–$W_{14}Al_{86}$ system *via* mechanical alloying and vacuum hot pressing (HP). WAl_{12} particulate is a intermetallic reinforcement, by increasing the weight percentage of WAl_{12}, both density and microhardness increased significantly.

Zohreh et al. (2014) used Ni, Ti and C powders were used as starting materials. Stoichiometric ratio of Ni–40 wt.% TiC was considered for the preparation of powder mixtures. Powder mixtures were milled by a planetary ball. The ball to powder weight ratio was chosen to be 10:1 and the diameter of the chromium steel balls was 15 mm. The hardened chromium steel vial was evacuated and filled with pure argon gas (99.99%) to prevent oxidation during the MA process with rotating speeds of 400 and 600 rpm. They considered two different approaches for powder preparation. The first approach is a double stage MA beginning with milling of Ni–Ti and Ni–C powder mixtures separately for 5 h in the first step. The mixture of the powders obtained from the first step was further MAed in the second step. In the second approach Ni, Ti and C powders were MAed together in a single stage MA process with rotating speeds of 400 and 600 rpm [9].

According to their results; MA of nickel, titanium and carbon powder mixture could result in a reactive powder with a thin layered structure. For the double step MA a milling speed of 600 rpm was needed to obtain the desired powder, while the direct single step with lower rotating speed of 400 rpm was suitable to prepare such powder. Resulting powders of both routes were capable of undergoing a reaction to form TiC during high temperature exposure. No traces of undesirable phases were observed after annealing of the powders.

2.2. Exothermic Dispersion (XD)

XD process includes clean particle–matrix interface, small particle size, high reaction rate and the easily available/low cost fabrication process. The displacement reactions between aluminum (powder or melt) and oxides have been chosen owing to lower cost of raw materials than conventionally used elemental powders [10-11].

Zhu *et al.* (2013) milled pure Fe, Ti and C powders in a stainless steel vacuum jar for 2 h and compacted into the cylindrical specimen with a diameter of 30 mm and a length of 5 mm at a pressure of 180 MPa. The cylindrical specimen were then heated to above 1473K in a vacuum furnace and then held at this temperature for 10 min and before cooling down to room temperature in the furnace [12].

According to differential scanning calorimetry results; the reaction between C and Ti occurs through one step and its activation energy is 2586.5 kJ/mol. Moreover, with the increase of the heating rate, the reaction peak shifts to the higher temperature. The whole reaction consists of three distinct stages: at the start, the reaction rate is very slow, and then it increases rapidly, finally it becomes slow again toward the completion.

Zhu *et al.* (2008) used Al, TiO_2 and B_2O_3 powder as raw materials. In powder blending, the B_2O_3/TiO_2 mole ratios were 0-0.5-1, respectively, in order to produce *in situ* composites. The powders were ball-milled in a stainless steel jar for 2 h and then compacted into green billets with a diameter of 30 mm. The billet was heated to above 1023K in a vacuum furnace to make the combustion reaction occur, held for 10 min and then subsequently cold down to the room temperature. Then, they were subsequently extruded to blades with a diameter of 6 mm, respectively, at an extrusion rate of 12:1 at 723K. They tested the composites to dry sliding wear tests in air at room temperature using a pin on-disc wear testing machine [3].

According to their results; the wear rate increases with the increase in sliding velocity, and when the sliding velocities increases to about 0.9 m/s, the wear rate increases to a maximum value and then decreases with further increase in sliding velocity. The wear resistance of the composites is improved by increasing the B_2O_3/TiO_2 mole ratios under the same wear test conditions. Without the B_2O_3, the reinforcements of the composite are composed of Al_2O_3 grains and Al_3Ti rods, and the composite exhibits poor wear characteristics. But when adding the B_2O_3, the wear resistance of the composites is improved significantly due to the decrease of the amount of Al_3Ti rods, and the increase of TiB_2 particles, and the refinement of the matrix grains and the great improvement in the mechanical properties of the composites. When the B_2O_3/TiO_2 mole ratio is increased up to 1, there is no Al_3Ti rod existing in the composites and the composites exhibit good wear resistance.

Durai *et al.* (2007) mixed the aluminum powder with 10 and 20 wt. % of ZnO powders with zirconia balls at 150 rpm using toluene medium in order to avoid oxidation or sticking of powder on the walls of the vial for 30 min. They were cold pressed the mixed powders at 650 MPa to form cylindrical green compacts with a diameter of 10 and 4 mm height. The compacts have been dehydrated for 2 h at 200 ^oC, before being heated to the reaction temperature of 950^oC for different sintering times in argon atmosphere.For each of material, tests have been conducted at two different nominal loads, 10 and 20N keeping the sliding speed fixed at 2.5m/s, for a test run of 2500 m [13].

According to their results; the hardness and density of the composites increase with the increasing sintering time and reinforcement volume fractions. The wear tests show that the wear resistance of Al MMCs increases with the increase in the reinforcement volume fraction and sintering time.

Zhu *et al.* (2007) used TiO_2, pure Al powder and B_2O_3 powder as raw materials. According to stoichiometric calculation, the mixed powders with 30% (volume fraction) reinforcements which B_2O_3/TiO_2 mole ratios were 0-0.5-1respectively were mixed by a ball-milling in the stainless steel vacuum jar for 2 hand then cold compacted into green billets with adiameter of 30 min. When the compacts were heated in vacuum furnace one by one at about 1073K, the combustion reaction occurred and held for about 10 min, and then the combusted compacts were cooled down to room temperature in the furnace [1].

According to their results; when the B_2O_3/TiO_2 mole ratio is below 1, the reaction products are composed of particle-like Al_2O_3, TiB_2 and rod-like Al_3Ti. The Al_2O_3 crystallites, resulting from the reaction between Aland TiO_2 or B_2O_3, are segregated at the grain boundaries due to a lower wettability with the matrix. SEM micrographs show that rod-like Al_3Ti phase distributes uniformly in the matrix. When the B_2O_3/TiO_2 mole ratio is around 1, the Al_3Ti phase almost disappears in the composites, and the distribution of Al_2O_3 particulates is improved evidently.

2.3. Reactive Hot Pressing (RHP)

RHP process is attractive due to its simplicity and flexibility. Ultrafine ceramic particulates are formed *in situ* by the exothermic reaction between the element constituents of composites under hot pressing conditions [14].

Roy *et al*. (2007) used aluminium powder (~20 µm) and nanosized TiO_2 powder (~12 nm) by dry milling were for preparing (Al + TiO_2) green powder mix. The powders were weighed separately and mixed in a small plastic jar containing 10 number of 4 mm steel balls. The weight of aluminium and nanosized TiO_2 powders was adjusted in such a way so that the composite formed by the complete *in situ* reaction would contain the expected

reinforcements (Ti-aluminide +Al_2O_3) content of 10, 20, 30, 40 and 50vol%. The powder mix, so prepared, was subsequently compacted in cylindrical (10mm dia.) graphite dies under an applied pressure of 25MPa in a hot press at 700-800-900 ^oC respectively. The pressing load on the specimen was applied for 15 min after the desired temperature was attained. The sample was then allowed to cool to room temperature and the cylindrical samples were stripped from the graphite die for characterization [15].

According to their results; aluminium matrix composites reinforced with well dispersed alumina and Ti-aluminide crystallites of size less than 1µm can be prepared by hot pressing the green compacts containing nanosized TiO_2 crystallites and commercially available aluminium powder. The fine reinforcements in the host matrix are formed by the *in situ* exothermal reaction between aluminium and nanosized TiO_2 crystallites during hot pressing. The initiation temperature of the *in situ* reaction decreases significantly with the use of nanosized TiO_2 crystallite. Evolution of the reinforcements (Ti-aluminide +Al_2O_3) by the *in situ* reactions is kinetically favored by increasing the temperature.

Tjong *et al*. (2003) used pure aluminum, TiO_2 and boron powders with an average size of 40, 60, 3 and 2 mm, respectively as raw materials. The amount of elemental powders were adjusted and blended so that the composites will have 20 vol. % reinforcement content. The powders were ball-milled in alcohol for 8 h and then dried. The cold compacted powder mixture was heated to above 800 ^oC in vacuum and held for 8 min, then cooled down to 600 ^oC and hot pressed. They were finally extruded at an extrusion rate of 20:1 at 420 ^oC. To determine the influence of *in situ* reinforcement on the thermal expansion behavior of composites, and the effects of thermal cycling between 50-300 ^oC, each cycle of thermal cycling process consisting of fast heating (15 ^oC/min) and relatively slow cooling rates (5 ^oC/min) [14].

According to their results; the *in situ* Al_3Ti plate and Al_2O_3 particle formed from the TiO_2-Al reaction leads to a decrease in the coefficient of thermal expansion (CTE) of Al. Moreover, *in situ* TiB_2 and Al_2O_3 particles developed from the TiO_2–Al–B system could further reduce the CTE of Al. Thermal cycling resulted in a net dimensional change of pure Al on the basis of the analysis of the thermal strains in the hysteresis loop. However, *in situ* composites exhibited a lower degree of damage compared to Al during thermal cycling.

Tjong *et al*. (2003) used Al, TiO_2 and boron powder as starting materials. *In situ* composites with a nominal composition of 20 vol. % reinforcement were prepared by adjusting the molecular ratios of B/TiO_2 to 5/3 and 6/3. In the process, the powders were ball-milled in alcohol for 8 h and then dried. The cold compacted powder mixture was heated to above 800 ^oC in vacuum and maintained for 10 min, then cooled down to 600 ^oC and hot pressed. The pressed billets were extruded at an extrusion ratio of 20:1 at 420 ^oC. Fatigue test specimens were machined from the as-extruded bars with the loading axis parallel to the extrusion direction. The gauge length and diameter of the specimens are 16 and 4 mm, respectively. Fatigue tests were performed with 100 kN load cell. Low-cycle-fatique (LCF) tests were conducted under fully reversed axial tension-compression loading. The tests were controlled under various total strain amplitude using a triangular push-pull wave shape and a constant nominal strain rate of $2x10^{-3}$/s. The tests were continuously run to fracture the specimens [16].

According to their results; the composite reinforced with *in situ* TiB_2 and Al_2O_3 particles exhibits a relatively stable cyclic response at low total strain amplitudes. At higher total strain amplitudes, cyclic softening from the onset of deformation was observed. However, the presence of Al_3Ti blocks led to a very slight cyclic hardening followed by softening at total strain amplitude of 0.4%. Moreover, the intermetallic Al_3Ti blocks reduced the fatigue life of *in situ* composites as they promoted microscopic cracking during cyclic deformation.

3. Assessments

Mechanical alloying is a process providing cost-effective and homogeneous microstructure.

By exothermic dispersion process, ultrafine ceramic particles can be produced in matrix.

To enhance the density of composites, reactive hot pressing is a suitable process.

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