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In-Situ Aluminothermal Reduction Synthesis of Ti_3AlC_2 Aluminium Composite by Friction Stir Processing

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Abstract

Max phase Ti_3AlC_2 composite, which generally requires high energy ball milling and reactions at higher temperature, has been synthesized at lower temperatures by Friction Stir Processing (FSP). To enable this TiO_2+C mixture is dispersed into aluminium matrix by FSP. FSP is a high strain rate plastic deformation process used for microstructural refinement in which a rotating non-consumable tool mixes the work piece material while moving forward. Six overlapping passes of FSP were done to disperse stoichiometric TiO_2+C mixture in to Aluminium matrix. Optical microscopy and scanning electron microscopy was done to confirm uniform distribution of particles. X-ray diffraction (XRD) was used for phase identification. XRD of as-processed sample showed peaks of Al, TiO_2 and C indicating absence of reaction during FSP. Differential Scanning Calorimetric (DSC) analysis done on this processed sample showed exothermic peaks at 633°C (below melting point of Al), 710°C (above melting point) and 951°C (above melting point). To evaluate the reactions at these temperatures, processed samples were heated to respective temperatures. For samples heated to 633°C, Ti_3AlC_2, Al3Ti, TiC, Al phases has been observed along with unreacted TiO_2 and C. For samples heated to 710°C, peaks of Ti_3AlC_2, Al_3Ti, Al and C were observed. For sample heated to 951°C, only Ti_3AlC_2 and Al_2O_3 phases were observed besides Aluminium (Fig. 1). Interestingly, TiC impurity normally associated with Ti_3AlC_2 synthesis was absent for the sample heated to 951°C. It is postulated that due to particle refinement during FSP, activation energy of the reaction is lowered. This lowering of activation energy also led to formation of Ti_3AlC_2 and TiC phases even for samples heated to 633°C. Based on the results a reaction path has been proposed.

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Keywords: Friction Stir Processing; Max phases; Aluminothermal reduction; Metal matrix composites; XRD

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1. Introduction

MAX phases are ternary carbides or nitrides, where M is translational metal; A is group IIIA or VIA element; and X is either carbon or nitrogen. These ternary phases combine unique features of both ceramics and metals. Like ceramics they are hard and light, with high modulus and high temperature strength and like metals they can resist thermal shock, are ductile, tough, easily machineable and electrically and thermally conductive[1]. Due to these interesting combinations of properties, there have been a lot of interest in synthesis of these phases, especially titanium based MAX phases. Of the possible Titanium based MAX phases, carbide phase Ti_3AlC_2 is of greater interest due to high temperature structural and functional applications [2]. Ti_3AlC_2 was synthesized by Nikolay V. Tzenov et al by ball milling of Titanium, graphite and Al_4C_3 powders for 12 hours and Hot Isostatic Pressing at 1400°C for 16hrs [3]. Ti_3AlC_2 was also synthesized by solid-liquid reaction, which has reduced the processing time considerably [4]. Ti_3AlC_2 and Al_2O_3 composites have been synthesized by Aluminothermal reduction of TiO_2 [5-6]. Reaction temperature to synthesize Ti_3AlC_2 using TiO_2 was above 900°C. Feasibility study on synthesis of Ti_3AlC_2 from elemental powders were done by M Zakeri et al and have found that a mechanical activation by ball milling for 6 hours, followed by heat treatment at 1100°C yields Ti_3AlC_2 and $Ti_4Al_2C_2$[7].

As stated, MAX phases are hard with high modulus and can be machined easily; hence they have many advantages over ceramic reinforcements which are widely used in metal matrix composites. W.J. Wang et al have reported the compressive yield strength of Ti_3AlC_2/Aluminium composites to be twice as that of aluminium matrix even at high temperatures [8]. For MAX phases to be successfully used as reinforcements, synthesis temperatures of MAX phases needs to be lowered. Synthesis temperature is largely dictated by activation energy of the reaction. The activation energy of reaction can be reduced by providing mechanical activation to reactants. Recently mechanical activation by FSP has been successfully used to produce in-situ composites [9-11]. FSP is a microstructure refinement tool, where a rotating tool with a cylindrical or conical probe is plunged into work piece. Due to friction, work piece softens; rotating probe induces large plastic deformation, which leads to microstructural refinement [12].

In this study TiO_2+C mixture is used to fabricate aluminium matrix composite. FSP is used as tool for mechanical activation. Evolution of different chemical phases at salient thermal events is explored. Study aims to answer following questions: (1) is it possible to synthesize titanium based MAX phase using FSP (2) or will mechanical activation of FSP possibly reduce reaction temperature to synthesize MAX Phase.

2. Experimental methods

In this study, commercially Pure (CP) aluminum plate of 6 mm thickness, 60 mm wide and 150mm long, with 3 mm wide and 4 mm deep groove was used as matrix. The Nano crystalineTiO$_2$ was prepared in house with process described by Mahshid, et. al. [13]. Graphite particles of 100µm used in this study was supplied by S. D. fine-Chem limited. Stoichiometric mixture of TiO_2 and graphite was thoroughly mixed and packed into groove. Groove was closed with 2 mm thick Al strip. A threaded steel tool, with 1mm pitch, 5mm height and 30 mm shoulder, was used for 6 overlapping passes on work piece. FSP was done on a five axis Friction Stir Welding machine (BISS-TWI, Bangalore) with a tool rotation speed of 1200 rpm and a traverse speed of 25 mm/min with 2° tool tilt.

Transvers section of the composite was cut for microstructural analysis. Microscopy was used to confirm even distribution of the particles. The processed composite was heated at a rate of 10°C/ min in Perkin-Elmer DSC-7. X-ray diffraction (XRD) analysis was done on PANalytical (JDX-8030) using CuKα radiation. Samples were ground with silicon carbide papers and polished with alumina prior to Scanning Electron Microscopy (SEM).

3. Results

3.1. Microstructure and particle distribution of Al-TiO$_2$-C composite

Stereo microscope image of the composite shows uniform distribution of TiO_2 and graphite into aluminium matrix (Fig.1). The SEM micrographs shown in Fig. 2(a-c) also confirm uniform distribution of the particles. There