Advanced Powder Technology 23 (2012) 343-348



Contents lists available at ScienceDirect

## Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

# Original Research Paper Role of intensive milling in mechano-thermal processing of TiAl/Al<sub>2</sub>O<sub>3</sub> nano-composite

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#### ARTICLE INFO

Article history: Received 6 July 2010 Received in revised form 19 March 2011 Accepted 18 April 2011 Available online 7 May 2011

Keywords: Mechanical alloying Nano composite Aluminothermic reduction TiAl

#### ABSTRACT

In this research a nano-composite structure containing of an intermetallic matrix with dispersed  $Al_2O_3$  particles was obtained via mechanical activation of  $TiO_2$  and Al powder mixture and subsequent sintering. The mixture has been milled for different lengths of time and then as a subsequent process it has been sintered. Phase evolutions in the course of milling and subsequent sintering of the milled powder mixture were investigated. Samples were characterized by XRD, SEM, DTA and TEM techniques.

The results reveal that the reaction begins during milling by formation of  $Al_2O_3$  and  $L1_2$   $Al_3Ti$  and further milling causes partial amorphization of powder mixture. DTA results reveal that milling of the powder mixture causes solid state reaction between Al and TiO<sub>2</sub> rather than liquid–solid reaction. Also, it was observed that the exothermicity of aluminothermic reduction is reduced by increasing the milling time and the exothermic peak shifts to lower temperatures after partial amorphization of powder mixture during milling. Phase evolutions of the milled powders after being sintered reveal that by increasing the milling time and formation of  $L1_2$   $Al_3Ti$  in the milled powder, intermediate phase formed at 500 °C changes from  $D0_{22}$   $Al_3Ti$  to  $Al_{24}Ti_8$  phase.

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#### 1. Introduction

Intermetallic alloys based on  $\gamma$ -TiAl are interesting materials for use in aerospace and structural applications for their relatively low density, high strength to weight ratio, good oxidation and corrosion resistance and adequate creep resistance at high temperatures. However, their brittleness and rapid crack growth rate at low to intermediate temperatures hinders their application [1,2]. It has been reported that grain refinement of these alloys to nano-scale may improve their room temperature ductility [3-6]. Nevertheless, nano-structure of the monolithic allovs is unstable at elevated temperatures which deteriorate the high temperature properties. Preparation of a nano-composite with intermetallic matrix and dispersed ceramic particles with thermodynamically compatible phases may stabilize the nano-structure at high temperatures [3,4]. Also, reinforcement of the intermetallic matrix with high strength ceramic particles improves high temperature creep strength and modifies fracture behavior to provide improvements in ambient temperature toughness [7].

There have been interests on development of in situ TiAl/Al<sub>2</sub>O<sub>3</sub> composite in recent years to overcome the problems associated with the monolithic  $\gamma$ -TiAl alloys [8]. TiAl/Al<sub>2</sub>O<sub>3</sub> and/or Al<sub>3</sub>Ti/

 $Al_2O_3$  in situ composites have been already prepared by mechanical alloying and subsequent heat treatment of  $TiO_2$  and Al powders by a number of research groups [9–12]. In most of these researches, no reaction was reported to take place in Al– $TiO_2$  powder mixture during the milling process. Only in one case under a severe milling condition, formation of a fcc TiAl phase was reported [13] although formation of this phase is in doubt by some researchers [14]. Therefore, phase evolutions of Al– $TiO_2$  powder mixture during intense milling process to prepare  $TiAl/Al_2O_3$  nano-composite has not been thoroughly investigated yet. It is expected that final phases formed after sintering of the powder mixture is related to the starting powder mixture composition. Also, formation of intermediate phases during the milling process may affect the phase evolution paths and related temperatures during sintering.

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In this research,  $\gamma$ -TiAl/Al<sub>2</sub>O<sub>3</sub> nano-composite has been synthesized using mechano-thermal processing of Al–TiO<sub>2</sub> powder mixture. Phase evolutions during the milling and further heat treatment stages were systematically studied.

#### 2. Methods

Commercially pure  $TiO_2$  (<0.2 µm, 99.8%), and Al (<100 µm, 99.8%) reagents have been precisely weighed according to the following reaction, even though 10 mol% excess Al powder was used to ensure the existence of sufficient Al for the reaction.

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