MECHANICAL ALLOYING AS A WAY TO PRODUCE ALUMINUM MATRIX COMPOSITE WITH TiB_2 PARTICLES AS A REINFORCEMENT

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Abstract

Solid state mixing, such as mechanical alloying (MA), represents technologically reasonable economical processing method how to achieve homogenous distribution of hard particles in composite microstructure. The advantage of working at ambient temperatures, without a process control agent (PCA), opens new route for the preparation of advanced materials. The use of mechanical alloying includes several advantages, namely control of the particle size or the compatibilization of hard immiscible blends. The aim of the current work is to prepare TiB_2 phase dispersion in aluminum matrix using mechano-chemical processing. For this purpose a mixture of aluminum, boron trioxide and titanium powders were subjected to high energy ball milling. The microstructural and phase analyses of powders particles after different milling times and after addition of aluminum (two-step mechanical alloying) was conducted by using scanning electron microscopy (SEM), X-Ray diffractometry (XRD) and scanning differential calorimetry (DSC). The results indicate that two-step milling method led to the production of aluminum matrix composite with a small amount of fine TiB2 particles distribution.

Keywords: Mechanical alloying, aluminum matrix composite, mechano-chemical processing, composite

1. INTRODUCTION

Metal matrix composites (MMCs) are materials where hard ceramic and/or intermetallic reinforcement are embedded into a ductile metal or metal alloy matrix. MMCs combine best properties of its constituent materials i.e. ductility and toughness of metal with high strength of ceramic or intermetallic particles. Aluminum is taken as one of the most popular matrix for MMCs. The Al alloys are attractive owing to their low density, their capability to be strengthened by precipitation, good corrosion (antioxidation) resistance and high thermal and electrical conductivity [1].

Mechanical alloying (MA) is a type of high energy processing for solid - state powders, in which elemental or alloy powder particles are repeatedly deformed. The MA processing is often used to obtain dispersion-strengthened aluminum alloys under the presence of a process control agent (PCA). The main role of PCA is to establish a balance between fracture and welding of powder particles. A PCA is occasionally added, especially when ductile materials are being milled, to minimalize the effect of cold welding and consequent formation of large pieces of powder. The MA process involves loading of the blended elemental powders along with the grinding medium in a vial and subjecting the mass to deformation. During the milling process, the powder particles are repeatedly flattened, cold welded, fractured and re-welded. The processes of cold welding and fracturing, their kinetics and predominance at any stage of milling depend on the deformation characteristics of the starting powders. The incipient impact of the milling ball causes the ductile metal powders to flatten and work-harden. The severe plastic deformation increases the surface to volume ratio of the particles and breaks the surface films of adsorbed contaminants. The brittle ceramics or intermetallic powder particles are getting fractured and refined [2, 3].During the collision between the milling balls and powder particles, the powder morphology can be modified in two ways. In the case the starting powders are soft, the flattened layers overlap and form cold welds. This leads to formation of layered composite powder particles composing of different combinations of the starting materials. The work-hardened elemental powder particles may be broken at the same time. The competing events of cold welding and fracturing continue repeatedly throughout the milling time. Eventually, the steady-state is reached when refined and homogenized microstructure is obtained and the chemical composition of the individual powder particles is comparable with the starting ratio of elements [4].

Mechano-chemical processing (MCP) can be defined as a powder processing technique involving deformation, fracturing and cold welding of the particles during repeated collision with a ball during high-energy ball milling [5]. The MCP involve loading of the mixed elemental or pre-alloyed powders along with the milling medium in a vial and subjecting the material to heavy deformation [6].Most of the mechano-synthesis reactions studied have been displacement reactions of the type:

$$MO + R \rightarrow M + RO \quad (1)$$

where a metal oxide (MO) is reduced by a more reactive metal (reductant, R) to a pure metal M. Metal chlorides or sulfides have also been reduced to pure metals in this this way [2].

TiB_2 (titanium diboride) is a potential choice as a second phase dispersion for MMC. TiB_2 as a refractory compound could be an attractive reinforcement for a range of applications possessing high modulus, excellent refractory properties and chemical inertness. The thermal expansion behavior of a metal matrix composite is influenced by several material parameters such as the type of constituents and their volume fractions, the thermal stresses between the constituents due to their coefficient of thermal expansion (CTE) mismatch, and the microstructure of composites [7].It was showed that composites with large particles (100 μm) have low density and poor thermal conductivity (TC) due to formation of pores at the interfaces [8].This phase is suitable as a reinforcing phase for Al based composites because of its thermodynamic stability [9, 10].Moreover, TiB_2 particles do not react with aluminum, thereby avoiding the formation of brittle reaction products at the reinforcement-matrix interface. They can be introduced into the aluminum matrix via casting and powder metallurgy (PM) routes. In the casting route, agglomeration of ceramic particulates often occurs during processing, leading to poor mechanical strength of the composites. A more uniform distribution of the ceramic particles in the Al matrix can be achieved via PM processing [11].

In this work, using Al - B_2O_3 - Ti mixture as precursor materials synthesis of aluminum matrix composites powder Al/TiB_2 was carried out via mechano-chemical process in high-energy planetary ball mill. Moreover, the effect of Al addition during the milling on formation of the MMCs is investigated.

2. EXPERIMENTAL

Elemental powders of Al (10 wt.%, particle size 25 - 65 μm, GTV), Ti (62 wt. %, purity 99.5 %, particle size < 45 μm, Alfa Aesar) and boron trioxide (28 wt.%, purity ≥ 98.5 %, particle size < 200 μm, Sigma Aldrich) were used as a starting materials.

Ball milling of the powder mixture (total weight 49 g) was carried out in a high - energy planetary ball mill (Fritch Pullverisette 6) at room temperature and under inert atmosphere (Ar). Milling media/balls were hardened chromium steel vial with hardened carbon steel balls (having diameter of 15 mm). The ball - to - powder weight ratio was kept to be 10:1. The milling process was interrupted at predetermined time intervals and small amount of powder was removed for characterizations. After 20 hrs of milling, additional aluminum powder (73 g) was added to the mixture and milling was than carried out for another 20 hours. This procedure has been called as two-step milling method.

Microstructures of powders were examined by using ZEISS Ultra Plus scanning electron microscope (SEM) that operated at the accelerating voltage of 10 kV. Since heavy elements can be seen more strongly in back scattered electron (BSE) mode comparing to light elements and, they appear to be brighter in the image, the BSE mode was utilized to detect contrast between areas with different chemical compositions. Energy Dispersive X-Ray Spectrometry (EDS) was also employed to investigate the chemical composition in selected areas. Phase transformation during milling was evaluated based on X - ray diffractions (XRD) of the powder samples in a Philips X`Pert diffractometer using filtered Cu Kα radiation (λ = 0.15406 nm). The operation was performed at 40 kV and 30 mA over a 2Θ range from 25° to 100°. „PANanalytical X`Pert High Score“ software was also employed to compare the XRD profiles with standards compiled by the Joint Committee on Powder Diffraction and Standards (JCPDS).

3. RESULTS AND DISCUSSION

3.1. XRD analysis and crystalline size

Figure 1 shows the XRD patterns of the Al - Ti - B_2O_3 powder mixture with different milling time. As can be seen after 2 hours milling the TiB_2 phase is created. Another peaks correspond with Ti and peaks with very slow intensity corresponding with aluminum. After 10 hours of milling the width of all peaks increased and their intensities reduced. This is cause to the refinement of the crystalline size and the enhancement of lattice strains during mechanical alloying. The Ti and Al peaks were not shifted during the procedure. This indicate that no Ti and Al solid solution was formed. Although the equilibrium solubility of Ti in Al at room temperature is insignificant, the solubility of Ti in the Al lattice can be increased up to 3 wt. % using mechanical alloying [12]. After addition of another aluminum can be seen mainly signals of Al.

The composition of the powder mixture after 40 hours of milling was as follow: the TiB_2 phase 3 wt. % and the crystalline size was 8.2 nm and aluminum was 97 wt.% with crystalline size 43.8.

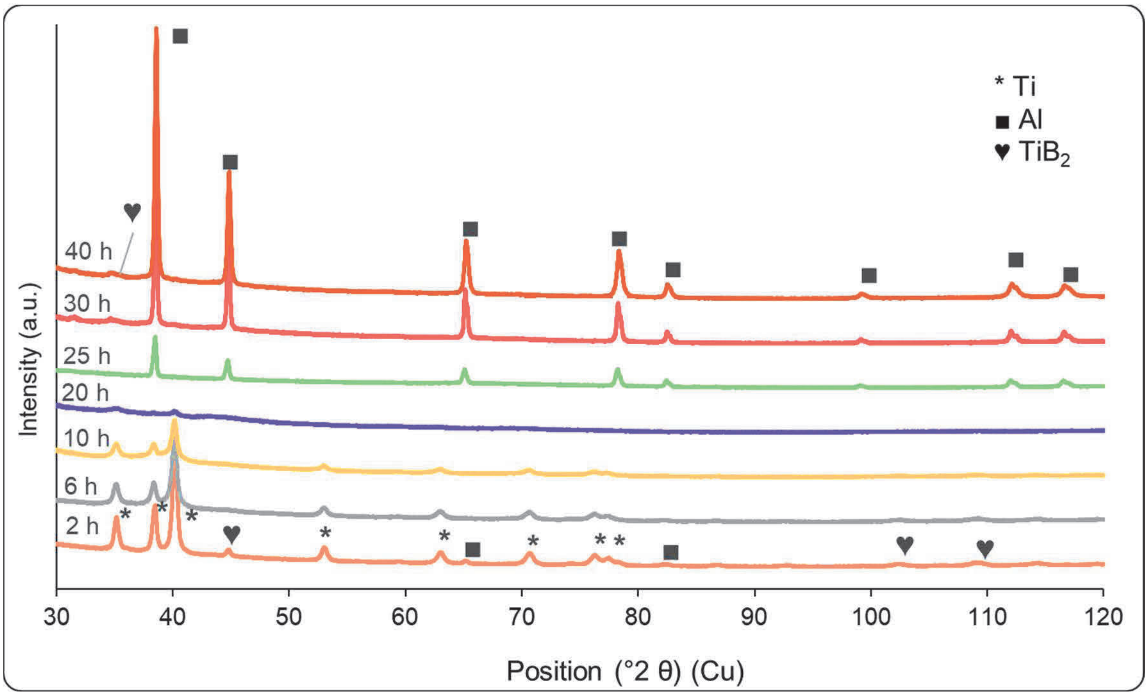

Figure 1 The XRD patterns of sample milled for 2, 6, 10, 20, 25, 30 and 40 hours

3.2. Microstructure analysis

At the beginning of the milling process, the ductile Al and Ti powders have gotten flattened by milling balls collisions, while the brittle B_2O_3 got fragmented. **Figure 2** shows a typical powder particle cross - sectional micrographs. In **Figure 2 (a)** powder mixture milled for 2 hours is shown. The EDS analysis (**Figure 6**) finds out that the light grey particle is Ti and the grey color represents Al - Ti solid solution phase. The titanium particles were detected in every stage of the milling process as well as the Al - Ti phase. After 6 hours of milling TiB_2 phase can be detected. In **Figure 2 (b)** the very small darkest grey particles represent this phase. The TiB_2 phase was observed until the end of milling.

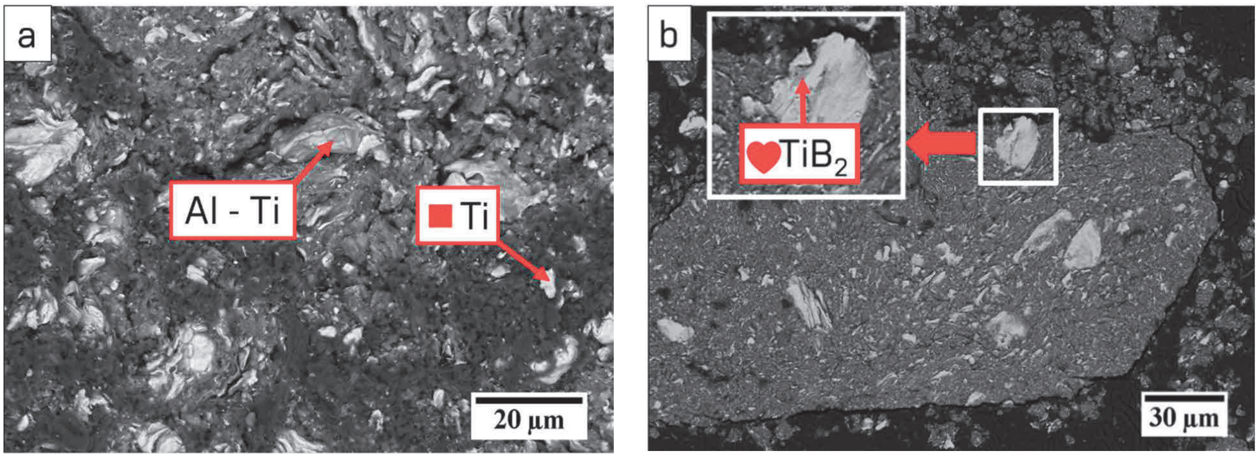

Figure 3 A cross-sectional SEM microstructures of powder mixture milled for (a) 2 and (b) 6 hours

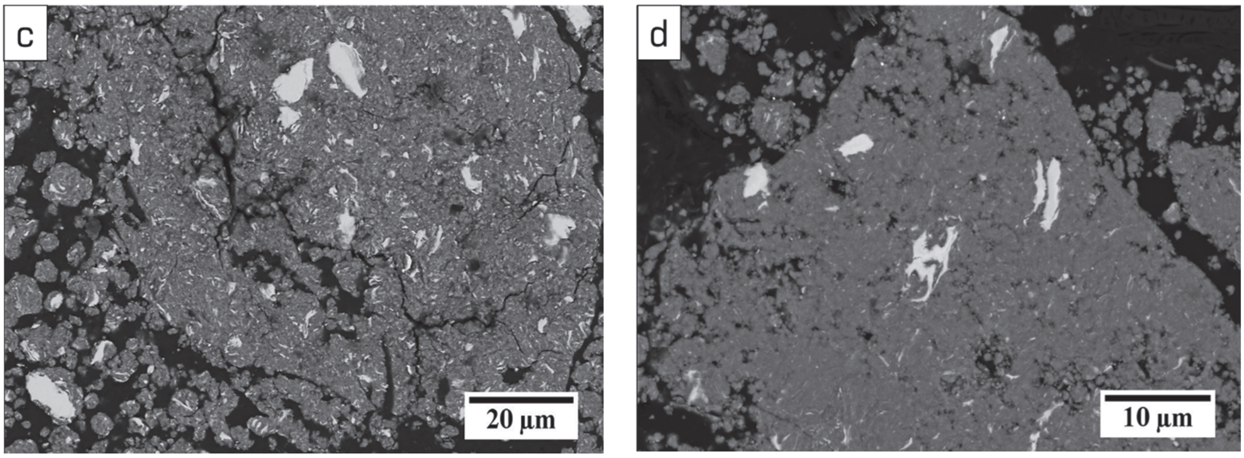

Figure 4 The cross-sectional SEM microstructures of powder mixture milled for (c) 10 and (d) 20 hours

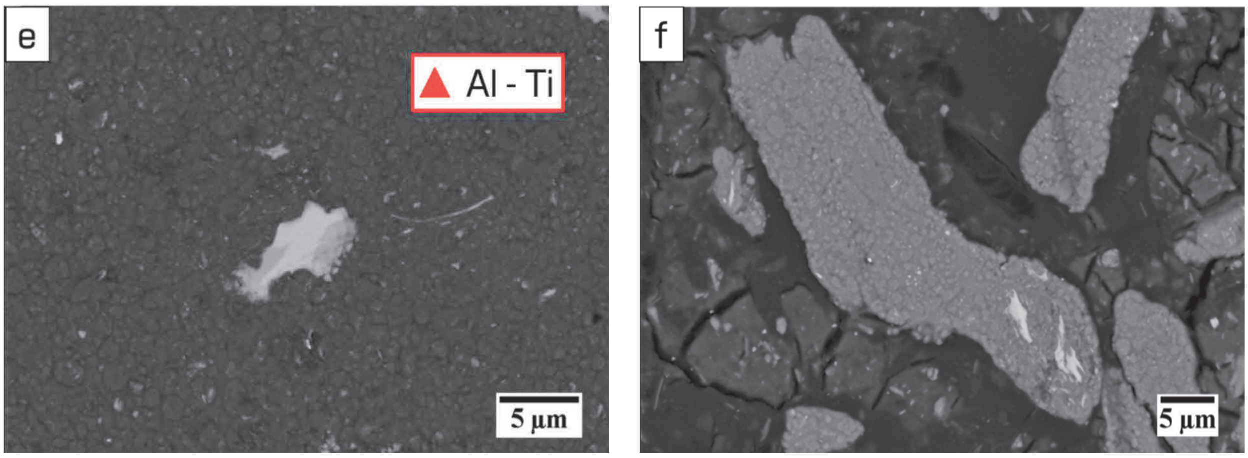

Figure 5 The SEM microstructures of powder mixture milled for (e) 25 and (f) 30 hours

Figure 3 shows the microstructure of powder mixture after 10 and 20 hours of milling, respectively. In this stage of milling the titanium particles in Al - Ti solid solutions can be seen very clearly. These particles are the light grey to white. The composite microstructure was refined as a result of the repeated cold welding and fracturing of powder particles.

After 20 hours ball milling, the aluminum powder was added to the pre-milled Al - Ti - TiB_2 powder. Milling continued for another 20 hours. **Figure 4** shows micrographs of powder mixture milled for 25 and 30 hours. As can be seen in **Figure 4 (e)**, the Ti particles are present in the Al - Ti matrix. This is also obvious for **Figure (f)**.

Figure 5 shows the final micrographs after total milling time of 40 hours. The EDS analysis showed that very small particles were TiB_2 phase. In addition, Ti particles are included in Al - Ti matrix along with TiB_2.

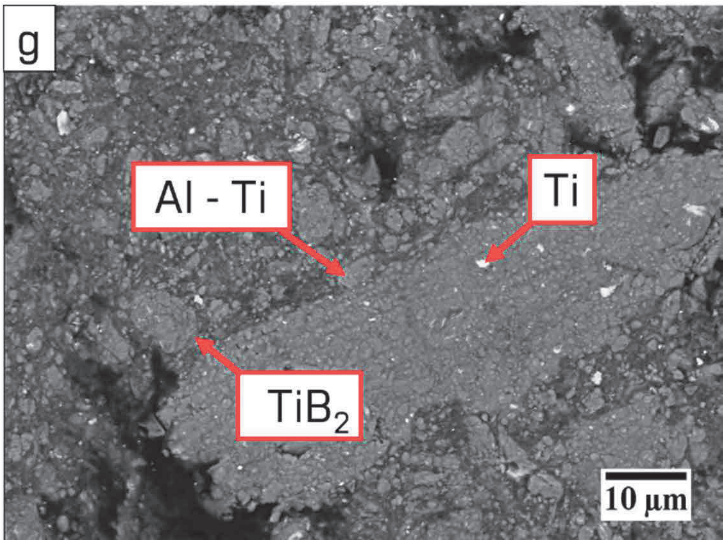

Figure 6 A cross - sectional SEM micrographs of powder mixture milled for (g) 40 hours

In **Figure 6** EDS analyses of selected locations are shown in dependence of the milling time (2, 6 and 25 hours). The EDS results for the milled powder mixtures qualified the Al - Ti solid solution, titanium and TiB_2 particles in different parts of microstructure. Moreless, these EDS results correspond with results from X-ray diffractometry.

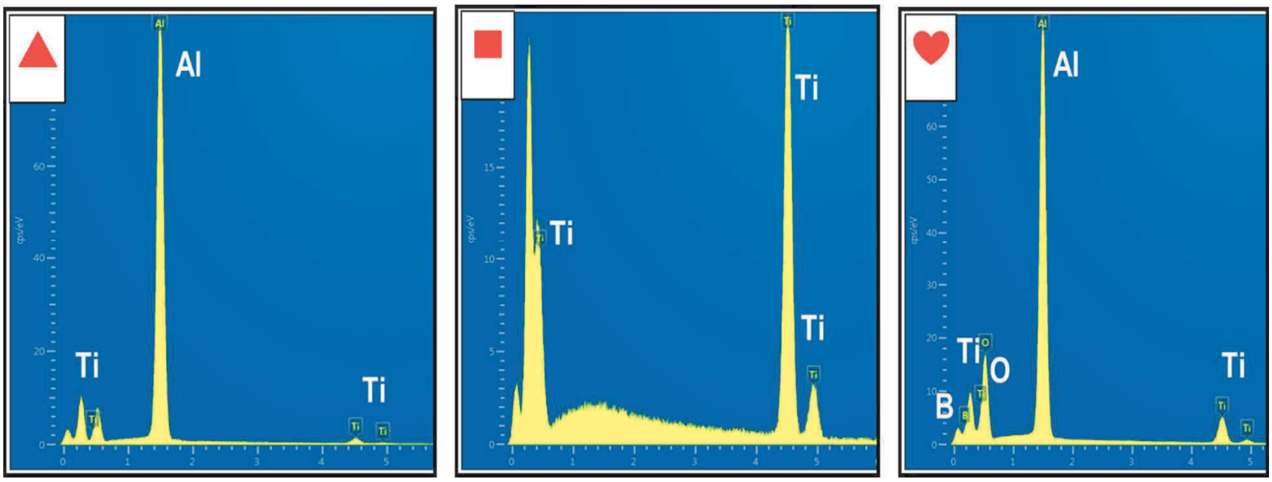

Figure 7 EDS analysis of selected areas of Al - Ti; titanium and TiB_2

4. CONCLUSION

In this work, the possibility of preparation TiB_2 phase in aluminum matrix with using mechano-chemical reaction in high - energy planetary ball mill was studied. The Al - TiB_2 composites can be synthetized from the aluminum, titanium and boron trioxides powders. The approach, which was used to obtain aluminum matrix composite powder containing TiB_2 particles as reinforcement, was two-step milling. In the first step, titanium and boron trioxide powders were milled with small amount of aluminum (3 g). Based on the obtained data the milling up to the 10 hours led to the formation Al - Ti matrix formed by Ti - Al solid solution with TiB_2 particles as a reinforcement. With increasing of milling time, the Bragg peaks of Ti, Al and TiB_2 became wider. This broadening is caused by reduction of the crystalline size. The product of the first stage was than subjected to further milling with additional aluminum powder (73 g). During the ball milling the Ti particles were mechanically alloyed in the aluminum matrix and the part of Ti particles reacted with boron trioxide and TiB_2 phase was created. At the end of the milling, 3 wt.% of the TiB_2 phase was present in the aluminum matrix.

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