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## Production and microstructural investigation of Al-nano TiB<sub>2</sub> composite produced by powder metallurgy technique

### ABSTRACT

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Automotive and aerospace industries are required to light materials which have high properties. This causes to reduce production cost and fuel consumption. One of these advanced materials that have been attracted at these decades is particulate metal matrix nanocomposites. These materials have a combination of ductile matrix alloy properties and ceramic reinforcement nanoparticles. Powder metallurgy is one of the significant methods to fabricate this kind of materials. In this research, aluminum alloy matrix nanocomposites reinforced with 1.5, 2.5, 3.5, 5, and 10 Vol. % TiB<sub>2</sub> nanoparticles were fabricated via powder metallurgy method. Fabrication was performed at two different sintering temperatures, viz. 650 and 680 °C. Optimum amount of reinforcement and sintering temperature was determined by evaluating density and microstructural studies of nanocomposites. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis illustrated that distribution of nano TiB<sub>2</sub> particles in aluminum matrix is suitable. Also, density results showed that with increasing the TiB<sub>2</sub> volume percent, density of composites increases.

**Keywords:** *Aluminum matrix composites; Nano TiB<sub>2</sub>; Microstructure; Powder metallurgy; Density.*

### INTRODUCTION

Generally, composite materials are divided into three major categories viz., metal matrix composites, polymer matrix composites and ceramic matrix composites [1]. Metal matrix composites are considered as a group of advanced materials which represent low weight, high strength, high modulus of elasticity, low coefficient of thermal expansion and good wear resistance. These characteristics could not be achieved together in monolithic materials [2].

A variety of methods have been conducted to produce aluminum MMNCs such as infiltration [3], squeeze casting [4], mechanical alloying [5], ball milling [6], stir casting [7], and powder metallurgy [8].

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Powder metallurgy technique for producing metal matrix composites (MMCs) has been developed to manufacture a wide range of engineering components. The one of the best properties of powder metallurgy for fabrication of composites can be obtained when the reinforcement is homogeneously dispersed in the matrix, as approved by experimental and theoretical studies [9–11]. Therefore, it could be nominated as one of the methods proper for fabricating these kinds of materials [12].

Aluminum is one of the best materials for matrix because of its low density, corrosion resistance, high conductivity and high toughness [13]. TiB<sub>2</sub> is a ceramic with high hardness (25-35 GPa @ R.T.) and elastic modulus (560 GPa), good abrasion resistance, chemical neutrality, good wettability with molten metal's, and high temperature resistivity. Hence, TiB<sub>2</sub> could be regarded as an excellent choice to produce composite [14-16].

In the present research, the effects of sintering temperature and TiB<sub>2</sub> content on density, microstructure and morphology of an Al/ TiB<sub>2</sub> composite produced by powder metallurgy method, was investigated. Eventually, the best conditions in order to obtain the most useful properties were introduced.

## EXPERIMENTAL

Al-TiB<sub>2</sub> nanocomposite was produced via powder metallurgy method using aluminum, powder with a purity of 99.97% and D<sub>50</sub> = 20 μm, as the matrix material and TiB<sub>2</sub> nanoparticles with D<sub>50</sub> = 80 nm as the reinforcement. The fabrication steps in powder metallurgy route were: Al powder alloy has been mixed with TiB<sub>2</sub> nanoparticles, and then cold isostatic pressing machine (CIP) was employed to produce the samples. Finally, green compacts were sintered at two different temperatures of 650, and 680 °C. Various TiB<sub>2</sub> contents of 1.5, 2.5, 3.5, 5 and 10 vol. % have been considered for each processing temperatures.

X-ray diffraction (XRD) method was performed for crystal structure and phase investigation via Philips PW1800 X-ray diffractometer (Cu-K<sub>α</sub> radiation, λ = 1.5405 Å, 40 kV, 30 mA). Bulk density was determined by

Archimedes method. Finally, morphological aspects of the samples have been investigated by scanning electron microscope (SEM) using Oxford CAMSCAN-MV 2300.

## RESULTS AND DISCUSSION

### XRD Phase Analysis

Figure 1 shows the XRD pattern of Al-3.5 TiB<sub>2</sub> nanocomposite sintered at 680 °C. All peaks could be indexed as cubic (fcc) aluminum (JCPDS #4-0787) and hexagonal TiB<sub>2</sub> (JCPDS #8-0121). No further crystallographic structure was detected in XRD pattern. This shows that no substantial interactions take place between base metal and the reinforcement (because of resistance of TiB<sub>2</sub> to molten aluminum, as mentioned before) phase during sintering which may result in formation of intermetallic phases. Since TiB<sub>2</sub> is resistant to oxidation up to 1100 °C [14], no peaks related to oxide phases like TiO<sub>2</sub>, TiBO<sub>3</sub>, B<sub>2</sub>O<sub>3</sub>, etc. were detected. However, nanometric TiB<sub>2</sub> particles are sensitive to surface oxidation due to high surface area that affects their wettability and sinterability -although, it is not so acute [17]. Undetected aluminum oxide peaks implying minor oxidation of aluminum powder as well. The relatively intense peaks of TiB<sub>2</sub>, is a sign of almost uniform distribution of ceramic particles in metal matrix.

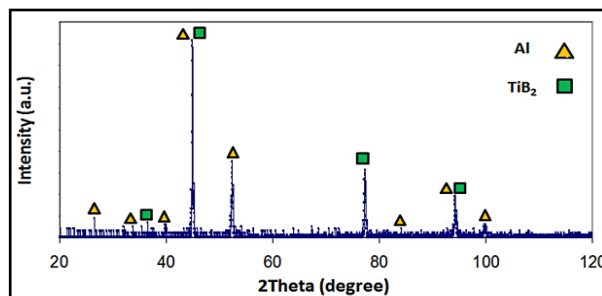


Fig. 1. XRD pattern of Al-3.5TiB<sub>2</sub> nanocomposite sintered at 680°C.

### Microstructure and Morphology

Scanning electron micrographs of the Al/TiB<sub>2</sub> nanocomposites with different amounts of reinforcement phase, sintered at 650 and 680 °C are presented in Figs. 2(a-e) and 3(a-e). The images are taken in BSE (back-scattered electron) mode in

order to distinguish between different phases based on the difference of average atomic numbers of each phase, mainly aluminum and  $\text{TiB}_2$ . In this respect, the notable microstructural phenomena are agglomerations and porosity voids which appear as lighter and darker areas compared with ambient gray color of background, respectively. As mentioned,  $\text{TiB}_2$  has a higher density than aluminum matrix and tends to appear brighter, accordingly. Notwithstanding, uniform distribution of reinforcement particles of  $\text{TiB}_2$  is attained in non-agglomerate areas (matrix phase). In the set of images for samples sintered at  $650^\circ\text{C}$  (Figure 2), it could be observed that with increasing the volume percent of  $\text{TiB}_2$  the scale of agglomeration and consequent microstructural deteriorations as porosity and crack increases. The agglomerated regions are appeared as brighter  $\text{TiB}_2$ -rich particles in the rather darker background which chiefly consists of aluminum. Agglomeration could be a result of localized distribution of powders during combination because of the difference in size and density of aluminum and  $\text{TiB}_2$  powders [18]. The porosity of samples tends to grow and propagate with increasing the amount of  $\text{TiB}_2$  corresponding the agglomeration and retarded sintering which confirms the results of density measurements.

The trend of microstructural characteristics in the set of images for samples sintered at  $680^\circ\text{C}$  (Figure 3) is almost the same as the samples sintered at  $650^\circ\text{C}$ , that is, the  $\text{TiB}_2$ -richer samples are more prone to microstructural deteriorations. However, the overall amount of porosity has been decreased in the samples sintered at  $680^\circ\text{C}$  due to amended sintering and coalescence of green compacts. Such contrast is especially more evident in corresponding samples (in 650 and  $680^\circ\text{C}$ ) with lower amounts of  $\text{TiB}_2$ . Microstructural deteriorations are more severe in case of  $\text{Al-5TiB}_2$  and  $\text{Al-10TiB}_2$  samples - as appears in the both sets of images (Figure 2 and Figure 3) - which would affect the mechanical properties of nanocomposites. This implies that the conditions of preparing composites should be adjusted with increasing the amount of reinforcement phase to achieve equivalent qualities and properties.

Finally, In spite of regional agglomerations, a uniform distribution of reinforcement particles could clearly be observed in the map images, which is in agreement with XRD results and previous explanations of SEM images.

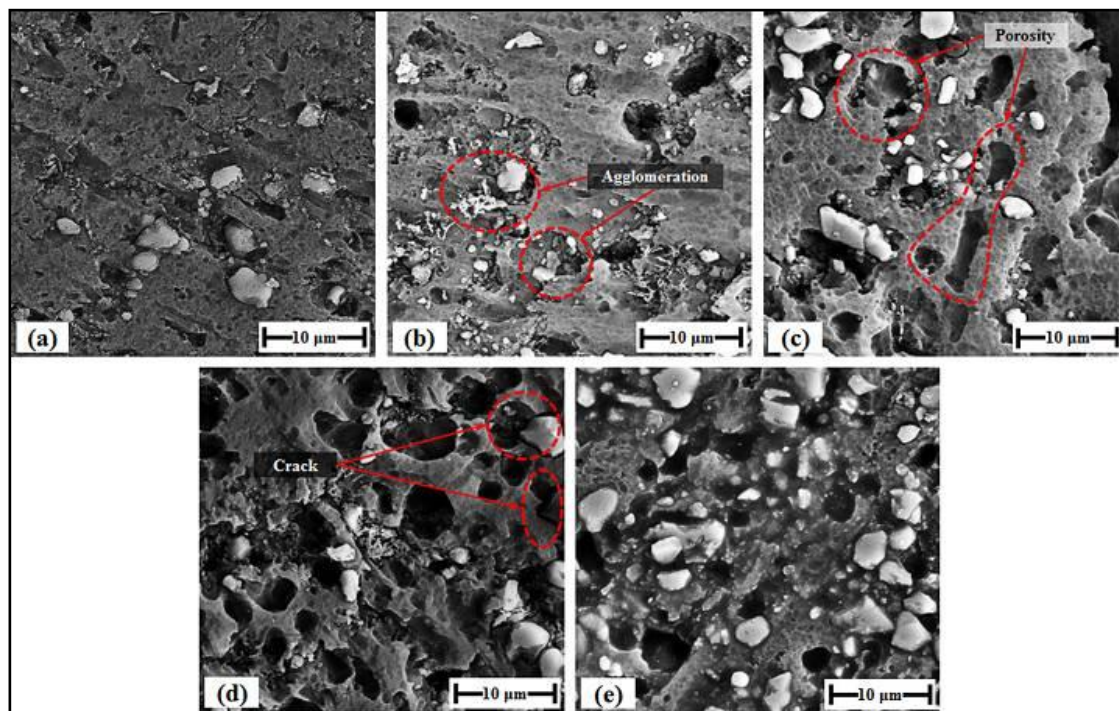


Fig. 2. Scanning electron micrographs of the  $\text{Al/TiB}_2$  nanocomposites with different amounts of reinforcement phase, sintered at  $650^\circ\text{C}$ : (a) 1.5, (b) 2.5, (c) 3.5, (d) 5, and (e) 10 Vol.%  $\text{TiB}_2$ .



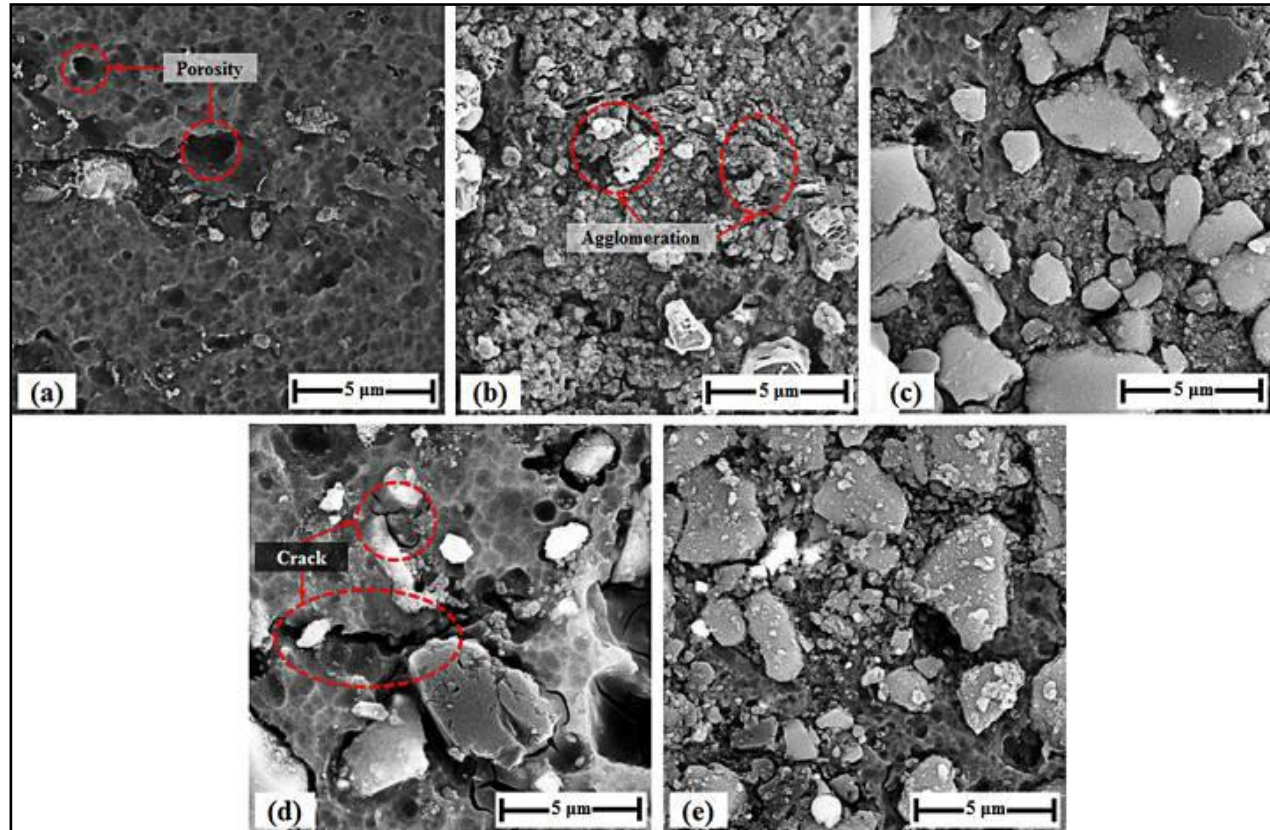


Fig. 3. Scanning electron micrographs of the Al/TiB<sub>2</sub> nanocomposites with different amounts of reinforcement phase, sintered at 680 °C: (a) 1.5, (b) 2.5, (c) 3.5, (d) 5, and (e) 10 Vol.% TiB<sub>2</sub>.

### Density Measurements

Bulk density, theoretical density, and the porosity factor for each TiB<sub>2</sub> volume percent and sintering temperature are presented in Figure 4a and 4b. Theoretical density of the composites is calculated according to the rule of mixtures (Eqn. 1) assuming the densities of aluminum and TiB<sub>2</sub> equal to 2.65 g/cm<sup>3</sup> and 4.52 g/cm<sup>3</sup>, respectively [19]:

$$\rho_{\text{composite}} = \rho_{\text{Al}} \cdot \chi_{\text{Al}} + \rho_{\text{TiB}_2} \cdot \chi_{\text{TiB}_2} \quad (1)$$

Where  $\rho_i$  stands for density, and  $\chi_i$  stands for volume fraction of each phase. Since the density of TiB<sub>2</sub> is more than that of aluminum, it is anticipated that with increasing the TiB<sub>2</sub> volume percent, density of composites increases. However, the incremental trend in theoretical density is not

exactly followed by the bulk density. In other words, the porosity factor ( $\epsilon$ ) of the composites also increases with increasing the TiB<sub>2</sub> volume fraction. Nevertheless, the slope of such trend is slightly decreased with increasing the sintering temperature from 650 to 680 °C demonstrating improvements in bulk densities of samples sintered at 680 °C. This occurs because of positive effect of elevated temperature on sinterability, diffusion rate, and bonding of matrix-reinforcement particles. Therefore, increasing the sintering temperature in a constant amount of TiB<sub>2</sub> improves the bulk density and lowers the porosity. However, in a constant sintering temperature, with increasing the volume percent of TiB<sub>2</sub>, bulk density increases due to higher density of TiB<sub>2</sub> phase and porosity increases as due to worsening wet ability and bindings.

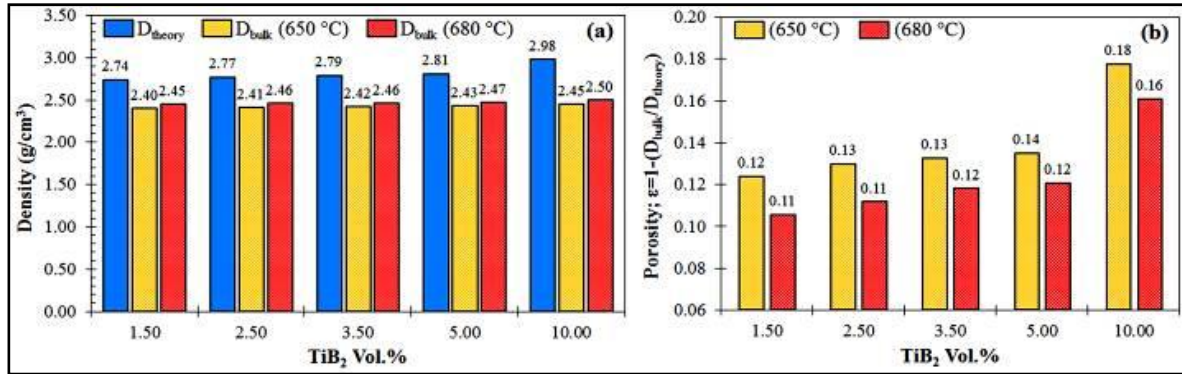


Fig. 4. (a) Theoretical and bulk density of Al/TiB<sub>2</sub> nanocomposites with different amounts of TiB<sub>2</sub> reinforcement phase sintered at different temperatures of 650 and 680 °C, and (b) porosity factors of the same samples.

Formation and development of undesirable porosity is an inherent problem of both the process of P/M and the ceramic-particles-reinforced MMCs. P/M-arisen porosity occurs due to lack of liquefaction of the matrix in sintering which results in partial fusion of metal phase, lower diffusion of atoms, and hence less seepage of matrix through the voids remained in inter-particle space especially in metal-ceramic interfaces [20]. Regardless of fabrication method, MMCs are basically susceptible to porosity arisen from combination of two different materials from perspective of bonding nature that causes some problems in terms of poor wettability (which is not as acute as other ceramics in comparison with TiB<sub>2</sub>), higher surface stress in the interfaces due to lattice mismatch (that gives rise to segregation and impede coalescence), and agglomeration of ceramic phase [21, 22]. This issue could partly be solved with increasing the time and/or temperature of sintering which, in turn, causes other problems as exaggerated coarsening of grains and more segregation.

## CONCLUSIONS

Fabrication of Al/TiB<sub>2</sub> nanocomposites with different contents of TiB<sub>2</sub> reinforcement nanoparticles is successfully accomplished via powder metallurgy in two different sintering temperatures of 650 and 680 °C. Density measurements of sintered samples revealed that the bulk density of samples increases with increasing the volume percent of ceramic phase mainly due to

higher density of TiB<sub>2</sub> phase. From another point of view, porosity of sintered samples is also increased due to the formation and proliferation of pores with increasing the TiB<sub>2</sub> content. In addition, samples sintered at higher temperature showed improvement in bulk density due to lower amounts of porosity. XRD phase analysis approved the uniform presence of TiB<sub>2</sub> in Al matrix with on signs of formation of other intermetallic phases. SEM micrographs of the samples illustrated several microstructural deteriorations as agglomeration, porosity, and crack that comply with the bulk density trend and grow with increasing the TiB<sub>2</sub> content and diminish in higher sintering temperature. At last, the optimum processing temperature to achieve better properties was 680 °C.

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