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Synthesis of Ceramic Particles (Al_2O_3 and TiB_2) in Aluminum Matrix

Metin ÖNAL[1], Mehmet GAVGALI[2]

ABSTRACT: Al_2O_3 and TiB_2 particles have been successfully synthesized in aluminum matrix with in situ reactions by hot pressing method. Process parameters; sintering temperatures 900-1000°C, pressure of cold pressing: 7 MPa-hot pressing: 1 MPa and holding times in furnace 5-10 minutes were selected. Composite microstructures and reinforcement particle numbers-sizes were investigated via SEM and EDS analysis. According to the analysis results; brittle Al_3Ti intermetallic transition phase frequently encountered in the literature could be eliminated.

Keywords: Composite, in situ, reinforcement particle, sintering

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ÖZET: Al_2O_3 ve TiB_2 partiküller, sıcak presleme yöntemi sayesinde in situ reaksiyonlarla alüminyum matris içinde başarıyla sentezlenmişlerdir. İşlem parametreleri; sinterleme sıcaklığı 900-1000°C, soğuk presleme basıncı: 7 MPa-sıcak presleme basıncı: 1 MPa ve fırında bekletme süresi 5-10 dakika olarak seçilmiştir. Kompozit mikroyapıları ve takviye partikül sayı-boyutları SEM ve EDS analizleri vasıtasıyla belirlenmiştir. Analiz sonuçlarına göre; literatürde sıkça rastlanan gevrek Al_3Ti intermetalik geçiş fazı elimine edilebilmiştir.

Anahtar kelimeler: In situ, kompozit, sinterleme, takviye elemanı

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[1] Yüzüncü Yıl Üniversitesi, Erciş Meslek Yüksekokulu, Elektrik ve Enerji Bölümü, Van, Türkiye
[2] Atatürk Üniversitesi, Mühendislik Fakültesi, Makine Mühendisliği Bölümü Konstrüksiyon ve İmalat Anabilim Dalı, Erzurum, Türkiye
Sorumlu yazar/Corresponding Author: Metin ÖNAL, monal@yyu.edu.tr

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INTRODUCTION

Al-Al alloys are preferred widely in metal matrix composites as matrix materials because of their low cost-melting point and density. At the same time; aluminum metal matrix composites are produced with mixture of whickers, fibers or particle shaped hard reinforced elements to improve existing properties of Al-Al alloys (Jun et al., 2004). SiC, TiC, Al_2O_3 etc. ceramic particles added into aluminum effect affirmatively tensile strength, elasticity module, wear strength and high temperature strength of composites (Kurt, 1992).

Composites with reinforced elements can obtain through two methods: particle synthesis technique in liquid matrix (in situ) and direct mixing particles into matrix (ex situ) (Sharifi et al., 2011). At in situ technique; preliminary powders are mixed homogeneously in a cast and then sintered. As the reinforcements are generated directly from chemical reaction within the matrix, the composites are of many excellent advantages, such as clean reinforcement-matrix interface, fine and thermodynamically stable reinforcements, good compatibility and high bond strength between reinforcements and the matrix, and low fabrication costs (Zhu et al., 2007). At ex situ technique; reinforcements are added into liquid matrix externally. High cost of starting materials and the resulting heterogeneous microstructure are of two important disadvantages of this method (Sharifi et al., 2011). Therefore, in situ synthesis technique is preferred as experimental process in this study. Particle reinforced MMCs are used in weight sensitive aviation, industrial sectors and transportation. Al_2O_3 reinforced MMCs are used in nozzles, cylinder liners and rotors whereas TiB_2 reinforced MMCs are used in cutters and armor materials. Al-TiO_2-B and Al-TiO_2-B_2O_3 triple powder systems can use for obtaining Al_2O_3 and TiB_2 ceramic particles in composite microstructure. But, both using pure boron (B) and titanium (Ti) powders would be more expensive than their oxides (Zhu et al., 2008) and the costs of production would be increase. In this study, oxide forms of powders are preferred to decrease the costs.

Obtaining a more intense material by compressing of starting powders forming the composites during production (at rising temperatures) process is called as 'hot pressing' (Elrakayby et al., 2015). A more dense and uniform microstructure is achieved due to this production technique actualized the simultaneously sintering and pressing (Chen et al., 2013). Reactant metallic powders are compacted to high cold pressures (10-15 MPa) after homogenously mixing. The powders are heated to happening temperatures of chemical reactions. Composites that applied set parameters are hot pressed at semi-solid/semi-liquid temperature of matrix element and then cooled. This way eliminates the need to a second operation such as extrusion.

MATERIAL and METHOD

Al powder (99% purity), TiO_2 powder (94% purity) and B_2O_3 powder (98.5 purity) with average sizes of 1-5, 0.3-1 and 250 μm were used as preliminary components for production of aluminum metal matrix composites respectively. Conventional hot pressing method was applied for sintering. Powders were mixed at low speed (30 rpm) in a kit during two hours. After mixing process, powders were dried in furnace at 70°C during an hour to resolve humidity. To determine reaction temperatures, DTA analysis was applied to 1 g powder mixture under argon gas (3 L min^{-1}) until 1100°C with 5.5°C min^{-1} heating, 6.5°C min^{-1} cooling rates. For increasing composite density, cold pressing was during two hours at 7 MPa pressure. The sintering process were applied at 900°C and 1000°C during 5-10 minutes in argon gas atmosphere. For minimize porosity, samples were hot pressed under 1 MPa pressure at semi-solid/liquid temperature of aluminum. The general view of the production system is in Figure 1. Then the composites were cooled down to room temperature on the outside of furnace. Microstructural characterization studies were carried out using scanning and electron dispersive spectroscopy microscope (SEM/EDS, Zeiss Evo LS-10).

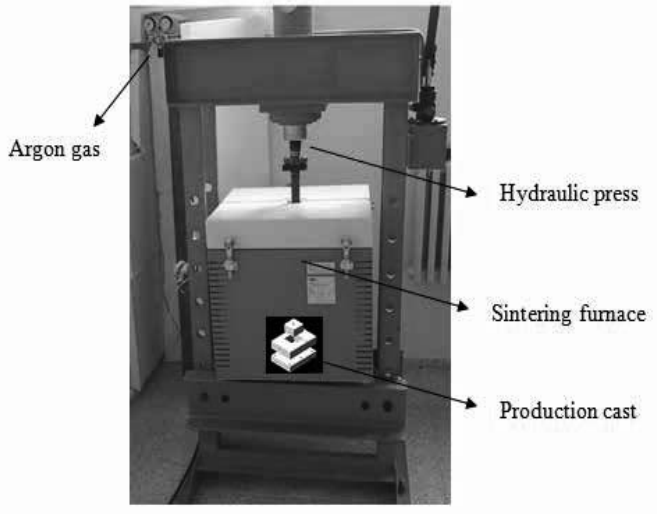

Figure 1. Composite production system

RESULTS and DISCUSSION

As a result of SEM images, the microstructure of the starting powders are shown in Figure 2.

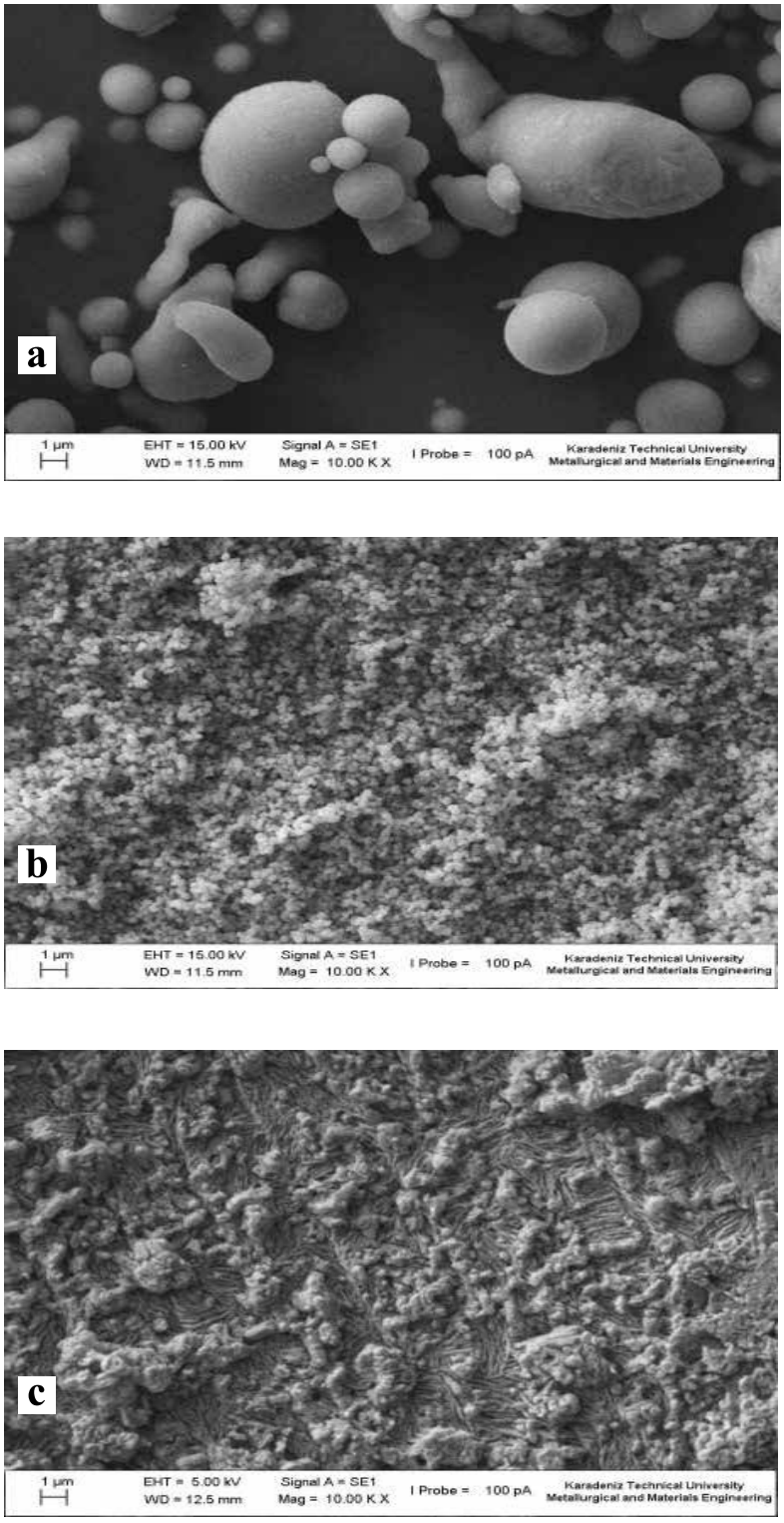

Figure 2. SEM images of powders a) Al b) TiO_2 c) B_2O_3

Curve of DTA analysis carried out in order to define the reaction temperature of the powder mixture are given in Figure 3.

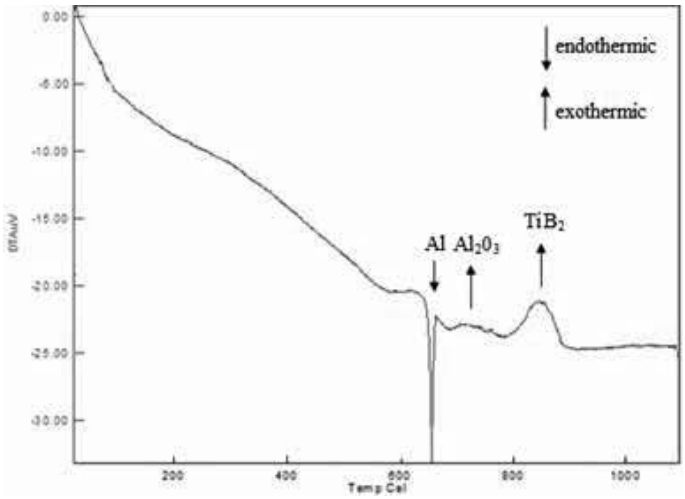

Figure 3. DTA analysis of powder mixture

In the graph, downward peak shows endothermic reaction (melting of Al), upward peaks show exothermic reactions (formation of Al_2O_3 and TiB_2). Aluminum melts at 670°C. Then Al_2O_3 and TiB_2 phases forms at around 720°C and 840°C, respectively.

A common SEM microstructure image of composites is in Figure 4. In general it has not been observed porosity in the microstructures of the composites. Applied cold pressing pressure of 7 MPa is consistent with the literature. Similar cold pressing process – under 100 bar (10 MPa) – has been used by Dikici et al. when producing TiC particles in liquid aluminum before sintering (Dikici et al., 2010). Pressure has been continuously supplemented. This has led to an increase in density of the composites.

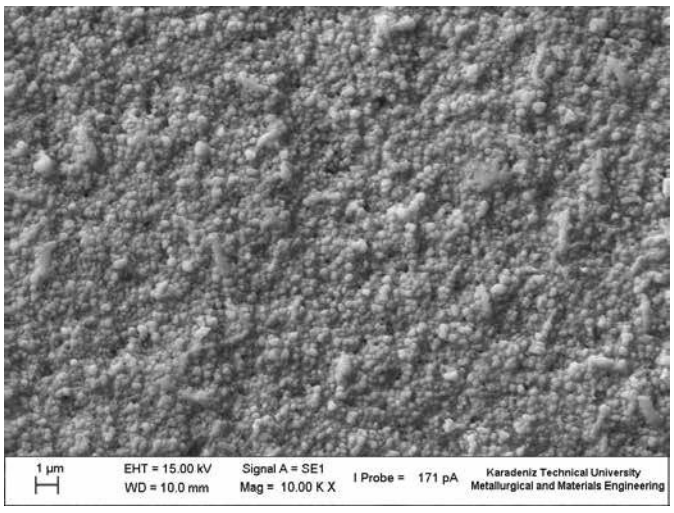

Figure 4. Common SEM microstructure image of composites

Figure 5 shows the SEM images of composites at 1000 magnification. EDS analysis that define mass and mole ratios of elements forming particles are observed as a result of elemental analysis in points shown with green colour. Analysis prove that the microstructures of composites consist of Al_2O_3 and TiB_2 particles.

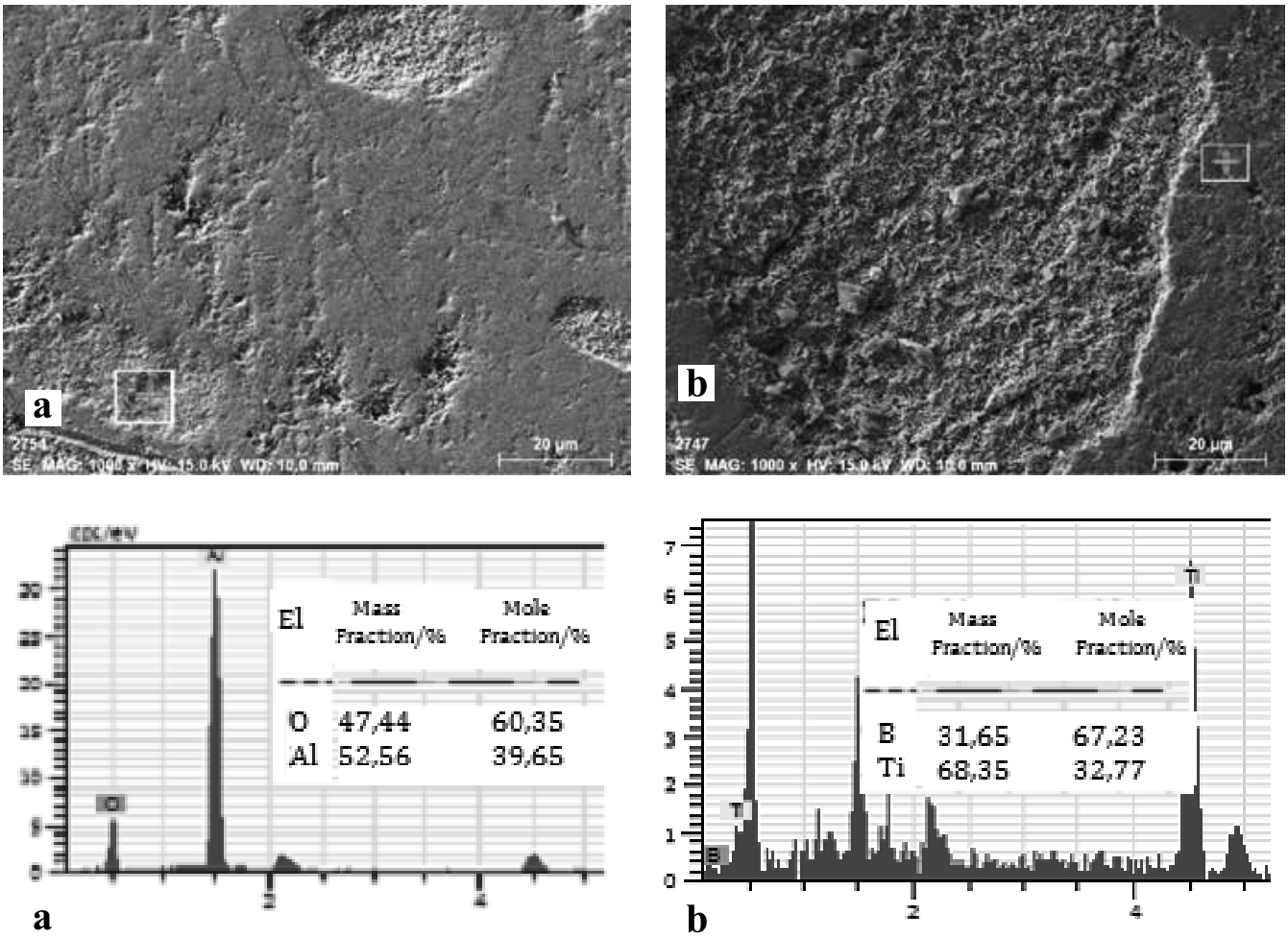

Figure 5. SEM images and EDS analysis of particles a) Al_2O_3 b) TiB_2

Sintering process was firstly carried out at 900°C and then at 1000°C to sight the effect of sintering temperature above microstructures of composites. 900°C temperature is one of the lowest temperature necessary for the formation of the desired phases can be seen in the above DTA analysis (Figure 3). 5 and 10 minute holding times are sufficient to observe changes in the microstructure. By 7.6°C min^{-1} for 900°C and by 5.8°C min^{-1} heating steps for 1000°C were reached to defined maximum temperatures according as furnace's heating power. Kayıkcı et al. also studied at steps close to these heating rates – 8°C min^{-1} - for producing AlB_2/Al composites (Kayıkcı et al., 2009). The samples were down to room temperature on the outside of furnace by average 6.5°C min^{-1} cooling rate after holding 5 and 10 minutes in furnace and hot pressed.

In literature, in some of the studies about Al and Ti are mentioned an intermetallic-transition phase named as Al_3Ti. Balci et al. observed a small amount of the Al_3Ti intermetallic compound in microstructure of all mechanically alloyed both micron and submicron-scale TiB_2 particle reinforced Al matrix samples after sintering at 650°C (Balci et al., 2014). Lu et al. found this phase in the composites sintered at different temperatures from 600 to 750°C for 2 h in carbolite furnace (Lu et al., 2001). In the EDS analysis and SEM images of the composites said phases are not observed. This may be due to several different reasons. One; selected production temperatures are quite high. Another; holding times made at the selected temperatures are long enough. Because intermetallic-transition phases transform to more stable phases by dissolving due to applied high temperatures and sintering times. Chu and Premkumar, in their study related to Al-Ti-C, say that reason of high production temperatures selection is to ensure dissolution of Al_3Ti particles (Chu and Premkumar,

1993). Another reason; depending on applied heating and cooling rates, required temperature range for the formation of these phases can be passed quickly. Because in the DTA analysis of mixed powders there is not any peak except Al, Al_2O_3 and TiB_2.

Effect of sintering temperature: SEM images of sintered composites at 900°C and 1000°C are shown in Figure 6.

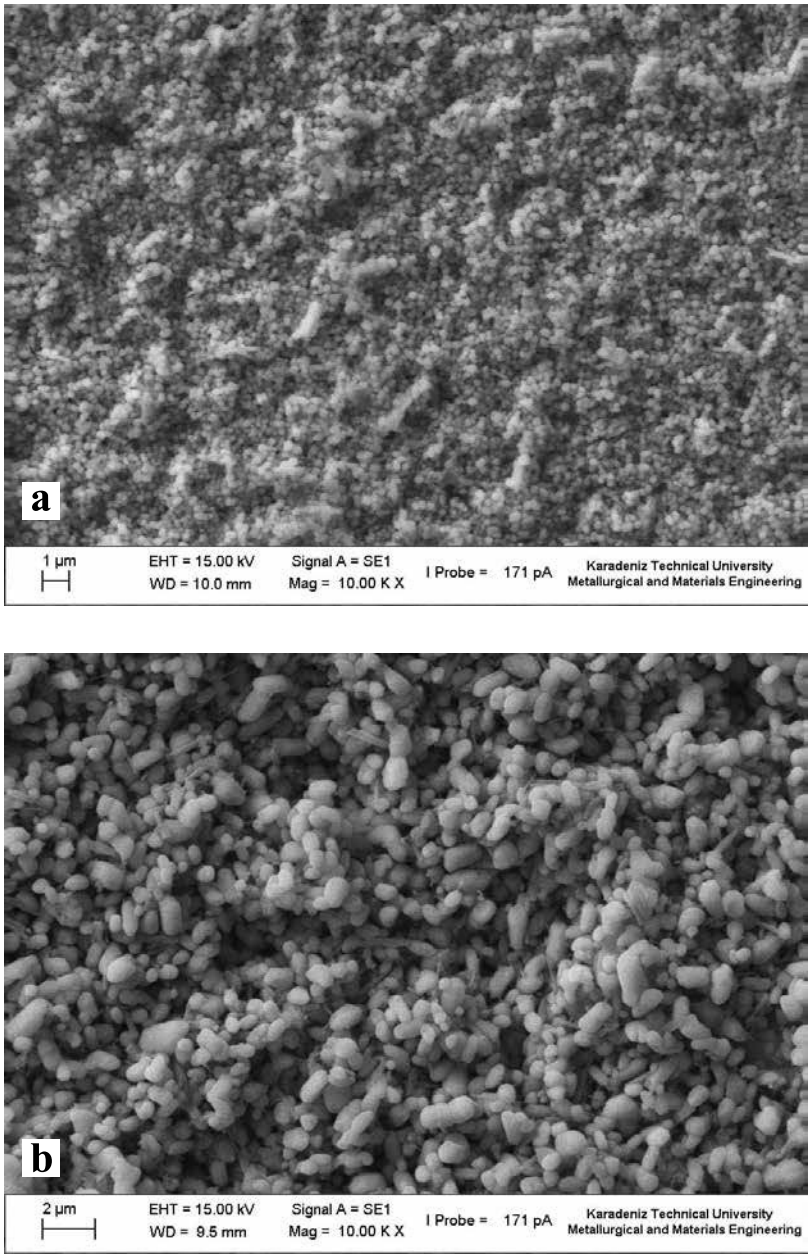

Figure 6. SEM images of grain sizes at different sintering temperatures: a) 900°C b) 1000°C

It is shown that composite sintered at 900°C has a fine grain microstructure in Figure 6a. Average particle sizes are less than 1 µm. Figure 6b exhibits the microstructure with coarse grain. According as increasing of temperature, particles changed shape by prolonged. German M. R. says that particles may coarse with increasing temperature at studies with about sintering in his Sintering Theory and Practice Book (German, 2013).

Effect of sintering (holding) time: In same temperature conditions (1000°C), SEM images of composites are shown in Figure 7.

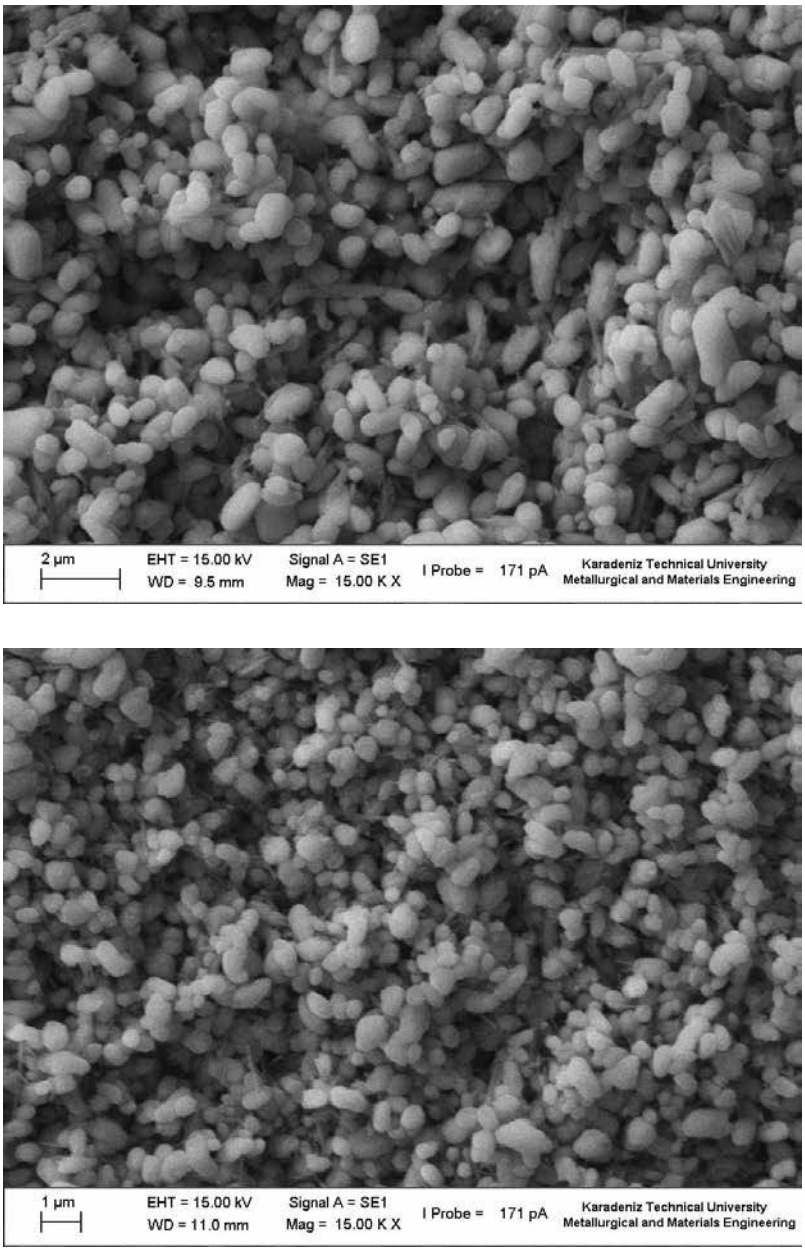

Figure 7. SEM images of composites sintered at different times a) 5 min. b) 10 min.

With increasing of holding time in furnace of samples (5-10 min.), it seems that particles consisting of more in Figure 7b. Dikici et al. point out a quick increasing of numbers and formation speed of α-Al_2O_3 particles with increasing sintering time in study about Al_2O_3 particle synthesis in pure aluminum (Dikici and Gavgali, 2013). With elevated sintering time, hardness and wear resistance of the composites will increase because of synthesized more reinforcing particles (Onal and Gavgali, 2015). Kök emphasizes the fact that the increase in the number and size of the Al_2O_3 particles obtained in 2005 via vortex method in 2024 aluminum alloy matrix increases the wear resistance of composites (Kok, 2006).

CONCLUSIONS

In aluminum matrix, as a result of working synthesis in situ reinforcement particles by hot pressing method, the following conclusions were reached:

1. Al_2O_3 and TiB_2 ceramic particles were able to be synthesized in the way of success with hot pressing method, using oxides of Ti and B powders cheaply. Use pure forms of starting components will raise costs of the production.
2. Applied cold pressing pressure of 7 MPa at production of composite is a suitable pressure and avoids porosity. This pressure value reduces the cast wear according to applied 10-15 MPa pressures in previous studies. Cold pressure must be continuously supplemented.
3. Sintering temperature is an effective parameter in increasing of particle sizes. The number of particles decreases with increasing temperature and are becoming larger.
4. With the increasing holding time in the furnace, increase the number of the formed particles was observed.
5. In this study; selected sintering temperatures and applied heating/cooling rates for composite production are quite high and sintering times are long enough. As a result of these parameters, brittle Al_3Ti phase could be eliminated. In order to optimize the production costs, effects of these parameters can be examined individually or in combinations in future studies.

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