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Microstructure, hardness and thermal properties of Al4.5Cu/TiO_2 composites produced by mechanical alloying

Mekanik alaşımlama ile üretilen Al4.5Cu/TiO_2 kompozitlerinin mikroyapı, sertlik ve termal özellikleri

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Microstructure, Hardness and Thermal Properties of Al4.5Cu/TiO_2 Composites Produced by Mechanical Alloying

Highlights

- *X Al4.5Cu/TiO_2 composites were fabricated by using a powder metallurgy process*
- *Microstructural evolutions were investigated depending on the milling time*
- *Thermal properties were investigated via DSC*
- *The maximum microhardness value was found to be 173±10 HV*

Graphical Abstract

Microstructural and thermal properties of the mechanically alloyed Al4.5Cu/TiO_2 powders were investigated by a combination of XRD, DTA, and SEM-EDX. Also, the microhardness of pressed and sintered samples was investigated.

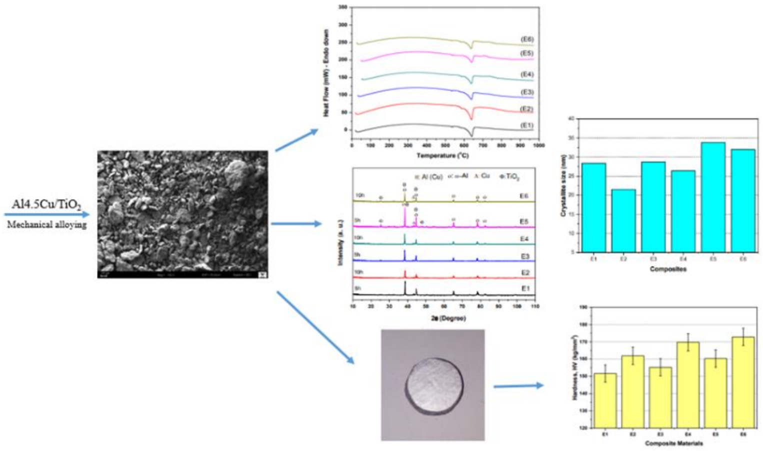

Figure. Experimental results in Al4.5Cu/TiO_2 composites produced by mechanical alloying

Aim

The aim of this study is to produce Al4.5Cu/TiO_2 metal matrix composite materials with superior properties and to examine their thermal, microstructural and hardness properties.

Design & Methodology

Mechanical alloying technique was used to produce Al4.5Cu/TiO_2 composites. Microstructural evolutions were examined by XRD and SEM. Thermal properties are investigated via DTA. Hardness measurements were taken after pressing, sintering and etching processes.

Originality

To the best of our knowledge, Al4.5Cu/TiO_2 composites with different wt% TiO_2 reinforcement were produced for the first time using mechanical alloying at 5-10h milling times, and their characterization was carried out.

Findings

α-Al, Al (Cu), Cu, and TiO_2 phases are observed in all Al4.5Cu/TiO_2 composite powders. Microstructural investigations showed that there were a more homogeneous structure and shrinkage in grain size due to the increased milling time. An endothermic peak of around 650 ^oC which indicates the melting of Al was found during continuous heating. Also, the maximum microhardness value of 173±10 HV was obtained for Al4.5Cu with 20 wt% TiO_2 composite after milled for 10h.

Conclusion

The Al4.5Cu/TiO_2 metal matrix composite is fabricated from its elemental powders by mechanical alloying technique. Thermal, mechanical and microstructural analyses of the produced composites were carried out.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Microstructure, Hardness and Thermal Properties of Al4.5Cu/TiO_2 Composites Produced by Mechanical Alloying

Araştırma Makalesi / Research Article

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ABSTRACT

Al4.5Cu/TiO_2 composites were fabricated from their elemental powders by the mechanical alloying method. Microstructural and thermal properties of the composites were investigated by a combination of differential thermal analysis (DTA), scanning electron microscopy with energy dispersive X-ray detection (SEM-EDX), and X-ray diffraction (XRD). Microstructural evolutions, phase transformations, and crystallite size changes were investigated depending on the milling time. XRD and SEM results showed that there were a more homogeneous structure and shrinkage in grain size due to the increased milling time. The DTA results showed an endothermic peak of around 650 ^oC which indicates the melting temperature of Al. Besides, the mechanical properties of the pressed and sintered composites were investigated by Vickers micro-hardness testing. The results showed that microhardness values significantly increased as milling time increased from 5h to 10h. The maximum microhardness value of 173±10 HV was obtained for Al4.5Cu with 20 wt% TiO_2 composite after milling for 10h.

Keywords: Metal matrix composites, thermal properties, microstructure, mechanical alloying.

Mekanik Alaşımlama ile Üretilen Al4.5Cu/TiO_2 Kompozitlerinin Mikroyapı, Sertlik ve Termal Özellikleri

ÖZ

Al4.5Cu/TiO_2 kompozitleri, mekanik alaşımlama yöntemiyle elemental tozlarından üretilmiştir. Kompozitlerin mikroyapısal ve termal özellikleri, diferansiyel termal analiz (DTA), enerji dağılımlı X-ışını algılamalı taramalı elektron mikroskobu (SEM-EDX) ve X-ışını kırınımının (XRD) bir kombinasyonu ile araştırıldı. Öğütme süresine bağlı olarak mikroyapısal evrimler, faz dönüşümleri ve kristalit boyutu değişiklikleri incelenmiştir. XRD ve SEM sonuçları, öğütme süresinin artması nedeniyle daha homojen bir yapı ve tane boyutunda küçülme olduğunu göstermiştir. DTA sonuçları, Al'nin erime sıcaklığını gösteren yaklaşık 650 ^oC'de bir endotermik pik gösterdi. Ayrıca, preslenmiş ve sinterlenmiş kompozitlerin mekanik özellikleri Vickers mikro sertlik testi ile incelenmiştir. Sonuçlar, öğütme süresinin 5 saatten 10 saate çıktığında mikrosertlik değerlerinin önemli ölçüde arttığını göstermiştir. 10 saat öğütme sonrasında maksimum mikrosertlik değeri, ağırlıkça %20 TiO_2'li Al4.5Cu kompozit için 173±10 HV elde edilmiştir.

Anahtar Kelimeler: Metal matrisli kompozitler, termal özellikler, mikroyapı, mekanik alaşımlama.

1. INTRODUCTION

Today, with the development of technology, it has become compulsory to offer higher quality products to the market with lower costs in an increasingly competitive environment. Therefore, the properties such as low density rigid, high corrosion resistance and high strength of new generation materials to be used in industries should be developed. In recent years, ceramic reinforced metal matrix composites (MMCs) have attracted the attention of researchers as an alternative to conventional alloys [1-3]. Aluminium matrix composites (AMCs) reinforced with ceramic particles such as SiC [4], TiC [5, 6], B_4C [7], TiB_2 [8, 9], and Al_2O_3 [10] are widely used as lightweight building materials in the aviation and automotive industry. Many studies [11-13] have been conducted on the development of nanocomposites using nano-sized reinforcing particles.

Different production techniques such as, stir casting, compo-casting, and squeeze casting, pressure infiltration, spray co-deposition and powder metallurgy methods have been used to produce MMCs [14]. Among these important techniques, the powder metallurgy has attracted considerable attention from researchers due to fine-grained structures and the chemical homogeneity of powders [15, 16]. Mechanical alloying (MA) is an

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interesting powder metallurgy technique as it produces powder material with superior homogeneity. This technique with powder mixing is effective in dispersing reinforcing particles and supports fine particle size in the matrix; thus, it increases the microhardness [17-19]. So, the mechanical alloying route in this study has been selected among various available methods mentioned above. In addition, the sintering technique, which affects the strength properties such as hardness in materials, is an important method used to obtain superior properties in materials produced by mechanical alloying. Therefore, in this study, conventional sintering technique was used from various sintering methods such as conventional sintering [20, 21], microwave sintering [22, 23] and spark plasma sintering [24, 25].

In the literature, it has been reported that AMCs such as Al (7010) -SiC [26], Al (2124) -SiC [27], Al (6061) - TiC/Al_2O_3 [28] are produced by mechanical alloying technique. Besides, a metal matrix composite was produced with Al4Cu alloy containing ZrC particles [14]. Also, as far as we can see, in the literature, there are very few studies on MMCs with TiO_2 reinforcement. While the carbides such as SiC, TiC, and B_4C are widely used, little attempt has been made to produce TiO_2 reinforced AMCs. In MMC studies, TiO_2 can be an excellent option because of its good chemical stability, high melting point, low density, high wear resistance and good hardness. In a similar study [29], Al5Cu/TiO_2 composite was developed by solidification method by adding 0.5-3.5wt% of TiO_2 powders to the molten Al-5Cu alloy, and dry sliding wear behaviours and surface properties of the produced bulk composites were investigated. This study aims to produce Al-Cu/TiO_2 metal matrix composite materials with superior properties and to examine the thermal and microstructural properties of the produced materials. Here, TiO_2 powder particles were selected as reinforcements to fuse with Al-4.5wt% Cu matrix alloy. After reviewing the literature studies on this subject, the most suitable production technique was thought to be the mechanical alloying technique, and thus Al4.5Cu/TiO_2 metal matrix composite was produced as a powder by mechanical alloying technique. Thermal and microstructural characterization of the powder materials produced was made depending on the reinforcement amount and milling time. For this purpose, X-ray diffraction (XRD), differential thermal analyser (DTA), scanning electron microscope with energy dispersive X-ray detection (SEM/EDX) and optical microscope (OM) studies were performed.

2. EXPERIMENTAL PROCEDURES and PROCESSING

In this study, Al4.5Cu/TiO_2 composites were fabricated by mechanical alloying technique by milling of 5-10 h by mixing TiO_2 reinforcement to Al4.5Cu alloy matrix. Some properties of purchased Aluminium (Al), Copper (Cu), and TiO_2 powders are given in Table 1. Powder materials are weighed in sensitive scales and argon gas atmosphere in desired proportions and placed in 250 ml jar with 10 mm diameter balls, both produced of stainless steel. Then, they were subjected to the mechanical milling process with the XQM-2 high energy planetary ball mill. In order to avoid contamination in the composite, the powders were milled without using any process control agents during the milling process. In milling processes, the ball-powder ratio was applied as 10:1, and the rotation speed was 350 rpm. The device was operated for 20 minutes and kept for 20 minutes to prevent the samples from being exposed to excessive heat. Production parameters are given in Table 2.

The produced powders were examined under monochromatic CuK_α radiation (λ = 0.154056 nm) by setting the XRD device (Philips X`Pert PRO) to 40kV and 30mA. For all samples, XRD measurements were taken at 0.2º/s from 10º to 110º at room temperature. Also, microstructural analyses were carried out by using a Zeiss EVO LS10 scanning electron microscope equipped with an energy-dispersive X-ray spectrometer (SEM-EDX). Moreover, thermal properties of produced powders were analysed by using an SII 6300 EXSTAR differential thermal analysis (DTA), where the samples were heated at 30 ºC min^{-1} in a pure nitrogen atmosphere.

Table 1. Properties of powders used in the experimental study

Materials	Purity (%)	Particle size (µm)	Melting emperature (ºC)	Density (g/cm^3)	Atomic mass (g/mol)
Aluminum (Al)	**99.8**	**≤ 48 µm**	**660.4**	**2.70**	**26.981**
Copper (Cu)	**99.9**	**≤ 48 µm**	**1085**	**8.93**	**63.546**
Titanium dioxide (TiO_2)	**99.5**	**≤ 70 µm**	**1843**	**4.23**	**79.866**

Table 2. Composite materials and production parameters

	Material code					
	E1	**E2**	**E3**	**E4**	**E5**	**E6**
Milling time (h)	**5**	**10**	**5**	**10**	**5**	**10**
Reinforcement TiO_2 (wt%)	**10**	**10**	**15**	**15**	**20**	**20**

Experimental tests were performed twice to ensure the repeatability of the analyses.

Some of the composite powders produced by milling for 5 and 10h were pressed in a unilateral hydraulic press at 250 MPa. Pressed samples were sintered under argon atmosphere at 600 °C for 1h, subjected to polishing processes for microstructure images, etched with 20% HF-30% HNO_3- 50% pure water solution, and photographs of the surfaces were taken with an optical microscope. Also, Vickers hardness measurements of these samples were carried out under 100g load and 10s dwell time with a Schimatzu microhardness device. The microhardness value for each sample was determined by averaging the ten measurement results taken from different regions on the sample surface.

3. RESULTS AND DISCUSSION

3.1 Microstructural evaluations

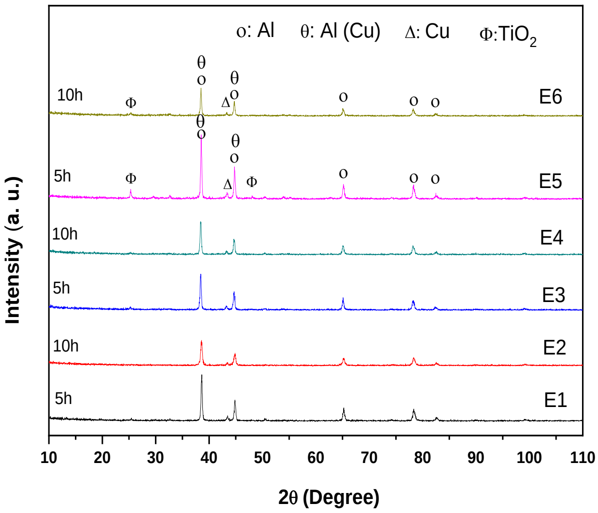

Figure 1. XRD patterns of mechanically milled $Al4.5Cu/TiO_2$ composites

In order to investigate the microstructural evaluations of the as milled $Al4.5Cu/TiO_2$ composite powders produced by mechanical alloying, the samples were firstly identified by X-ray diffraction (XRD). The XRD results of mechanically milled $Al4.5Cu/TiO_2$ composites are given in Figure 1. As seen in Figure 1, α-Al, Al (Cu), Cu, and TiO_2 phases are observed in all $Al4.5Cu/TiO_2$ composite powders. The absence of any intermetallic phases in the produced composite structure indicates that alloying has not occurred. Kaftelen et al. [14] have reported similar results for the Al–4wt.% Cu alloy matrix composite with ZrC reinforcement. As seen in Figure 1, with the milling time increasing from 5h to 10h, the diffraction peaks of Al and Al(Cu) become broadened with decreasing intensity. This change seems to result from the refinement of the crystal grain size and an increase in lattice strain [30]. This is mainly due to heavy deformation, repeated fracture and cold-welding. Figure 2 shows the XRD patterns of sintered $Al4.5Cu/TiO_2$ composites. As seen in Figure 2, if the composite sample is sintered, new intermetallic phases (i.e. Al_2Cu, AlTi) are observed with a weak peak intensity. This indicated that diffusions and new reactions occurred during sintering with the effect of high temperature. As reported by Shin et al. [31], the AlTi phase was formed as a result of a series of reactions of the TiO_2 and Al phases. During the continuous heating, occurred reactions are that $2Ti_2O \leftrightarrow \alpha\text{-}Ti + Ti_3O_2$ [32], and L (Liquid Al) + α-Ti ↔ AlTi [33]. It is also thought that the Al_2Cu phase is formed in the Al4.5Cu alloy after sintered. Moreover, as seen in Figure 2, the peak related Al in E6 sample is higher than those of other phases. This indicates a free interface between the Al-Cu matrix and TiO_2 particles, and that TiO_2 particles form aggregates within the Al4.5Cu alloy, and thus the grain boundary lengths increase. Therefore, found results are in good agreement with those reported in the literature [34-38].

The crystallite sizes (*D*) of $Al4.5Cu/TiO_2$ composite powder were calculated from the broadening of XRD peaks (in Figure 1) using a well-known Debye Scherrer equation [39, 40]. The equation is given as follow,

$$D = \frac{0.9\lambda}{\beta Cos\theta} \quad (1)$$

where λ is the CuK_α radiation wavelength (0.154 nm), β is the full width at half maximum intensity (FWHM), and θ is the Bragg diffraction angle.

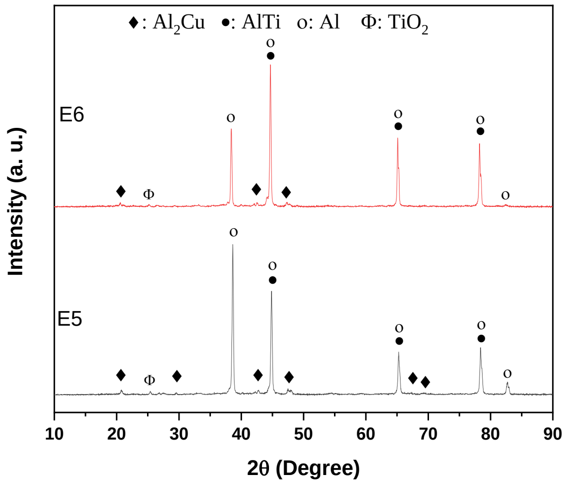

Figure 2. XRD patterns of sintered $Al4.5Cu/TiO_2$ composites

Figure 3 exhibits the changing of crystallite size of the milled $Al4.5Cu/TiO_2$ composite powders. As can be seen in Figure 3, with the increasing the TiO_2 reinforcement, the crystallite size increases, but when the TiO_2 percentage is kept constant and the milling time is increased, the crystallite size decreases. Among the composite samples, the E2 composite has the smallest crystallite size with 21.5±2 nm, while the E5 composite has the largest crystallite size with 34±2 nm. As a result, one can produce smaller crystallite-size composite materials at lower reinforcement rates and higher milling time. Similar results were also reported for the Ti and TiO_2 powders by Avar et al. [41], and others [42]

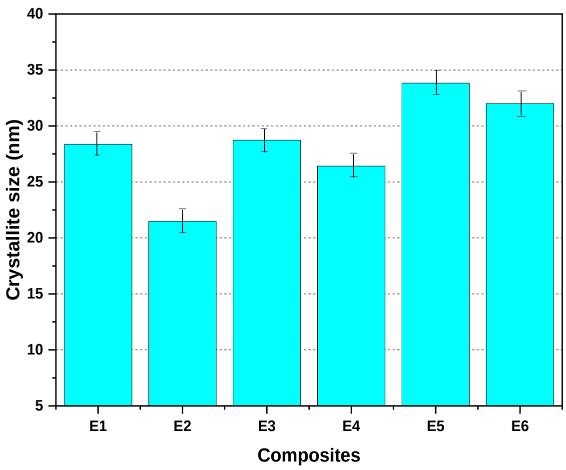

Figure 3. The crystallite sizes of the mechanically milled $Al4.5Cu/TiO_2$ composite powders.

Therefore, from these results, it could be said that, under the 5-10h milling conditions, no indications for a formation of intermetallic phase (i.e. Al_2Cu) were detected from the XRD patterns, and increased milling time decreased the crystallite size. This observation is in agreement with that recently reported by Kursun et al. [43]. It should be noted that the intermetallic Al_2Cu phase is formed only a long milling time and after sintered.

With the aim of examining the structural change of $Al4.5Cu/TiO_2$ composite powders during mechanical alloying in more detail, such as particle size and particle distribution, the mechanically alloyed powders were also subjected to morphological examination with scanning electron microscopy (SEM). Various SEM images of mechanically alloyed powders are given in Figure 4. When Figure 4 is examined, it can be seen that $Al4.5Cu/TiO_2$ composite particles have different shapes and sizes as a result of mechanical milling [30]. The evaluations occurring in the microstructures during mechanical alloying depend on the milling time and the amount of reinforcement (wt%). In the images, which can be seen in Figure 5 taken by the high magnification of the SEM, it is seen that the particles remain between the colliding balls during the milling of the powders and they form an approximately spherical structure by repeated cold-welding and subsequent fracturing. The cold-welding takes place as a result of the powder crushed between high energy balls to be more stable. These powders can sometimes give a rather large single powder ball appearance. In addition, as the rate of reinforcement increased, it was clearly seen that there were agglomerations due to cold-welding between the particles

It is also possible to make a comparison about particle sizes in terms of changing milling time in SEM studies. Avar et al. [30] determined the average particle size distribution of the milled powder samples from the SEM images depending on the milling time. The images (Figure 5) clearly show that with increasing milling time, the particle size shrinks. However, in this study, with the milling time increasing from 5h to 10h, the particles reached a more homogeneous and softer-edged spherical structure. Also, larger size particles formed as a result of agglomerations are encountered in the structure. Failure to use a process control agent during the milling process may cause strong agglomerations between the particles adhering to the walls and the balls [44]. As the milling time increases, the morphology of the particles has changed and the reinforcing phase and the main phase are mixed and a more homogeneous structure is obtained. Besides, it has been observed that bond-free bonding occurs as a result of the fusion between the matrix phase and the reinforcement phase. These results were found to agree with previous studies [45-48]. As a result, clustering occurs as the rate of reinforcement increases, and the particle size decreases as the milling time increases.

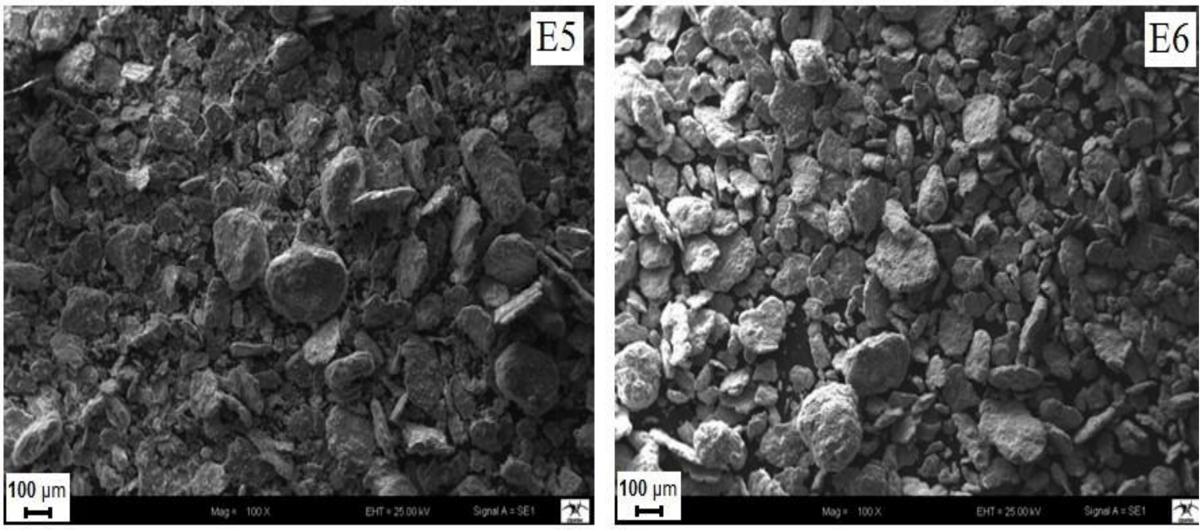

Figure 4. The SEM images of the mechanically milled E5 and E6 composite powders.

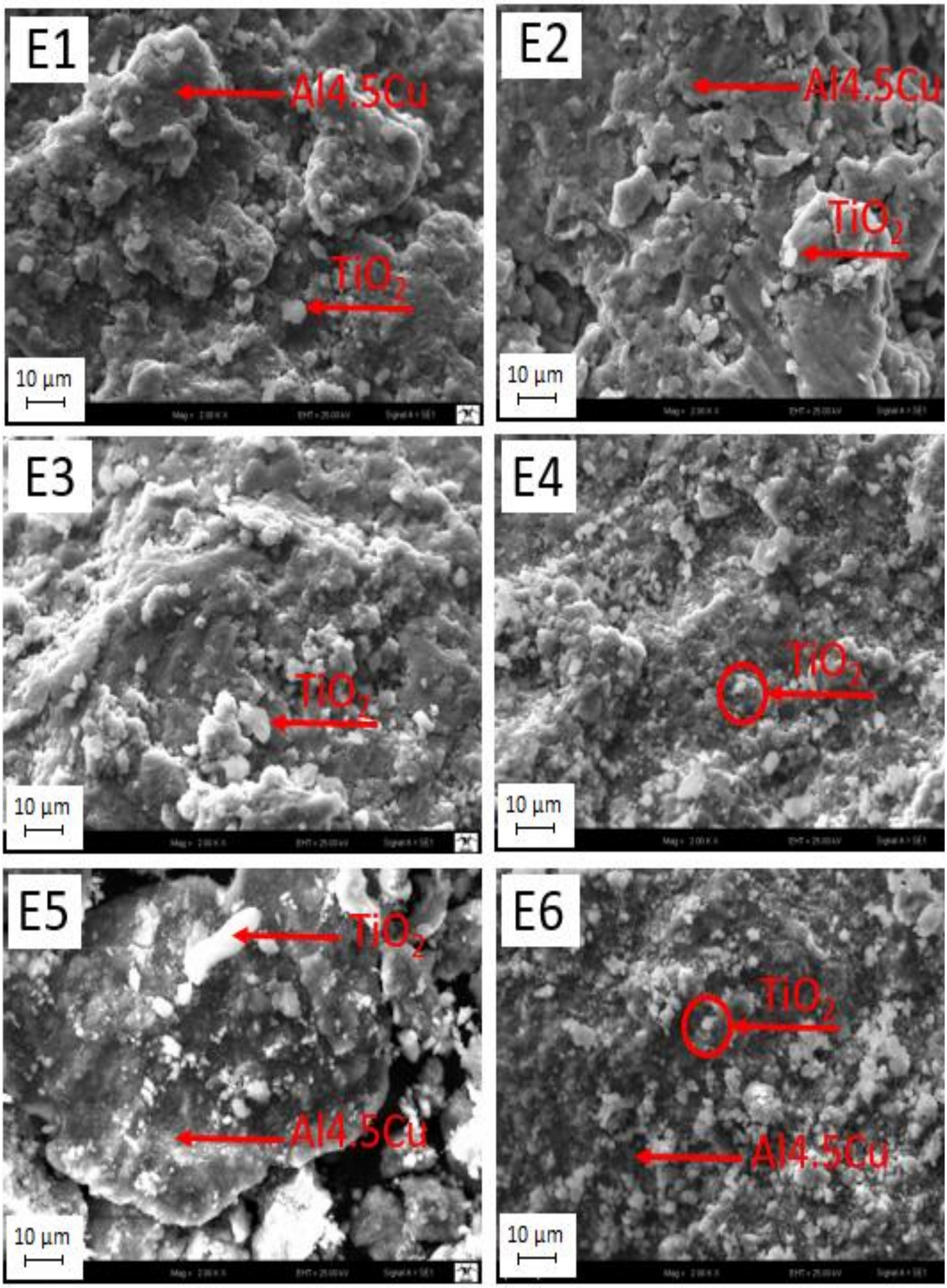

Figure 5. The SEM images of the mechanically milled Al4.5Cu/TiO_2 composite powders.

Moreover, EDX analysis of the composite powders produced was performed. As can be seen in Figure 6, in the composite produced, no element different from its components was seen. The SEM and EDX results obtained were in agreement with the XRD results.

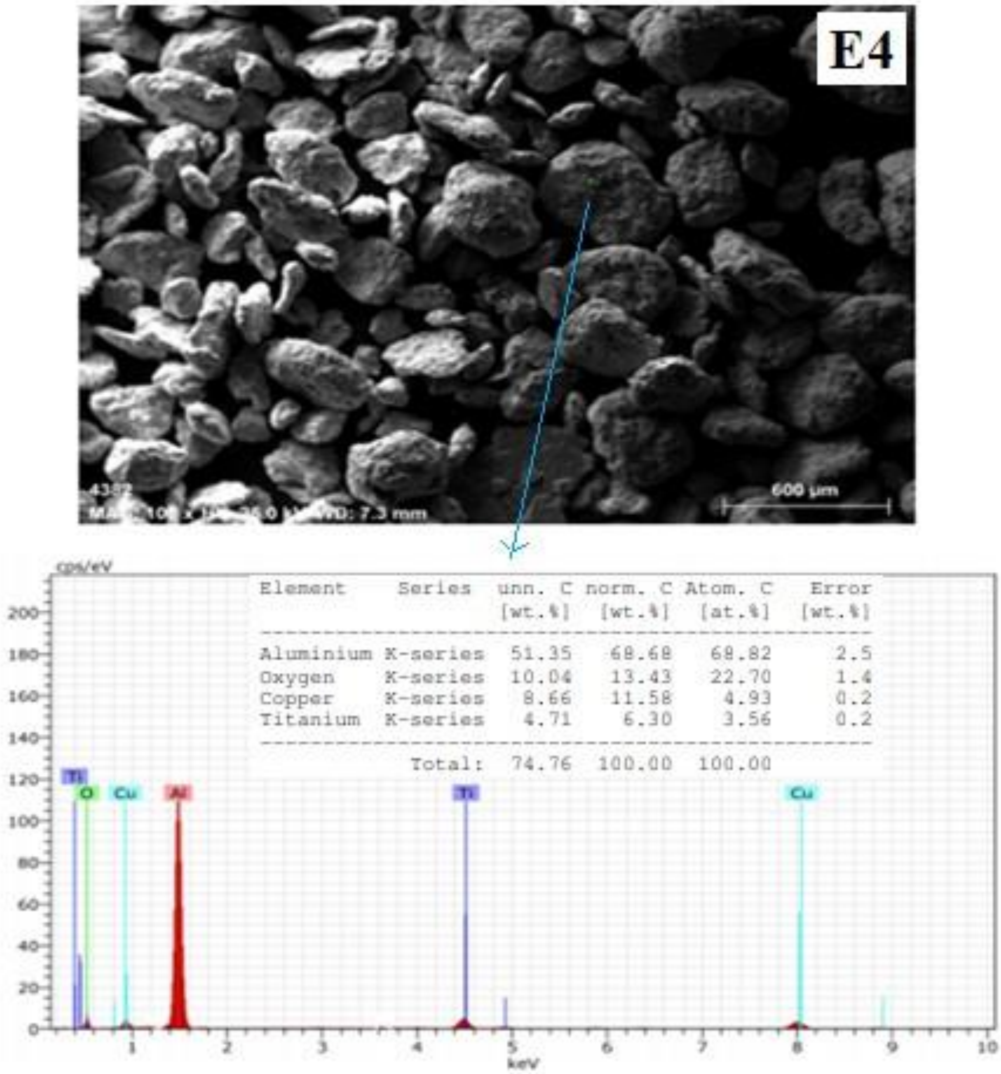

Figure 6. The EDX results of mechanically milled E4 composite powder.

3.2. Thermal properties

Thermal properties of $Al4.5Cu/TiO_2$ composite powders were investigated by SII 6300 EXSTAR Differential Thermal Analysis (DTA). For DTA measurements, composite powders were weighed to approximately 25-45 mg and placed in a platinum container. Measurements were made by continuous heating from room temperature to 970 ºC under a nitrogen atmosphere at a heating rate of 20 ºC/min. The results obtained are given in Figure 7. As seen in Figure 7, mechanically milled $Al4.5Cu/TiO_2$ composite powders exhibit similar thermal behaviour which one endothermic peaks seen in the range of 500-600 ºC indicate that the Al 4.5Cu solid solution phase has started to melt at the grain boundaries. The endothermic peaks seen around 640 ºC show that the α-Al phases consisting of TiO_2 started to melt. Also, exothermic peaks observed in the 700-750 ºC temperature range indicate that the AlTi phase was formed in the composites.

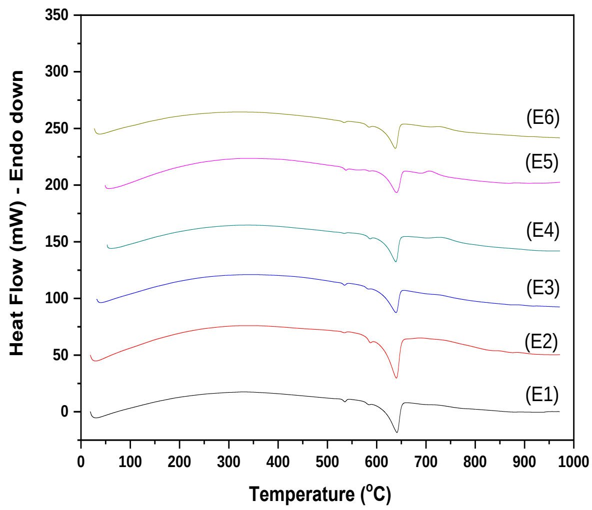

Figure 7. DTA thermograms of mechanically milled $Al4.5Cu/TiO_2$ composite powders

3.3. Microhardness

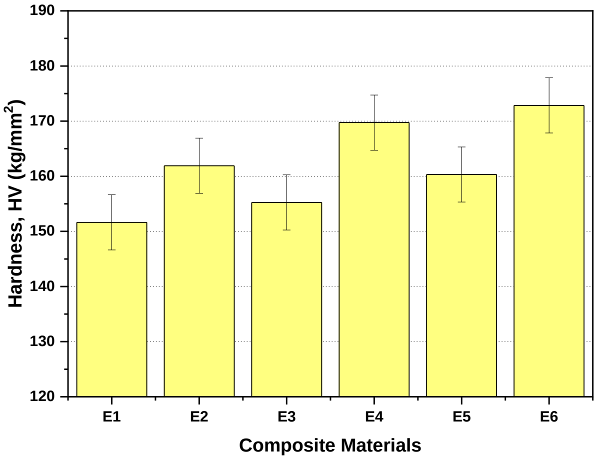

Figure 8. Microhardness values of sintered Al4.5Cu/TiO_2 composites

Mechanical properties of the sintered Al4.5Cu/TiO_2 composites samples were measured by a Vickers indenter. Average values of Vickers microhardness measured from different regions of the surfaces of the sintered samples are given in Figure 8. Considering the change in the amount of reinforcement in the samples, the microhardness values of the composite samples increased as the amount of reinforcement increased from 10% to 20%. The reason for this situation is thought to be due to the high hardness of TiO_2 reinforcement. It also acts as a barrier to dislocations because the TiO_2 particles are harder than the base matrix Al4.5Cu. Here, not only TiO_2 acts as a barrier of dislocations but Al_2Cu precipitates also act as barrier for dislocation movement. So, hardness increases due to both Al_2Cu precipitates and also TiO_2 reinforcement. In the literature [29, 49-51], it was reported that the hardness increased with increasing the TiO_2 reinforcement in the Al-based metal matrix composites. On the other hand, microhardness values also increased due to the work hardening of the powders during the fracturing process as milling time increased from 5h to 10h. On the other hand, the hardness values have also increased as a result of the particles coming closer to each other or fused with the effect of sintering temperature at the grain boundaries and near the porosity. Also, in a similar study [52], the hardness of the Al-4.5Cu alloy was measured as approximately 90 HB (Brinell Hardness), while the hardness of the 6wt% SiC doped Al-4.5Cu alloy was measured as approximately 120 HB. In this study, as seen in Figure 8, the hardness of Al-4.5Cu/TiO_2 composites produced by milling for 10 h was measured as approximately 162, 170, and 173±10 HV.

Besides, the surface properties of pressed, sintered, polished, and etched Al4.5Cu/TiO_2 composite samples were examined with an optical microscope (OM). Figure 9 shows the OM images of the composites, as a typical example of composite fabricated by the dispersion of TiO_2 in Al-4.5Cu alloy. In the images, TiO_2, which is a reinforcing material in the composite, is seen in gray, the matrix material Al4.5Cu is white, and the porosities are black colour. When the images were examined, a completely homogeneous structure was not formed in the samples with a milling time of 5h. With increasing milling time from 5 h to 10 h, the size of particles and agglomerations was decreased due to continuous fracturing and re-welding, and also a more homogeneous structure was formed. With the increasing of percentage of reinforcement TiO_2 particles, the porosity and particle size increased. The unregulated and clustered precipitates of bright Al4.5Cu particles are seen in the matrix, which is surrounded in the form of grain boundary by TiO_2. Due to the non-uniform distribution of TiO_2 particles in the matrix material, porosities and gap defects have occurred in the grain boundaries. These results show that pressing pressure and milling time should be increased to create a perfect structure for these samples. As a result, this study is in line with the literature [49-61] studies, and it has been clearly seen that the mechanical properties of the

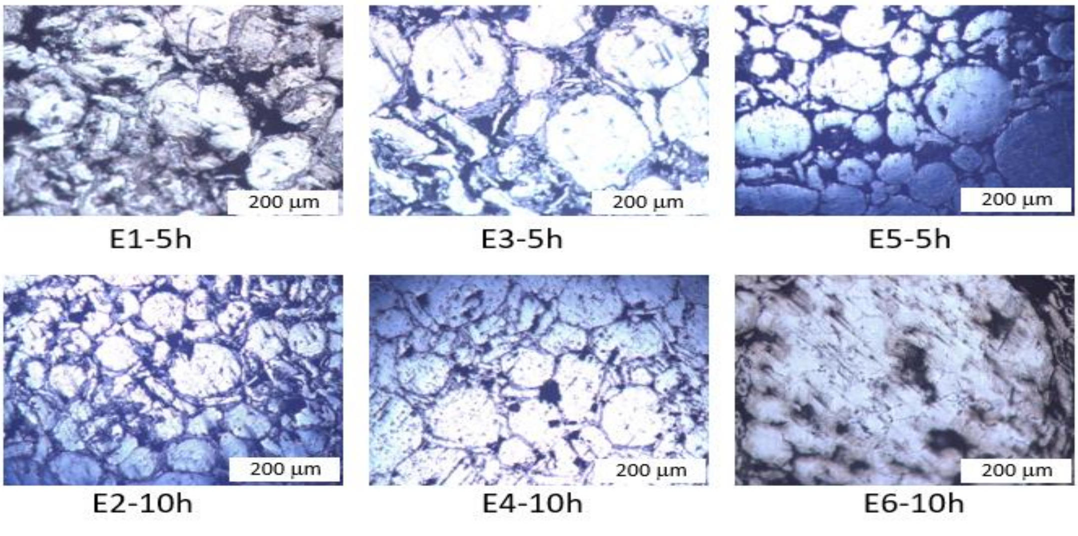

Figure 9. Optical microscope images of sintered Al4.5Cu/TiO_2 composite samples

Al-4.5Cu/TiO_2 composite powders produced by the mechanical alloying technique have improved compared to that studies conducted.

4. CONCLUSION

The Al4.5Cu/TiO_2 metal matrix composite powder is fabricated from its elemental powders by mechanical alloying technique. Thermal and microstructural analyses of the produced composites were carried out. The results are given below;

- As indicated from XRD results, Al, Al(Cu), Cu, and TiO_2 phases were observed in all of the Al4.5Cu/TiO_2 composite powder samples. The absence of a different intermetallic phase showed that the desired composite was produced.
- As a result of sintering, a weak peak intensity of intermetallic phases such as Al_2Cu and AlTi were observed.
- DTA curve of the composite showed one endothermic peak in the range of 500-600 ºC. This endothermic peaks indicating the melting of Al(Cu) and α-Al phases. Also, one exothermic peaks observed in the 700-750 ºC temperature range indicate that the AlTi phase was formed in the composites.
- According to SEM results, with increasing the milling time from 5h to 10h, the particles have reached a more homogeneous and spherical structure with softer edges. Also, it was observed that as the milling time increased, the particle size decreased and fusion started. It has been observed that there are aggregations in the Al4.5Cu matrix as the amount of TiO_2 reinforcement increases from 10wt% to 20wt%.
- The crystallite size of 10h mechanically milled and 10wt% TiO_2 doped Al4.5Cu/TiO_2 powder was found to be 21.5±2 nm.
- The maximum microhardness value was found to be 173±10 HV as a result of sintering of the composite produced with 20wt% of TiO_2 and milling for 10 h.

ACKNOWLEDGEMENT

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DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Mustafa OKUMUŞ: Performed the experiments, Data analysis, Writing - original draft, Writing - review & editing, Supervision.

Esma KAYA: Performed the experiments, Data analysis, Writing - original draft.

Musa GÖĞEBAKAN: Data analysis, Writing - review & editing,

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