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Araştırma Makalesi / Research Article

Production and Characterization of Al-SiC Composites Prepared by Mechanical Milling and Pressureless Sintering

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Abstract
This study investigates the effect of SiC amount and milling time on microstructure, density and hardness in the aluminum composites produced by mechanical milling. In the study, aluminum composites were mechanical milled for different periods (30, 60, 90 and 120 min), adding SiC at different amounts (5, 10, 15 and 20 wt. %). High chromium steel balls with a diameter of 6 mm, a ratio of 10:1 ball-powder and 2% stearic acid were used as milling component in the production of composites in a vibrating mill. The production of composite powders was carried out in an argon atmosphere. The composite powders produced were characterized using scanning electron microscopy, EDS analysis, optical microscope and powder size analyzer. As a result, it was observed that as the amount of added SiC and milling time increased, the powder size decreased, and the hardness values increased. In addition, as the amount of added SiC increased, the densities of aluminum composites increased.

Keywords: Aluminum composite, mechanical milling, powder metallurgy, vibrating mill, SiC.

Mekanik Öğütme ve Basınçsız Sinterleme ile Al-SiC Kompozitlerin Üretimi ve Karakterizasyonu

Öz
Bu çalışmada, mekanik öğütme ile üretilen alüminyum kompozitlerde SiC miktarının ve öğütme süresinin mikro yapı, yoğunluk ve sertlik üzerine etkisi incelenmiştir. Çalışma kapsamında alüminyum kompozitler, farklı miktarlarda (%5, %10, %15 ve %20) SiC ilave edilerek farklı sürelerde (30, 60, 90 ve 120 dak) mekanik öğütülmüştür. Titreşimli değirmende yapılan kompozitlerin üretiminde öğütme elemanı olarak 6 mm çapında yüksek kromlu çelik bilya, 10:1 bilya-toz oranı ve %2 stearik asit (işlem kontrol kimyasalı) kullanılmıştır. Kompozit tozların üretimi argon ortamında yapılmıştır. Üretilen kompozit tozlar tarama elektron mikroskobu, EDAX analizleri, optik mikroskop ve toz boyut analizörü kullanılarak karakterize edilmiştir. Yapılan çalışmalar sonucunda, ilave edilen SiC miktarı ve öğütme süresi arttıkça toz boyutunda azalma görülürken, sertlik değerlerinde artış görülmektedir. Bununla birlikte ilave edilen SiC miktarı arttıkça da alüminyum kompozitlerin yoğunlukları artmaktadır.

Anahtar Kelimeler: Alüminyum kompozit, mekanik öğütme, toz metalürjisi, titreşimli değirmen, SiC.

1. Introduction

Today, many industrial applications require materials with lower density (light) and higher rigidity and strength. Aluminum matrix composites (AMC) have become ideal materials for these requirements due to their various advantages [1, 2]. Many researchers have used different production techniques to investigate AMCs, which can be produced using liquid or solid-state techniques reinforced with hard particles such as carbides, oxides, and nitrides [3]. Al2O3 and SiCp reinforced aluminum composite materials (ACM) are increasingly preferred due to their very good mechanical and physical properties,

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lightness, suitability to high temperature applications and good wear resistance [4]. Different methods such as in-situ (in-situ nucleation) [6], spray [6], and PVD (physical vapor deposition) have been used in the studies [7]. Liquid state processes such as infiltration, squeeze casting, stir casting [8], powder metallurgy (liquid phase sintering) [9], and spray deposition [10] stand out among these methods. Powder metallurgy (PM) is a solid-state production method which can successfully be carried out at and below room temperature. Numerous researchers have previously produced composite materials using MA method [11-15]. The powder metallurgy method is known to be a good method to produce particle-reinforced metal matrix composites. Low treatment temperature is a significant advantage of the powder metallurgy (PM) method compared to melting techniques [11]. Khadem et al. [16] investigated the structure and morphology of the 5% SiC reinforced composites produced by attritor type mechanical grinder with various milling times. They reported that hardness of the material increases and the grain size decreases by the increasing grinding time. Simsek et al. [17] investigated the variation of microstructure, hardness and density of the composites produced in an attritor mill which are produced at different ratio and milling time. Studies report that the hardness and density of the material increase by the increasing ratio of reinforcement in the matrix, and the grain size decreases and the hardness increases by the increasing milling time. Ozyurek and Tekeli [4] used mechanical alloying method to produce Al-SiC composite material which is a powder metallurgy method. They reported that as the ratio of reinforcement increases, hardness value increases and the reinforcement material is distributed in the matrix more uniformly. Although the P/M method is accepted to be a good method to produce metal matrix composites, some problems are encountered in production of AMCs using this method [18]. One of the most significant problems encountered is the flocculation of particles added into the Al matrix and the non-homogeneous distribution of the ceramic reinforcement elements. Different methods are used to prevent such problems, one of which is the use of different mill types depending on the application's purpose [19].

Mechanical alloying/milling method, which is a powder metallurgy technique, employs different equipment. This equipment has different alloying/milling capacity and capabilities. In this study, commercially pure Al and SiC ceramic reinforcement element was mechanically milled with different amounts and different milling times using a mechanical vibration mill (in argon atmosphere). The purpose of the study was to investigate the effects of the different SiC amounts and milling times on the hardness, density and microstructure of the AMC mechanically milled using a vibration mill.

2. Material and Method

The effects of different SiC amounts (5%, 10%, 15%, 20%) and milling times (30, 60, 90, 120 min) on AMCs milled in a vibration mill were investigated in this study. 99.5% purity and 53 µm diameter gas atomized Al powder, and 13 µm diameter SiC powder were used to produce particle reinforced Al-SiC composite powders. A ball/powder ratio of 10:1 and 6 mm high-chrome steel balls are used in the vibration mill, and 2% stearic acid was used as the process control agent. The powder and ball charge was performed in argon environment for mechanical milling (MM). The composite powders produced were moved into glove box (under argon). Figure 1 shows the schematic view of the vibration mill used in experimental studies.

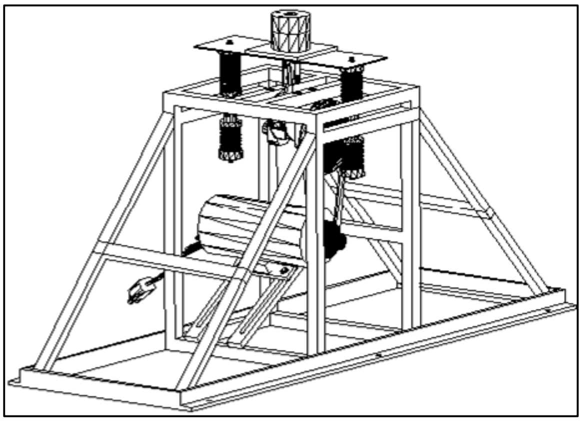

Figure 1. Schematic view of the vibration mill

Powder size measurements and optical and scanning electron microscope examinations were performed to determine the powder size and shape depending on the MM time. The powders were cold-pressed under 630 MPa pressure to produce preformed composite samples. These samples were sintered in argon environment at 560 °C for 30 minutes. After all samples were prepared by standard metallographic procedures for microstructure studies, they were etched for 15-20 s with 1 ml HF, 1.5 ml HCl, 2.5 ml HNO3 and 95 ml H2O (Keller's) solution. Hardness measurements of the sintered aluminum composite samples were performed using an Affri universal hardness tester (HV2). Hardness of the material was measured at 10 different points of three samples and the mean value was calculated. Powder size analysis of the composite powders was performed using the laser technique in a Malvern Mastersizer-X device mixing the powders with distilled water and stirring for 5 minutes. Density measurements were performed according to Archimedes principle using PRECISA XB 220A density measuring device with a max capacity of 220 g and a precision of 1/10000 g. Density was measured at three samples and the mean density value was calculated.

3. Results and Discussion

Figure 2 shows the change in powder size depending on milling time for Al-SiC composites produced in a vibration mill using different milling times.

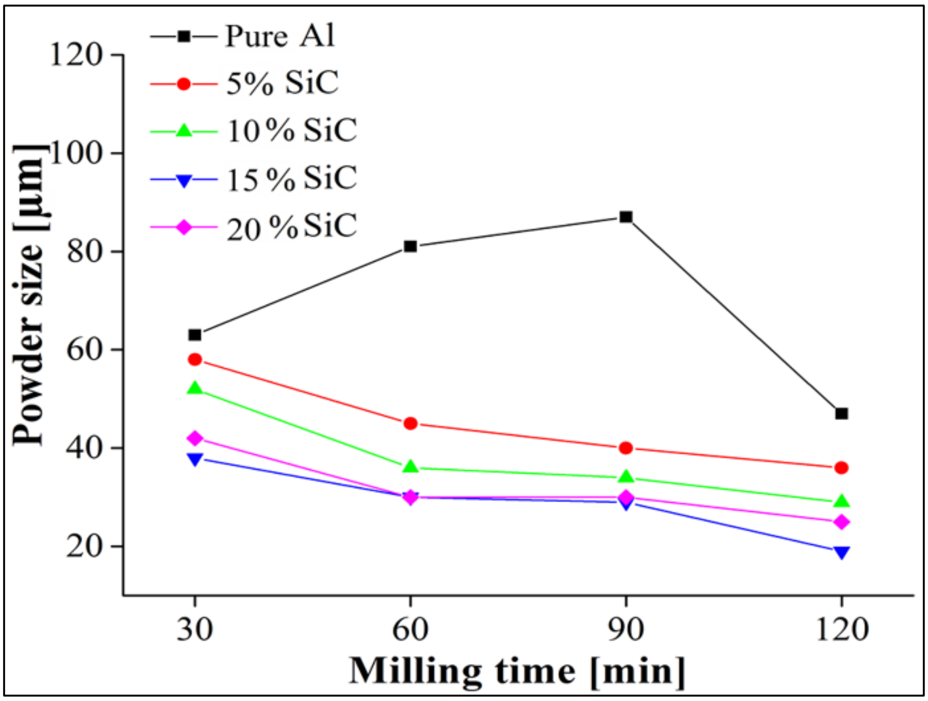

Figure 2. The change in powder size depending on milling time for Al-based composites mechanically milled using different milling times

As shown in Figure 2, the pure aluminum powders mechanically milled for 30-90 min showed an increase in size (63-87 µm), while a decrease was observed in powder size (81 µm) when a milling time of 120 min was used. This is due to the enlargement of ductile metal powders with cold welding. Ductile powders which present a cold welding effect in the first stage of mechanical milling undergo deformation hardening in parallel with the increase in milling time, which causes them to fracture and shrink [20]. Powders in composites containing different amounts of SiC fracture and shrink due to constant milling effect. For this reason, the powder size in aluminum composites containing SiC decreases with increasing milling time. Similar results have been obtained in previous studies as well [15, 17, 19]. The powder size chart shows that the smallest powder size was obtained for the aluminum composites containing 15% SiC. Figure 3 shows the optical microscope images of the Al + 20% SiC composite powders mechanically milled in the vibration mill using different milling times.

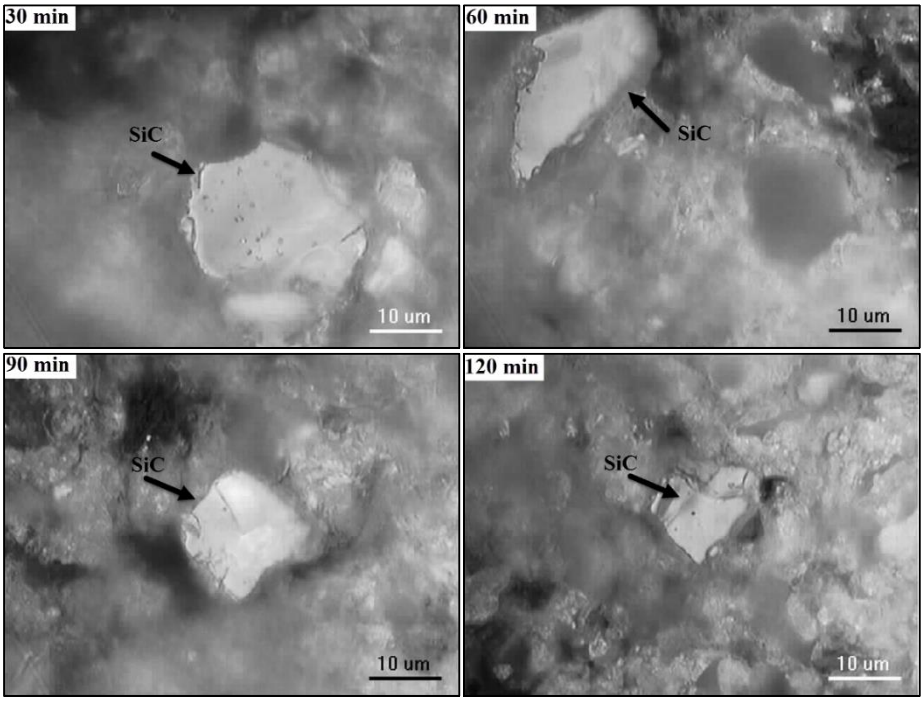

Figure 3. The optical microscope images of the AMCs containing 20% SiC mechanically milled using different milling times

As evident from Figure 3, the optical microscope images of the aluminum composite powders containing 20% SiC showed a more coarse appearance after 30 min of mechanical milling compared to other mechanically milled composite powders. The SiC particles added to the matrix were observed to fracture and shrink more as a result of 120 min of MM compared to other milling times. One of the most significant problems encountered in the production of particle reinforced composite materials is the non-homogeneous distribution of the ceramic reinforcement elements [21]. It is a very common occurrence that reinforcement elements do not show a homogeneous distribution when short milling times are used. The increase in mechanical milling time allows for producing composite powders with a more homogeneous distribution. A relatively more homogeneous reinforcement material distribution has been achieved using longer milling times in a previous study [18].

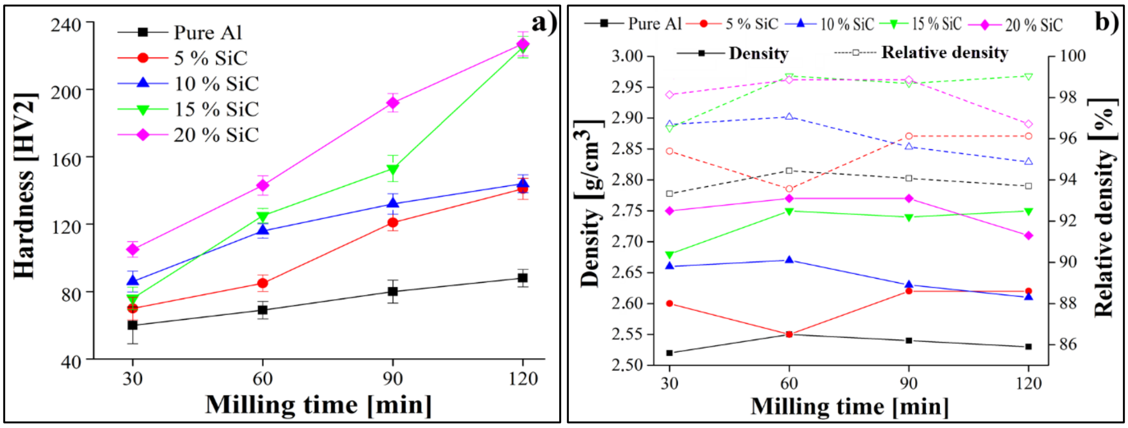

Figure 4. The change in hardness (a) and density (b) depending on milling time for AMCs mechanical milled using different milling times

As shown in Figure 4.b, the density of pure aluminum powders was lower than the known density of aluminum (2.7 gr/cm^3) after the MM process. The composite materials containing 5%, 10%, and 15% SiC had increased density proportional to reinforcement material amount after 60 minutes of MM. In case of 90 minutes of MM, the composite materials containing 5% and 20% SiC once again displayed an increase in density, while the density of the pure Al and the composite materials containing

10% and 15% SiC decreased. Parvin et al. [15], have reported that there was a decrease in density due to milling time, and the reason behind this decrease was the deformation hardening caused by mechanical milling. Density of the sample MM'ed for 120 min decreased which means that, the density and particle size are inversely proportional. This is due to the partial density increase in produced composites. The partial density and the measured density vary inversely to each other. The main determining factor in the increase of partial density is the increase of surface roughness of the powder. The form and surface conditions of powder change with the increasing surface roughness during MA/MM. While surface roughness of the powder increases, actual measured density decreases as milling time increases. The highest density value was obtained for the composite materials containing 20% SiC, whereas the lowest density value was obtained for the composite materials containing 5% SiC The density increased as the amount of SiC added to the matrix increased. This is a caused by the higher density of SiC compared to Al. The lowest hardness value was obtained for the pure aluminum as shown in Figure 4.a. The hardness value increased as the SiC amount added to the aluminum matrix and milling time increased. The lowest hardness value for the composite materials containing 20% SiC was 105 HV obtained after 30 minutes of milling, whereas the highest hardness value was 227 HV obtained after 120 minutes of milling. Zhao et al. [18], have reported that powders which had not been deformed with MM were softer, the deformation increased with increasing milling time, and the hardness value increased in parallel. Figure 5 shows the SEM images (a) EDS results for the composites containing 10% SiC after 120 (b) and 90 (c) minutes of milling.

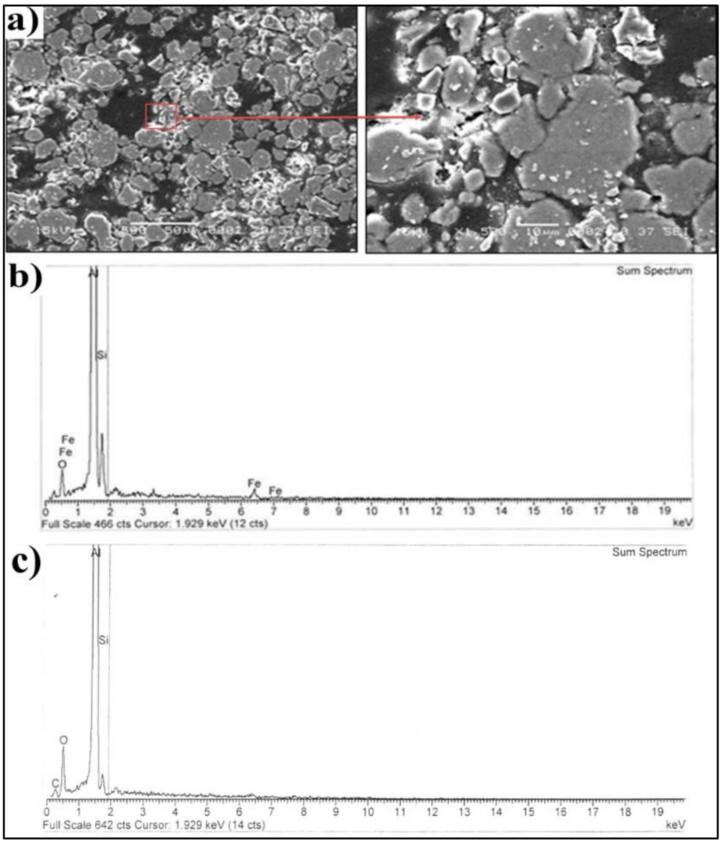

Figure 5. The SEM images (a) of AMCs containing 10%SiC, and EDS results for 120 min (b) and 90 min (c) of milling

The SEM images given in Figure 5.a show that SiC particles (with sharp edges) within the matrix sunk into the matrix. The EDS analysis (Figure 5.b. and Figure 5.c.) indicates no Fe contamination in the SiC reinforced Al composite materials produced by grinding for 90 min in the vibration mill. However, there was some staining (such as Fe) in the SiC reinforced Al composite materials produced in vibration mill due to the milling equipment used. Although the MM process was performed in an atmosphere-controlled environment (argon environment), some O_2 staining was observed as well. During the milling process, constant crashes between ball-powder and ball-powder-milling chamber lead to clean surfaces on powders (within the milling chamber). Oxidation may occur on these clean surfaces due to the small amount of O_2 within argon or due to contact with atmosphere while taking the powders from the mill.

4. Conclusion

The following results were obtained as a result of this study conducted to investigate the effects of the SiC amount and the mechanical milling time on SiC reinforced aluminum composites produced using a vibration mill.

The powder size of the composite powders produced by MM decreased with increasing mechanical milling time. Also, the powder size was observed to decrease with increasing amount of reinforcement phase (SiC). The smallest powder size was obtained for the composite material containing 20% SiC and milled for 120 min.

In general, the increase in the milling time led to a decrease in the density of the aluminum composites, albeit slightly. Also, the density of the composites was observed to increase in parallel with the increase in the amount of reinforcement element.

The hardness value of the composites produced by MM increased with the increasing amount of SiC. Also, the increase in the milling time led to an increase in hardness as well.

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