Avrupa Bilim ve Teknoloji Dergisi
Özel Sayı 28, S. 758-765, Kasım 2021

Araştırma Makalesi

www.ejosat.com ISSN:2148-2683

European Journal of Science and Technology
Special Issue 28, pp. 758-765, November 2021

Research Article

Investigation of Mechanical Properties of Si_3N_4 Reinforced Composites Produced from Aluminum Waste[i]

Burak Öztop [1*], Mevlüt Gürbüz [2]
[1*] Amasya University, Merzifon Vocational School, Amasya, Turkey, (ORCID: 0000-0003-3947-9103), burak.oztop@amasya.edu.tr
[2] Ondokuz Mayıs University, Departmant of Mechanical Engineering, Samsun, Turkey, (ORCID: 0000-0003-2365-5918), mgakademik@gmail.com

(1st International Conference on Applied Engineering and Natural Sciences ICAENS 2021, November 1-3, 2021)

(**DOI:** 10.31590/ejosat.1010738)

ATIF/REFERENCE: Öztop, B., Gürbüz, M. (2021). Investigation of Mechanical Properties of Si_3N_4 Reinforced Composites Produced from Aluminum Waste. *European Journal of Science and Technology*, (28), 758-765.

Abstract

In this study, first of all, waste aluminum beverage cans were melted and poured into molds to obtain matrix material in the form of ingots. Stir casting process was used for composite material production. 0.5%, 0.75% and 1% by weight Si_3N_4 reinforcement was added to the matrix material. The effects of reinforcement ratios on the density, hardness, tensile strength, impact strength and microstructure of composite materials were investigated. In Si_3N_4 reinforced composites, tensile strength increased from 54,5 MPa to 105,17 MPa, impact strength increased from 4,94 J to 13,18 J, and hardness increased from 70 HV to 87,9 HV depending on the increased reinforcement ratio. Also, in the density measurements, density and porosity ratio increased with the increase of the reinforcement ratio. From the scanning electron microscope (SEM) images of the produced composites, it was determined that the particle distribution increased with the increase in the reinforcement ratio and the reinforcement material was homogeneously dispersed.

Keywords: Waste Aluminum, Graphene, Stir Casting, Aluminum Matrix Composite, Mechanical Property, Microstructure.

Alüminyum Atıktan Üretilmiş Si_3N_4 Takviyeli Kompozitlerin Mekanik Özelliklerinin İncelenmesi

Öz

Bu çalışmada öncelikle, atık alüminyum içecek kutuları ergitilip kalıplara dökülerek külçe halinde matris malzemesi elde edilmiştir. Kompozit malzeme üretimi için karıştırma döküm yöntemi kullanılmıştır. Matris malzemesine ağırlıkça % 0.5, % 0.75 ve % 1 oranlarında Si_3N_4 takviyesi yapılmıştır. Takviye oranlarının kompozit malzemelerin yoğunluk, sertlik, çekme dayanımı, darbe dayanımı ve mikroyapısına etkisi incelenmiştir. Si_3N_4 takviyeli kompozitlerde, artan takviye oranına bağlı olarak, çekme mukavemeti 54,5 MPa'dan 105,17 MPa'ya, darbe mukavemeti 4,94 J'den 13,18 J'ye ve sertlik 70 HV'den 87,9 HV'ye yükselmiştir. Yapılan yoğunluk ölçümlerinde takviye oranının artmasıyla yoğunluğun ve gözenek oranının arttığı görülmüştür. Üretilen kompozitlerin taramalı elektron mikroskobu (SEM) görüntülerinden, takviye oranının artmasıyla partikül dağılımının arttığı ve takviye malzemesinin homojen olarak dağıldığı belirlenmiştir.

Anahtar Kelimeler: Atık Alüminyum, Grafen, Karıştırma Döküm, Alüminyum Matrisli Kompozit, Mekanik Özellik, Mikroyapı.

[*] Corresponding Author: burak.oztop@amasya.edu.tr

1. Introduction

The need for advanced technology materials in important fields such as defence, aerospace, automotive, and maritime is increasing. Single component materials do not meet the needs of advanced engineering applications such as resistance to high temperatures associated with the melting point of the material, high strength, high elastic modulus, high fracture toughness, high fatigue strength, high wear resistance and low density. Therefore, the need for composite materials with superior properties is increasing day by day (Özkök, 2004; Kılıç, 2008; Nazik, 2013).

Metal matrix composite (MMC) materials have become an important engineering material by combining the ductility and toughness of metal materials and the high strength properties of ceramic materials. Aluminum (Al) and its alloys are widely used in the production of MMC as matrix materials due to their easy formability, low density, high electrical and thermal conductivity. As reinforcement material, silicon carbide (SiC), silicon nitride (Si_3N_4), aluminum oxide (Al_2O_3) etc. ceramic particles are used. Si_3N_4, one of these ceramics, has become an important reinforcement material due to its properties such as high hardness, strength, fatigue life, wear resistance, oxidation resistance and corrosion resistance, high thermal shock resistance due to its low coefficient of thermal expansion (Özkök, 2004; Kılıç, 2008; Kalemtaş, 2014).

Aluminum matrix composites have superior properties such as high modulus of elasticity, high strength, high fatigue resistance, high abrasion resistance, high rigidity, stability at high temperature, high strength to weight ratio, low coefficient of thermal expansion and low density. Due to these properties, the production of aluminum matrix composites and their use in the automotive, aerospace and aviation industries are increasing. Generally, aluminum matrix composites are produced by powder metallurgy or liquid phase methods. However, they are produced more easily via the liquid phase as particle reinforced. One of the most used methods in liquid phase production techniques is the stir casting method because it is both a low cost and a simple method (Hashim et al., 1999; Sur et al., 2005; Surappa, 2003).

The recycling of aluminum, which has been used in many fields, especially in construction, aviation, space and transportation in recent years, is important for the protection of the environment as it will reduce the use of both natural resources and energy resources (Pulat et al., 2014). Beverage cans stand out are both one of the areas where aluminum is most widely used and the cornerstone of the aluminum recycling market (Ediz, 2011; İzgi, 2011). In line with this information, it is predicted that the commercial use of aluminum matrix composite materials will become widespread by reducing the production costs by using waste aluminum beverage cans, which are industrially reusable, as matrix material in the production of composite materials.

Gui et al. (2000), in their studies involving the production of SiC reinforced Al-Si matrix composite materials by casting, applied the semi-solid mixing process after the liquid mixing process. They stated that the tensile strength of reinforced composite materials ((15%, 20% by volume) increased by 15% and 26%, yield strength by 40% and 54%, and modulus of elasticity by 30% and 40%, respectively, compared to the unreinforced matrix material. Kok (2005), produced composite material by reinforcing Al_2O_3 particles in different sizes (16, 32, 66 μm) and different percentage weight ratios (10%, 20%, 30%) to 2024 aluminum alloy by applying vortex method and then pressing under pressure. Density measurements have shown that the samples contain very little porosity. He found that the amount of porosity increased with increasing percent by weight reinforcement ratio and decreasing particle size. The hardness and tensile strength of the composites increased with increasing particle weight percentage and decreasing particle size. Singla et al. (2009), used a stir casting technique in which two-stage mixing is applied to develop MMK with aluminum-based SiC particles and to ensure homogeneous distribution of ceramic material in the matrix during production. The alloy was mixed by hand while it was in a semi-solid state, and then it was heated until it became completely liquid and was mixed with a mechanical mixer. The experiments were carried out by changing the SiC reinforcement ratio to 5%, 10%, 15%, 20%, 25% and 30% by weight. It was observed that the hardness and impact strength increased as the weight percentage of SiC increased. The best results were obtained at a reinforce rate of 25% by weight. With the increase of this reinforcement ratio, hardness and impact resistance values decreased due to aggregation of SiC particles. Bajaj (2011), has produced composite materials with SiC, Al_2O_3 and SiC+Al_2O_3 reinforced (2.5%, 5, 7.5, 10 wt%), aluminum casting alloy (LM6) matrix using the stir casting method. The hardness, tensile and impact tests and mechanical properties of the obtained samples were investigated. It was determined that the hardness, tensile strength and impact strength increased with the increase in the reinforcement ratio. The highest increase in mechanical properties was determined in the composites obtained by adding SiC and Al_2O_3 reinforcement elements together. Sujan et al. (2012), have investigated the physical and mechanical properties of SiC and Al2O3 particle reinforced aluminum matrix composites. Composite materials were produced by the stir casting method by adding 5, 10, 15% Al_2O_3 by weight and SiC at the same proportions to Al356 alloy. The results showed that composite materials exhibit improved physical and mechanical properties such as high tensile strength, high impact strength, high stiffness and low coefficient of thermal expansion. Singh et al. (2013), have produced aluminum matrix composites using the mixed casting method. The effects on hardness, impact strength and tensile strength of input process parameters such as Al_2O_3 particle size (75, 105, 150 μm), weight percent Al_2O_3 (3, 6, 9%) and mixing time (15, 20, 25 min) were investigated. All these mechanical properties increased with the mixing time and the increase in the weight percent of the reinforcement element and the decrease in the reinforcement particle size. Hindi et al. (2015), have produced composite material by reinforcing 2, 4 and 6 wt% SiC particles to Al 6063 aluminum alloy by using stir casting method. It was determined that with the increase of SiC reinforcement, ductility decreased, while hardness and tensile strength increased. It was observed that the impact strength initially increased and then decreased with increasing SiC reinforcement. Sharma et al. (2015), have investigated the mechanical properties of aluminum matrix composites produced by conventional stir casting by adding Si_3N_4 particles at various weight percentages (3, 6, 9, 12) to the AA6082-T6 alloy. Mechanical properties such as tensile strength and hardness improved as the weight percentage of Si3N4 particles in the aluminum matrix increased. Density and porosity showed an increasing trend. Kumar et al. (2015), have produced Si_3N_4, AlN, ZrB_2 reinforced Al2618 matrix composites at 0, 2, 4, 6, 8 wt% ratios by stir casting method. They determined that the hardness, tensile strength, compressive strength and corrosion resistance of the composites increased with increasing reinforcement ratio.

In this study, the effects of Si_3N_4 reinforcement at different reinforcement ratios (0.5, 0.75, 1 wt%) on the density, hardness, tensile strength, impact strength and microstructure of composites produced from waste aluminum by stir casting method were investigated.

2. Material and Method

2.1. Matrix and Reinforcement Material

In this study, waste aluminum beverage cans were used as matrix material instead of pure aluminum or commercial aluminum alloys used in previous studies. Si_3N_4 powders were used as reinforcement material. Si_3N_4 has been preferred especially because of its superior mechanical properties such as high strength, hardness, wear resistance and fracture toughness. Since the Si_3N_4 powder particle size used is 0.5-0.8 µm on average, it was decided to use low reinforcement ratios (0.5, 0.75, 1 wt%) considering the problems such as agglomeration of the powder and inability to ensure the homogeneity of the mixture in the reinforcements to be made at high rates.

2.2. Production of Composite Material

First of all, waste aluminum beverage cans were sequentially thrown into the melting pot at a temperature of approximately 1000 ºC and the paint layer on them was burned. Aluminum ingots with an average weight of 170 g were obtained by melting 16 beverage cans for each matrix sample. Electric melting furnace with mixing system has been designed and manufactured for the production of composite materials. An electric furnace that can be adjusted of up to 900 ºC temperature was obtained by connecting a 1.5 kW single louvered ceramic plate resistance on the outer crucible made of stainless steel material. A single module control device and M12 K-type thermocouple with a standard 8 mm union were used to control the temperature inside the furnace. Also, another crucible made of stainless steel was placed inside the furnace. This crucible is designed as both a melting and casting crucible. The mixing shaft used to perform the mixing process is made of stainless steel material and has four wings with a diameter of 40 mm at one end. In order to mix the reinforcement materials into the matrix material, a desktop mechanical mixer with an adjustable speed (200-3000 rpm) was used.

Si_3N_4 particles were oxidized in the heat treatment furnace at 1100 ºC for 5 hours to increase their wettability. Si_3N_4 particles were prepared by weighing them according to the determined by weight percent reinforcement ratios. The matrix material in the crucible was melted by increasing the temperature of the melting furnace to 700 ºC. During the addition of reinforcement and stirring process, the temperature was reduced to between 630-650 ºC to facilitate the wettability of the reinforcement material and to form the matrix-reinforcement interface. The reinforcement material, which was prepared by weighing for each ratio, was added to the matrix material in three equal amounts. After the first and second parts of the reinforcement material were added, the mixture was stirred at 400 rpm for 30 seconds. After the third part of the reinforcement material was added, the mixing process was applied at 400 rpm for 5 minutes. The temperature was increased to 700 ± 10 ºC and the mixture was mixed at the same speed for 1 minute, and homogenization was made. The mixture was kept at this temperature for 10 minutes and the oxide film formed on the surface of the liquid melt was removed just before casting. Immediately after, the mixture was poured into a preheated (450-500 ºC) metal mold, and the composite material was left in the mold for a while, then removed from the mold and allowed to cool at room temperature. By applying this method, by adding 0.5%, 0.75% and 1% by weight Si_3N_4 to the aluminum matrix, 15 pieces of composite materials were produced, 5 for each ratio. Composite materials produced were coded as shown in Table 1 in order not to mix the samples during the application of the tests and to record the test results correctly.

Table 1. Codes of Si_3N_4 reinforced composite materials

Material	Code
Al	AL
Al - % 0.5 Si_3N_4	ALSN05
Al - % 0.75 Si_3N_4	ALSN075
Al - % 1 Si_3N_4	ALSN1

2.3. Hardness Test

Hardness measurements were made to determine the hardness values of the unreinforced matrix material and composite materials and to determine the hardness changes that occur with increasing the reinforcement ratios. Hardness measurements were carried out under 1.96 N (200 g) load for 20 seconds on a TMTECK brand HV-1000B type hardness measuring device using vickers hardness measurement method. Measurements were made 5 times for each sample and the hardness values were determined by taking the average of the measured values.

2.4. Tensile Test

Tensile test was applied to evaluate the mechanical behavior of Si_3N_4 reinforced composite materials and non-reinforced matrix material and to determine the changes in tensile strengths with increasing reinforcement ratios. A total of 12 tensile test specimens were prepared, three of each of the unreinforced matrix and composite materials. Tensile tests were carried out on a 10 ton capacity Mares brand tensile testing device. The tensile test was performed at a constant speed of 5 mm/min for all samples. The tensile strength values of both the unreinforced matrix and the composite materials were determined by taking the average of the measured values for each material type.

2.5. Impact Test

This test was applied to determine the amount of energy required for the fracture of unreinforced matrix and Si_3N_4 reinforced composites under dynamic stress and to determine the changes in impact strength depending on the increasing ratio of reinforcement by weight. Three test specimens were prepared from each of the non-reinforced matrix and composite materials. The average impact energy was calculated by taking the average of 3 test results for each material in ALŞA brand ZBC 2000 model charpy impact test device.

2.6. Density Measurement and Porosity Ratio

Experimental densities (ρ_D) of Si_3N_4 reinforced composite materials and non-reinforced matrix material were determined using Archimedes method. The saturated mass (m_D), suspended mass (m_A) and post-drying dry mass (m_K) of the samples were measured, respectively. The experimental densities of the samples were calculated using Equation (1).

$$\rho_D = [m_K/(m_D - m_A)] \times \rho_{SU} \quad (1)$$

Then, the theoretical densities (ρ_T) of all materials were determined by using Equation (2) according to the mixtures rule. Porosity ratios were calculated using Equation (3).

$$\rho_T = [(\text{density of matrix x percent matrix weight ratio}) + (\text{density of reinforcement material x percent reinforcement weight ratio})] \quad (2)$$

$$\%\ \text{porosity} = (\rho_T - \rho_D / \rho_T) \times 100 \quad (3)$$

2.7. Microstructure Analysis

X-ray diffraction (XRD) analyzes of composite materials and Si_3N_4 powders used in composite production were performed with Rigaku Smartlab brand X-ray diffraction device. EDS equipped Jeol JSM 7001F type scanning electron microscope (SEM) was used for the microstructure examinations performed on the fractured surfaces of the samples broken after the impact test. In addition, SEM analyzes of the reinforcement material used in the production of composites were made.

3. Results and Discussion

3.1. Hardness

The measured hardness values of unreinforced matrix material and Si_3N_4 reinforced composite materials are given in Table 2. In addition, the changes in the hardness values of the materials according to the reinforcement ratio are given graphically in Figure 1.

Table 2. Hardness test results

Material	Hardness (HV) 1st	Hardness (HV) 2nd	Hardness (HV) 3rd	Hardness (HV) 4th	Hardness (HV) 5th	Average Hardness (HV)
AL	70,15	68,5	71,16	69,72	70,47	70
ALSN05	81,48	77,61	78,58	80,53	81,3	79,9
ALSN075	84,37	83,61	85,4	83,85	85,07	84,46
ALSN1	88,78	87,18	86,27	89,58	87,69	87,9

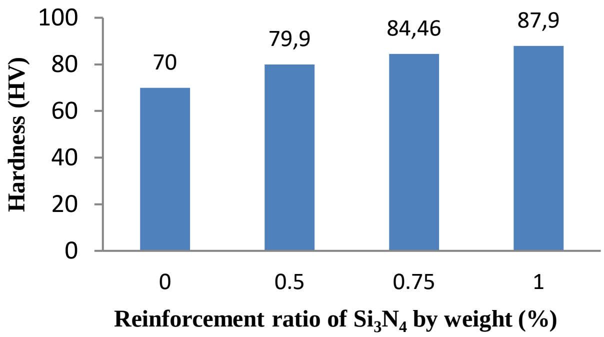


Figure 1. Change of hardness of the materials with Si_3N_4 percent weight ratio

In the literature review, it was determined that the hardness of the composite materials increased with the increase in the reinforcement ratio (Bajaj, 2011; Hindi et al., 2015; Kok, 2005; Kumar et al., 2015; Sharma et al., 2015; Singh et al., 2013, Singla et al., 2009; Sujan et al., 2012). The hardness of composite materials increased from 70 HV to 87.9 HV with the increase of Si_3N_4 weight percent. It has been observed that this result is consistent with the information in the literature.

3.2. Tensile Strength

The tensile strength values calculated by taking the average of the values obtained as a result of the tensile tests are given in Table 3. The graph showing the change of tensile strength values of materials according to reinforcement ratio is given in Figure 2.

Table 3. Tensile test results

Material	Tensile Strength (MPa) 1st test	Tensile Strength (MPa) 2nd test	Tensile Strength (MPa) 3rd test	Average Tensile Strength (MPa)
AL	54,29	55,11	54,1	54,5
ALSN05	81,85	80,14	80,56	80,85
ALSN075	92,49	93,53	93,28	93,1
ALSN1	105,1	104,89	105,52	105,17

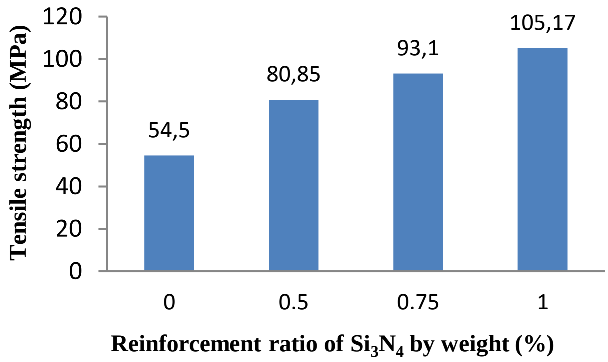


Figure 2. Change of tensile strength with Si_3N_4 percent weight ratio

In previous studies, it has been determined that increasing reinforcement ratio increases the tensile strength of composite materials (Bajaj, 2011; Gui et al., 2000; Hindi et al., 2015; Kok, 2005; Kumar et al., 2015; Sharma et al., 2015; Singh et al., 2013; Sujan et al., 2012). The tensile strength of composite materials increased from 54.5 MPa to 105.17 MPa with the increase of Si_3N_4 weight percent. It has been observed that this result is consistent with the information in the literature.

3.3. Impact Strength

The average impact strength values were calculated by taking the averages of the values obtained as a result of the impact tests (Table 4). The graph showing the change of the impact strength values of the materials according to the reinforcement ratio is also given in Figure 3.

Table 4. Impact test results

Material	Impact Strength (J) 1st test	Impact Strength (J) 2nd test	Impact Strength (J) 3rd test	Average Impact Strength (J)
AL	4,62	5,22	4,98	4,94
ALSN05	6,81	6,21	6,66	6,56
ALSN075	11,58	10,98	12,45	11,67
ALSN1	12,65	13,85	13,04	13,18

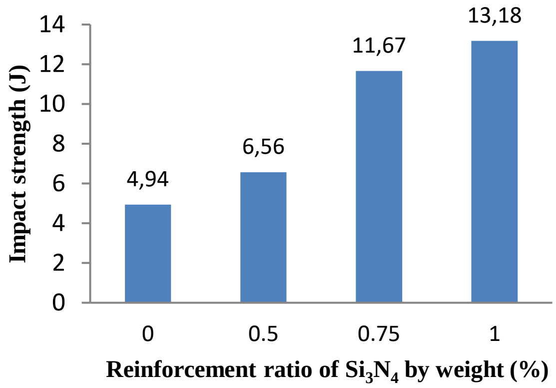


Figure 3. Change of impact strength with Si_3N_4 percent weight ratio

There are different results in the literature regarding the changes in impact strength of composite materials depending on the increasing reinforcement ratio. However, it was mostly seen that the impact strength increased with increasing reinforcement ratio. Impact resistance values increase in production methods where the reinforcement material is well wetted by the matrix material and the formation of undesirable secondary phases at the interface is not formed or the formation is reduced (Bajaj, 2011; Singh et al., 2013; Singla et al., 2009; Sujan et al., 2012).

In this study, it was observed that the impact strength increased from 4.94 J to 13.18 J with the increase of Si_3N_4 reinforcement ratio. It is thought that this situation occurs as a result of the uniform distribution of Si_3N_4 particles in the matrix and the strong interfacial bonding between the aluminum matrix and Si_3N_4.

3.4. Density and Porosity

For each of the unreinforced and reinforced materials, the theoretical density determined according to the mixtures rule, the experimental density obtained according to the Archimedes principle and the porosity ratios calculated using these density values are given in Table 5.

Table 5. Theoretical and experimental densities and porosity ratios of materials

Material	Theoretical density (g/cm^3)	Experimental density (g/cm^3)	Porosity (%)
AL	2,700	2,530	6,29
ALSN05	2,703	2,532	6,32
ALSN075	2,704	2,534	6,28
ALSN1	2,710	2,538	6,35

The change of the theoretical and experimental densities of Si_3N_4 reinforced composites according to the reinforcement ratio is given in Figure 4. In addition, the change of the porosity ratios with the reinforcement ratio is given in Figure 5. Since the density of Si_3N_4 is higher than the density of the matrix, it has been observed that the densities of the composite materials increase linearly with the increase of the reinforcement ratio by weight. In addition, it was determined that the porosity increased with the increase of Si_3N_4 reinforcement ratio.

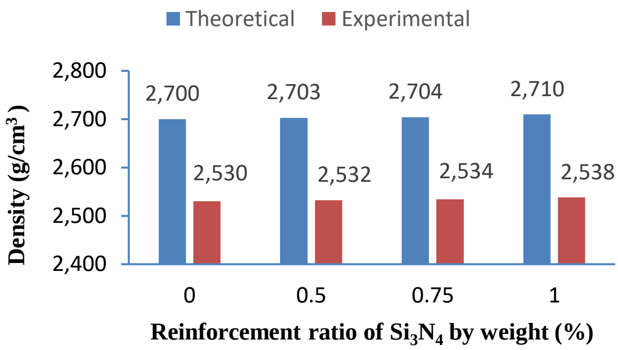


Figure 4. Change of theoretical and experimental density with Si_3N_4 percent weight ratio

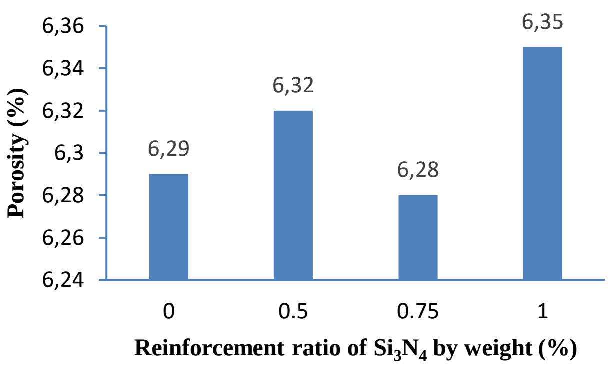
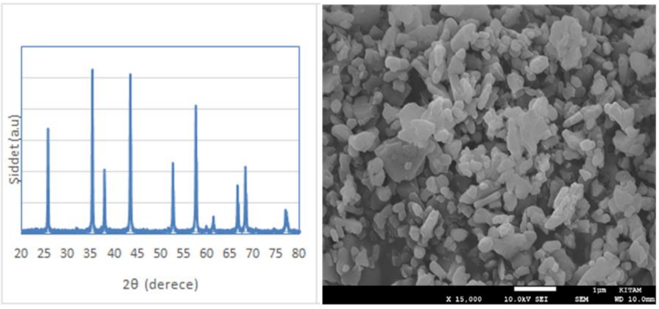


Figure 5. Change of porosity ratio with Si_3N_4 percent weight ratio

3.5. Microstructure

XRD and SEM analyzes of Si_3N_4 powders used in composite production are given in Figure 6. XRD analysis shows that the powders used are completely Si_3N_4. SEM analysis shows that the average particle size of Si_3N_4 powder is around 500-800 nm.



(a) (b)

Figure 6. XRD (a) and SEM (b) analyzes of Si_3N_4 powders

In Figure 7, XRD analysis of unreinforced Al matrix material and Si_3N_4 reinforced composites is given. The whole structure gave peaks belonging entirely to aluminum. The reason for the absence of Si_3N_4 peak in the structure is that the amount used is less than 5% by weight and the detection limit of the device is low.

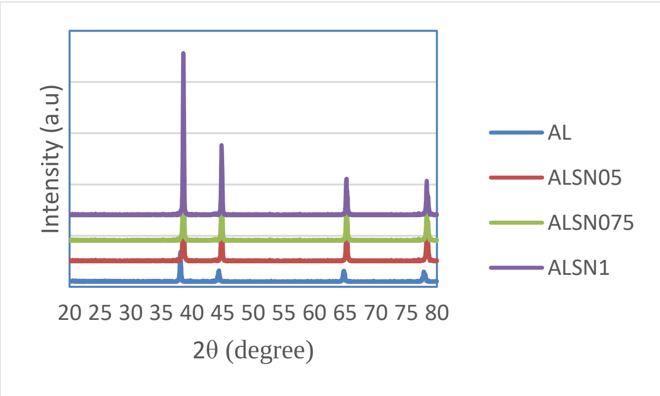


Figure 7. XRD analysis of unreinforced Al matrix and Si_3N_4 reinforced composites

In Figure 8, fractured surface SEM images of composites are given. From the images, it is seen that the grains form necks and have a low porous structure. As can be seen from the figure, the particle distribution increases with increasing Si_3N_4 amount. The homogeneous dispersion of the reinforcement material can be shown as the reason for the increase in the mechanical properties with the increasing amount of dust.

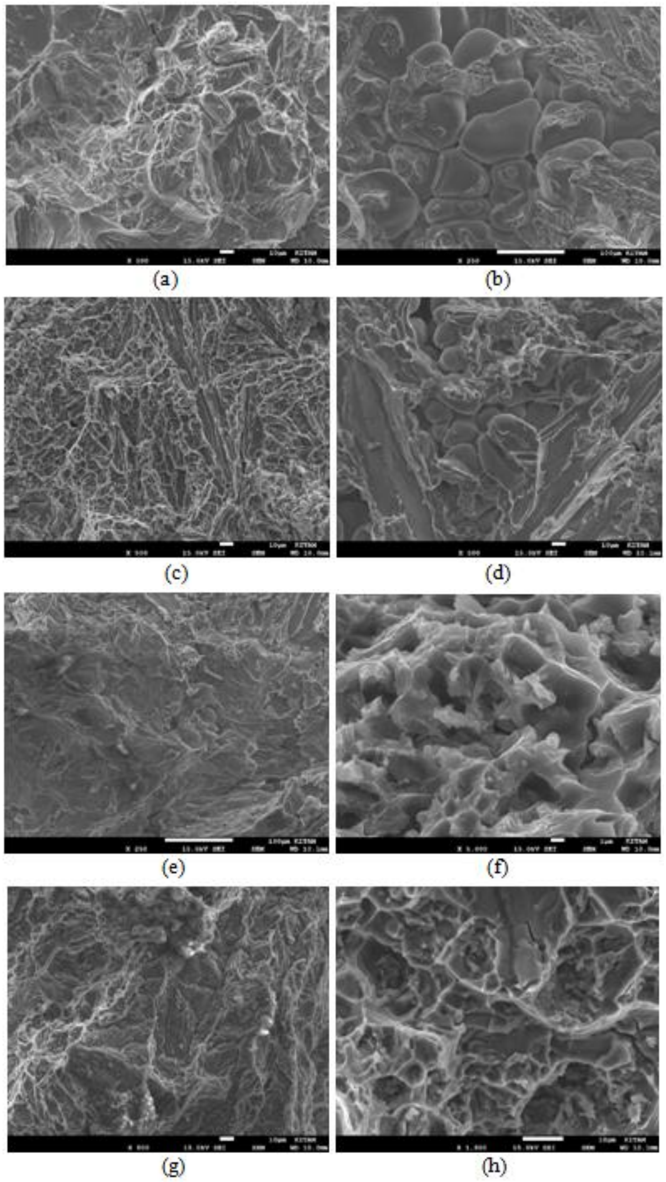


Figure 8. Fractured surface SEM images of unreinforced Al matrix and Si3N4 reinforced Al composites: (a-b) AL, (c-d) ALSN05, (e-f) ALSN075, (g-h) ALSN1

Elemental mapping results are given for the unreinforced Al matrix in Figure 9 and for ALSN1 with the best mechanical properties in Figure 10. As seen in the material containing unreinforced Al, there are Al, Si, Mg and Mn elements in the structure. On the other hand, in the mapping made for ALSN1, which shows the best mechanical properties, nitrogen is also added to these. In addition, the distribution in the places where Si and N are found is similar to each other, showing that the dust is homogeneously dispersed.

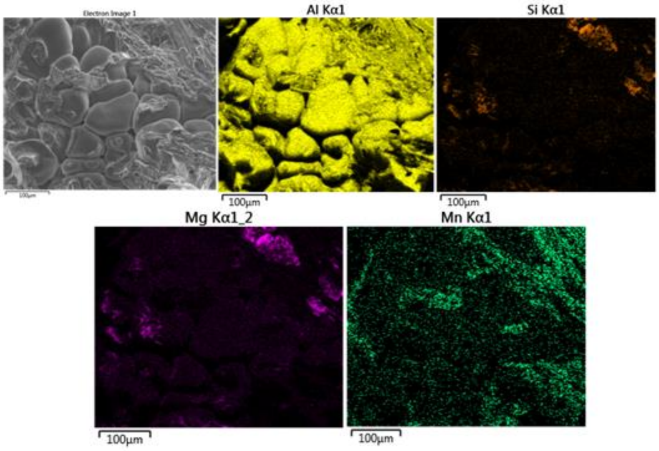


Figure 9. Elemental mapping results for unreinforced Al

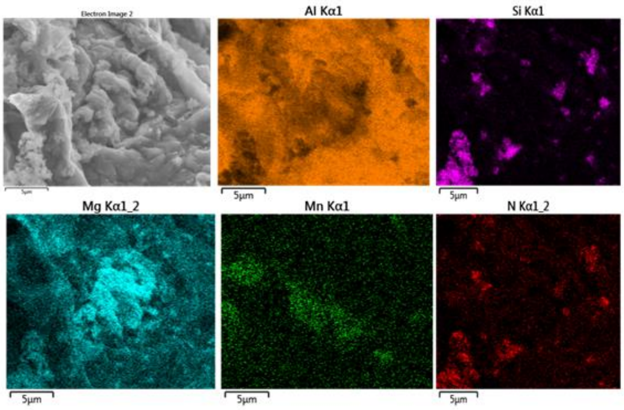


Figure 10. Elemental mapping results for ALSN1

4. Conclusions and Recommendations

In this study, the effects of Si_3N_4 reinforcement at different reinforcement ratios (0.5, 0.75, 1 wt%) on the density, hardness, tensile strength, impact strength and microstructure of composites produced from waste aluminum by stir casting method were investigated. The results of the experimental studies are summarized as follows.

1) It was determined that the hardness increased from 70 HV to 87.9 HV with increasing Si_3N_4 percentage weight ratio. The highest hardness was obtained at the rate of 1% reinforcement by weight.

2) It was observed that the tensile strength increased from 54.5 MPa to 105.17 MPa with the increase of Si_3N_4 weight percentage. The highest tensile strength was obtained at the rate of 1% reinforcement.

3) The impact strength increased from 4.94 J to 13.18 J with the increase of the Si_3N_4 percent weight ratio, and it was observed that the highest value was obtained at the 1% reinforcement rate.

4) It was determined that the density and porosity of the composite materials increased with the increase of the Si_3N_4 percent weight ratio. The density of the composite material at 1% reinforcement ratio was determined as 2,538 g/cm^3, and the porosity rate was determined as 6.35%.

5) From the fracture surface SEM images of Si_3N_4 reinforced composites, it was seen that the grains formed neck. In addition, it was determined that the particle distribution increased depending on the increasing amount of Si_3N_4 and the reinforcement material was distributed homogeneously.

5. Acknowledge

The authors acknowledge Black Sea Advanced Technology Research and Application Center (KITAM) in Ondokuz Mayıs University (OMU) for SEM and XRD analyses.

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[i] This study was derived from the master's thesis titled "Characterisation and Production of Graphene and Silicon Nitride Reinforced Composites with Using Waste Aluminum" completed at OMU Institute of Science.