

## EFFECT OF FULLERENE ON MECHANICAL PROPERTIES OF HOT-EXTRUDED ALUMINUM MATRIX COMPOSITE

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### ABSTRACT

Aluminum matrix composites have attractive attention for scientists and manufacturers. In this study, fullerene (C60) nanoparticle reinforced aluminum matrix composite was fabricated via semi powder metallurgy method. Microstructure analyzes were completed by X-ray diffraction (XRD) and Scanning Electron Microscope (SEM). Hardness, compression and tensile properties of unreinforced aluminum and C60 reinforced composite were evaluated. Results show that nanoparticle was homogeneously distributed in matrix and there were no cracks or macro defects. Hardness was significantly improved with the addition of fullerene and extrusion process. Al-C60 composite exhibited better compression and tensile performance than pure aluminum.

**Keywords:** Aluminum, fullerene, mechanical properties, SEM

## FULLERENİN SICAK EKSTRÜZYONLU ALÜMİNYUM MATRİSLİ KOMPOZİTİN MEKANİK ÖZELLİKLERİNE ETKİSİ

### ÖZ

Alüminyum matrisli kompozitler sanayiciler ve bilim insanları için dikkat çekici bir öneme sahiptir. Bu çalışmada fulleren (C60) takviyeli alüminyum matrisli kompozit malzeme, yarı toz metalürjisi yöntemiyle üretilmiştir. X Işını Kırınımı (XRD) ve Taramalı Elektron Mikroskopuyla (SEM) numunelerin mikro yapı analizleri gerçekleştirilmiştir. Saf alüminyum ve Al-C60 kompozitinin sertlik, basma ve çekme mukavemetleri hakkında değerlendirmeler yapılmıştır. Sonuçlardan yola çıkarak, fullerenin alüminyum matrisi içerisinde homojen dağıldığı gözlemlenmiştir ve herhangi bir çatlağa ya da makro hatalara rastlanmamıştır. Fullerenin ilavesiyle ve ekstrüzyon prosesiyle alüminyumun sertliğinde önemli bir artış görülmüştür. Al-C60 kompoziti saf alüminyuma kıyasla daha iyi basma ve çekme mukavemeti performansları sergilemiştir.

**Anahtar kelimeler:** Alüminyum, fulleren, mekanik özellikler, SEM

### 1. INTRODUCTION

Metal matrix composites (MMCs) have big potential in engineering applications especially automotive and aerospace industries [1]. High strength-to-weight ratio and high specific strength properties of materials have importance for scientists and industrialists. The harder reinforcement particles are generally incorporated in order to develop mechanical, thermal and corrosion performance of metals and their alloys. Among the metals, aluminum-based materials are gaining interest because of their low densities. However, they have poor mechanical performance such as strength and rigidity compared to copper and iron based alloys [2]. Researchers have been trying to develop strength, stiffness, thermal, corrosion and wear resistance of aluminum matrix composites using traditional ceramic and fiber particles. But they cannot meet the demands of the manufacturing industries sufficiently [3]. Nowadays carbon based nano-size materials have become popular and important

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reinforcements depending on the requirement. Multiwall carbon nanotube (MWCNT) and graphene nanoplatelets (GNPs) were chosen as reinforcement materials for aluminum matrix in some studies. Results showed that carbonaceous materials play important role to determine the mechanical performance of aluminum due to high surface area, bond structure and specific strength of MWCNT and GNPs [4,5,6]. The other allotrope of carbon atoms is known as fullerene which has spherical shape. C60 form (Bucky minister) of fullerene has 12 pentagons and 20 hexagons [7]. It can be used as excellent reinforcing agent for light metals. Our previous study indicates that fullerene has positive effect on compression and tribological performance of magnesium [8]. Choi et.al [9] fabricated aluminum based fullerene reinforced composite using ball milling and they reported that 2 vol. % fullerene had improved hardness performance. Beffort et.al [10] investigated the microstructure and interface characteristic of fullerene nanoparticle and aluminum matrix. However, the effect of C60 and extrusion on the mechanical performance of aluminum matrix composites has been rarely reported so far. In this work, the influence of C60 on hardness, compression and tensile strength of aluminum was investigated. Also, the other objective was to fabricate hot-extruded Al-C60 nanocomposite using semi powder method.

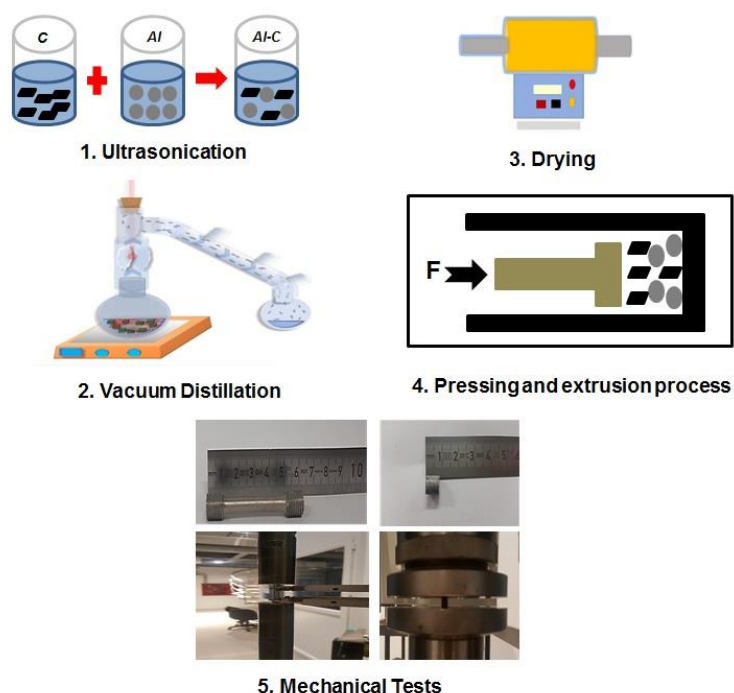
## 2. EXPERIMENTAL STUDIES

### 2.1. Fabrication

Aluminum powder (99.7% purity and 325 mesh size) was chosen as a matrix material. Fullerene (0.25 wt.%) was used as a reinforcement. Powders were purchased from Nanografi Co. Ltd. Turkey.

Semi-powder metallurgy technique was used for fabrication of samples. Schematic of fabrication process is shown in Figure 1. C60 was ultrasonicated in 60 ml. ethanol solution using an Alex Ultrasonic Machine for 2 hours. Then aluminum powder was poured into the solution stage by stage. Powders were mixed in vacuum distillation system by magnetic stirrer (600 rpm and 120 °C) until ethanol removed from the powders. Finally, dried powders were exposed in atmosphere controlled furnace at 90 °C overnight. Cold press was used to compact powders. The diameter of specimens with  $\phi 40 \times 25$  mm cylindrical. Pure Al and Al-C60 specimens were sintered under high purity argon atmosphere. Table 1 presents the compaction and sintering parameters.

The obtained samples were machined into cylindrical bars with a diameter of 32 mm and a length of 20 mm for the extrusion process. Hot extrusion was carried out at an initial billet temperature of 380 °C with an extrusion ratio of 16:1 and a ram speed of 0.3 mm/s.



**Figure 1.** Fabrication and experimental stages of Al-C60 composite

**Table 1.** Production parameter of specimens

Mold	Steel
Applied Pressure	400 MPa
Pressure Time	30 seconds
Sintering Temperature	580 °C
Sintering Time	1.5 hours

**2.2. Characterization**

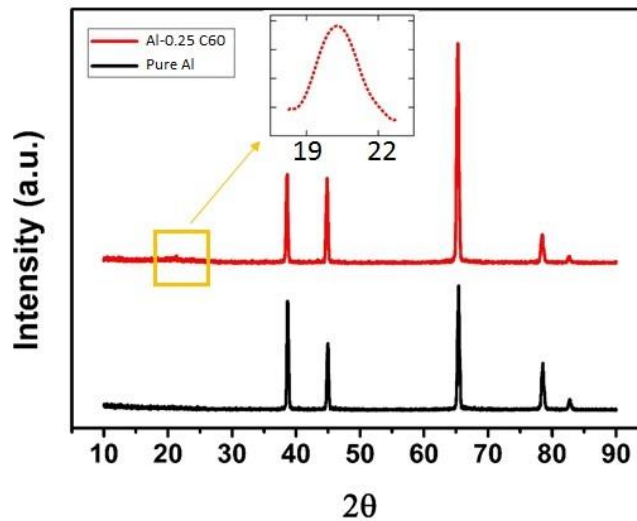
Rigaku X-ray diffraction test machine was used to characterize present phases in composites. Microstructure of specimens was investigated by Carl Zeiss Ultraplus SEM equipped with EDX (Energy Dispersive X-ray) after metallographic process (grinding, polishing and etching with picric acid) had been applied. SEM Image (1000X magnification) was taken with SE detector at 10kV and working distance was 5.2 mm. Vickers micro hardness values of the composites were calculated using five successive indentations by a QNESS Q10A+ hardness test machine under a load of 500 g. with dwelling time 15 seconds. EN 6507-1 standard was applied for hardness measurement. Compression tests were completed with 0.2 mm/min and tensile tests were performed with 0.017 mm/sec. Compression test specimens were prepared and test was conducted in accordance with TS 206 and ASTM E9 standards. Tensile test was performed using EN ISO 6892-1 standard. Macro extensometer was used to obtain stress-strain curves more precisely. Both of tensile and compression tests were performed parallel to the extrusion direction.

**3. RESULTS AND DISCUSSION**

**3.1. X-ray Diffraction**

Figure 2 illustrates the X-ray diffraction (XRD) patterns of polished extruded unreinforced Aluminum and Al-C60 composites. No intermetallic phases such as Al<sub>x</sub>C<sub>y</sub> could be detected according to the XRD results. Pure aluminum can be clearly seen at 2θ equal to 38.5°, 44.74° and 65.13°. When fullerene incorporated to the aluminum, no extra peak was observed. This might be attributed that nano-size and low amounts of carbon materials are found in composite [11].

The database of test machine provides the maximum peak (between 2θ equal to 19° and 22°) of pure C60 nanoparticle. So, fix time method (much slower measurement) was applied at defined angles. New peak was obtained at 2θ equal to 20.82° in <311> plane. As a result, the existence of C60 in composite was confirmed.



**Figure 2.** XRD patterns of produced samples

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Figure 3 demonstrates the pole figures {111}, {200} and {220} of pure Al and Al-C60 composite. Calculated I<sub>max</sub> values are presented in Table 2. Examination was carried out to the parallel directions to the extrusion. It can be see small amount of enhancement of the pole figure of {111} with the addition of C60. However, {200} pole intensities did not exhibit a remarkable change. When I<sub>max</sub> values of the pole figure of {220}, C60 led to increase of texture intensity. The change of texture symmetry between pure Al and C60 reinforced composite and enhancement of pole figures intensities can enhance the tensile properties.

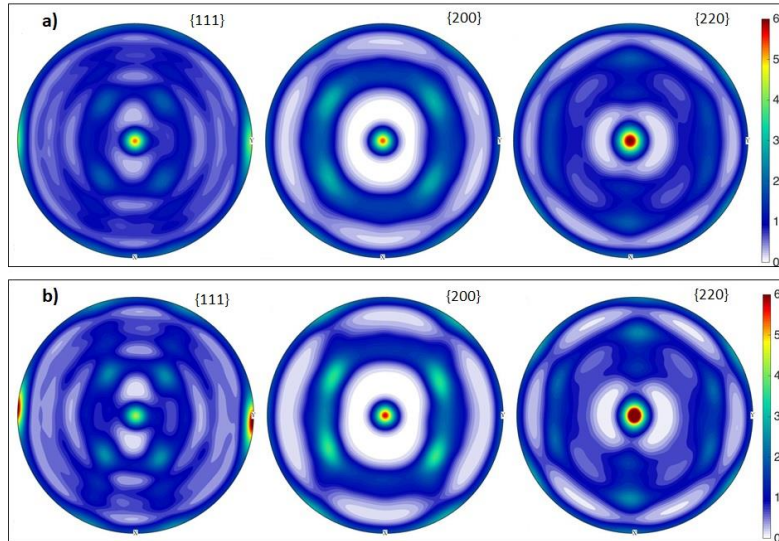


Figure 3. Pole figures of samples

Table 2. Crystallographic texture results of samples

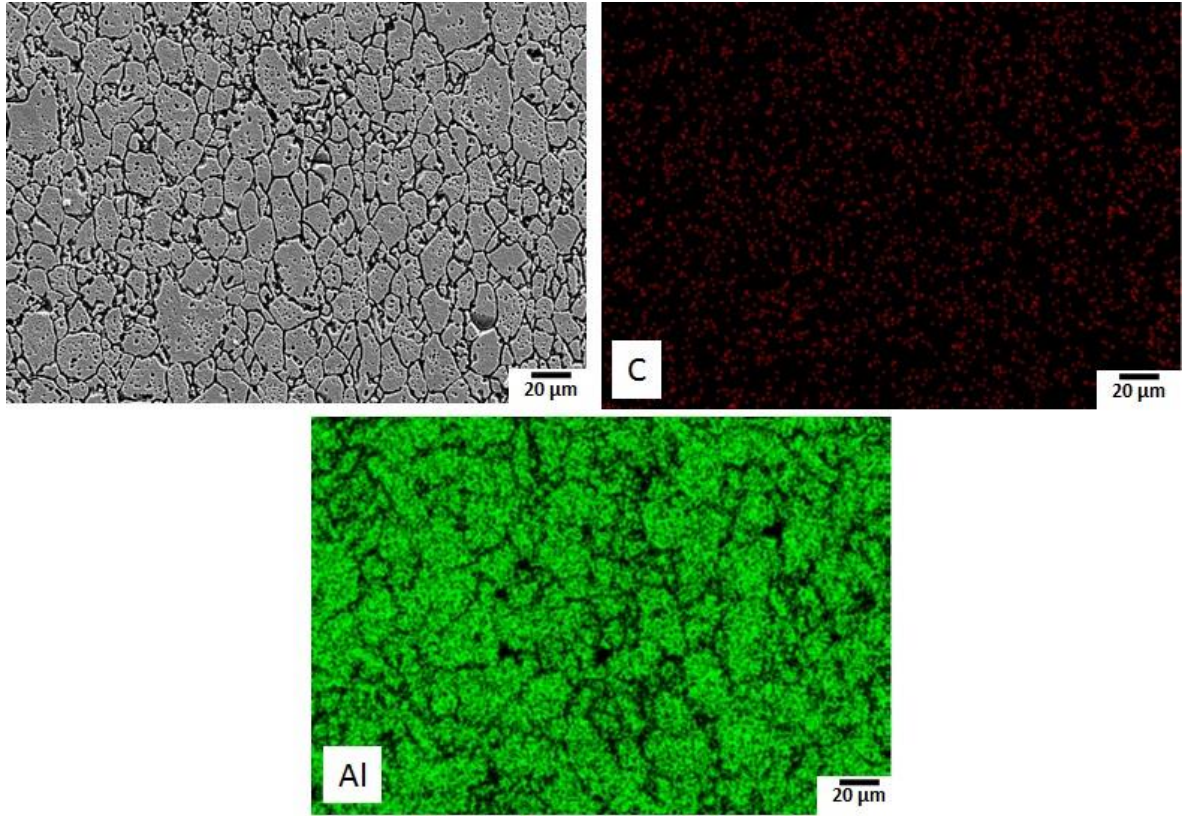
Materials	Pole Figures	I <sub>max</sub>
Pure Al	{111}	5.3
	{200}	5.5
	{220}	6.4
Al-0.25 C60	{111}	6.3
	{200}	6.8
	{220}	7.8

### 3.2. Microstructure Analysis

Figure 4 shows SEM microstructure of C60 reinforced aluminum matrix composites. Sample is lack of macro porosities and it has smooth surface. Grains are clearly seen and there might be some small negligible oxide formations. Mapping analysis was also used to examine distribution of carbon atoms on aluminum matrix. It can be observed that carbon atoms are embedded uniformly in microstructure.

The chemical reaction might be occurred between aluminum and carbon and this situation leads to the formation of carbide such as Al<sub>4</sub>C<sub>3</sub>. But, in the light of EDX and XRD analysis, no intermetallic phases can be seen in composite.

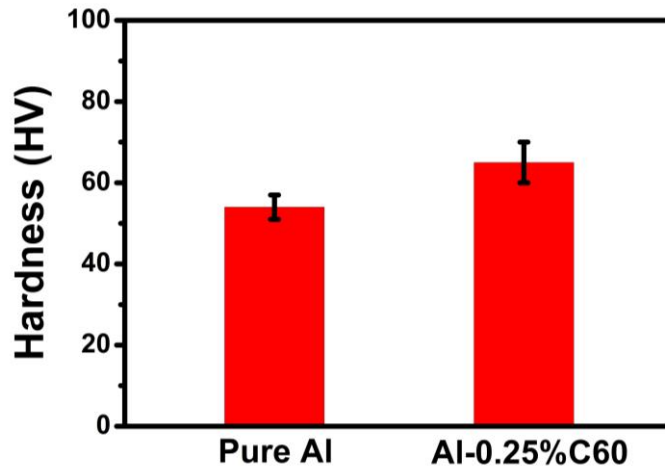
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**Figure 4.** SEM and mapping images of Al-C60 composite

**3.3. Mechanical Test Results**

Figure 5 illustrates the hardness performance of pure aluminum and composite material. C60 addition lead to the enhancement of hardness up to 17% of pure aluminum. It can be attributed to the presence of higher strength particle in matrix. The harder particle might cause the formation of localized matrix deformation. Also, C60 particles can affect the dislocation movement so hardness value was improved.



**Figure 5.** Hardness results of specimens

Table 3 exhibits the compression and tensile test results of unreinforced and Al-C60 composite. Figure 6a) shows the engineering stress and engineering strain curves for compressive properties of samples. A slightly

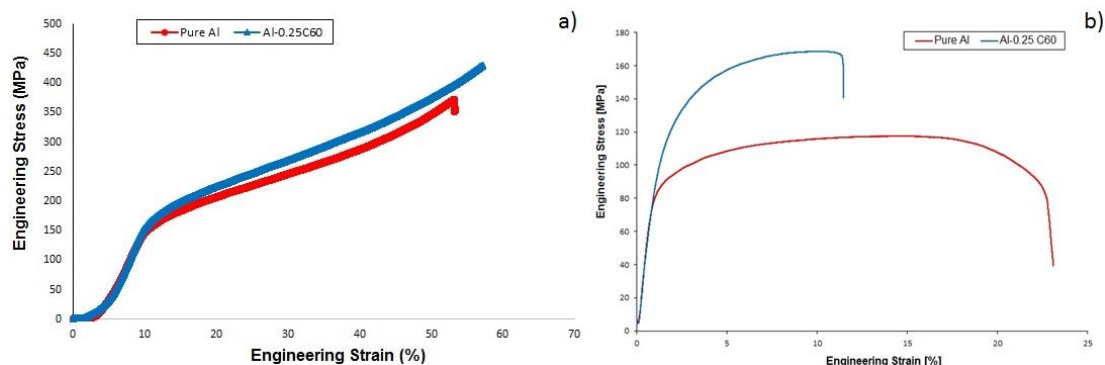
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increase of the ultimate compressive strength of aluminum with the addition of C60 can be seen from the data. This can be explained by uniform distribution and nano size reinforcements embedded in matrix. However, there is no considerable change of compression yield strength.

**Table 3.** Mechanical test results of samples

Materials	%0.2 CYS (MPa)	UCS (MPa)	0.2%YS (MPa)	UTS (MPa)	δ (%)
Pure Al	184	391	85	117	23
Al-0.25 C60	190	424	106	168	12

CYS: Compression Yield Strength  
 UCS: Ultimate Compression Strength  
 YS: Yield Strength  
 UTS: Ultimate Tensile Strength  
 δ: Fracture Strain



**Figure 6.** Mechanical test performance of samples (a) Compression (b) Tensile

Tensile test results are presented in Table 3 and Figure 6b). There is a significant enhancement of yield and tensile strength with the addition of C60. Orientations of carbon nanoparticles in Al matrix play key role to determine the strength of material. If the carbon atoms are aligned the tensile direction, positive effect on load transfer can be seen. Also, dislocations can occur at interfaces between matrix and reinforcement due to mismatch between the coefficient of thermal expansion (CTE) and the elastic modulus (E) [12], [13]. These mismatches might cause the formation of thermal residual stress, so strength of materials can be increased [14]. Rashad et. al [15] incorporated graphene nanoplatelets in pure aluminum and they observed 14.7% enhancement of yield strength. In this study, near percentage of fullerene was used and closer test speed was chosen. As a result 24.7% enhancement in yield strength of matrix was observed.

However, surface area of reinforcements effects the density of dislocations. High dislocation density can be generated by nano size particle and as a result, yield strength and tensile strength are improved with the restriction of dislocation movements. However, when C60 incorporated to the aluminum, fracture strain decreased significantly. This might be caused stacking of C60 particle as a result of van der Waals interactions between carbon atoms.

#### 4. CONCLUSIONS

C60 nanoparticle and Aluminum were mixed using semi powder metallurgy consists of ultrasonication and distillation. Pressing, sintering and hot extrusion process were applied successfully. In the light of the results, following findings were observed.

- Uniformly embedded C60 particles was seen according to the SEM mapping analysis.
- Considerably change of pole figure intensities could not observed.

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- Hardness was improved with the addition of nanoparticle.
- A small increase of ultimate compression strength could be achieved by C60
- There was no difference about compression yield strength between pure Al and composite.
- According to the tensile test results, yield and tensile strength was improved considerably.

**5. REFERENCES**

- [1] NAG, A., ŠČUČKA, J., HLAVACEK, P., KLICHOVÁ, D., SRIVASTAVA, A., HLOCH, S., DIXIT, A., FOLDYNA, J., ZELENAK, M., “Hybrid aluminum matrix composite AWJ turning using olivine and Barton garnet”, *International Journal Advanced Manufacturing Technology*, 94, 2293-2300, 2018.
- [2] MA, B., WANG, J., LEE, H., DORRIS, S., WEN, J., BALACHANDRAN, U., “Microstructural characterization of Al<sub>4</sub>C<sub>3</sub> in aluminum–graphite composite prepared by electron-beam melting”, *Journal of Material Science*, 53, 10173–10180, 2018.
- [3] GAO, X., YUE, H., GUO, E., ZHANG, H., LIN, X., YAO, L., WANG, B., “Preparation and tensile properties of homogeneously dispersed graphene reinforced aluminum matrix composites”, *Materials Design*, 94, 54–60, 2016.
- [4] GHASALI, E., SANGPOUR, P., JAM, A., RAJAEI, H., SHIRVANIMOGHADDAM, K., EBADZADEH, T., “Microwave and spark plasma sintering of carbon nanotube and graphene reinforced aluminum matrix composite”, *Archives of Civil Mechanical Engineering*, 18, 1042-1054, 2018.
- [5] PÉREZ-BUSTAMANTE, R., GÓMEZ-ESPARZA, C., GUEL, I., MIKI-YOSHIDA, M., LICEA-JIMÉNEZ, L., PÉREZ-GARCÍA, S., MARTÍNEZ-SÁNCHEZ, R., “Microstructural and mechanical characterization of Al–MWCNT composites produced by mechanical milling”, *Material Science and Engineering A*, 502, 159-163, 2009.
- [6] LI, J., ZHANG, X., GENG, L., “Improving graphene distribution and mechanical properties of GNP/Al composites by cold drawing”, *Materials Design*, 144, 159-168, 2018.
- [7] TAYLOR, R., WALTON, D., “The chemistry of fullerenes”, *Nature*, 363, 6431, 1993.
- [8] TURAN, M.E., SUN, Y., AKGUL, Y., “Mechanical, tribological and corrosion properties of fullerene reinforced magnesium matrix composites fabricated by semi powder metallurgy”, *Journal of Alloys and Compounds*, 740, 1149–1158, 2018.
- [9] CHOI, K., SHIN, S., BAE, D., CHOI, H., “Mechanical Properties of Aluminum-Based Nanocomposite Reinforced with Fullerenes”, *Transactions of Nonferrous Metals Society of China*, 24, 47-52, 2014.
- [10] KHALID, F., BEFFORT, O., KLOTZ, U., KELLER, B., GASSER P., VAUCHER, S., “Study of microstructure and interfaces in an aluminum–C60 composite material”, *Acta Materialia*, 51, 4575–4582, 2003.
- [11] RASHAD, M., PAN, F., YU, Z., ASİF, M., LİN, H., PAN, R., “Investigation on microstructural, mechanical and electrochemical properties of aluminum composites reinforced with graphene nanoplatelets”, *Progress in Natural Science and Material International*, 25, 460–470, 2015.
- [12] DENG, K., WANG, X., WU, Y., HU, X., WU, K., GAN, W.M., “Effect of particle size on microstructure and mechanical properties of SiCp/AZ91 magnesium matrix composite”, *Material Science and Engineering A*, 543, 158–163, 2012.
- [13] NGUYEN, Q.B., QUADER, I., SHARON NAI, L., SEETHARAMAN, S., WAI LEONG, W., ALMAJID, A., GUPTA, M., “Enhancing hardness, CTE and compressive response of powder metallurgy magnesium reinforced with metastable Al90Y10 powder particles”, *Powder Metallurgy*, 59, 209–215, 2016.
- [14] HABIBI, M.K., HAMOUDA, A.S., GUPTA, M., “Hybridizing boron carbide (B4C) particles with aluminum (Al) to enhance the mechanical response of magnesium based nano-composites”, *Journal of Alloys and Compounds*, 550, 83–93, 2013.
- [15] RASHAD, M., PAN, F., TANG, A., ASIF, M., “Effect of graphene nanoplatelets addition on mechanical properties of pure aluminum using a semi-powder method”, *Progress in Natural Science and Material International*, 2, 101–108, 2014.