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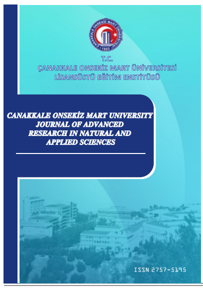


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# Shot Peening Effect on Reduced Graphene Oxide-based AA1070 Alloys Produced by Stir Casting Technique

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***Research Article***

**Abstract** − The shot peening effects of reduced graphene oxide (rGO) additive on the structural, residual stress, hardness and surface roughness of AA1070 composites were investigated and reported in detail for the first time. It can be said that with increasing amounts of rGO added into AA1070 alloy, rGO is randomly distributed into the Al matrix with increasing amounts. The XRD analysis shows that there is no trace of carbon in the Al matrix, showing the the carbon structure did not diffuse into Al matrix during casting. However, in the EDS analysis, it was deter-mined that the carbon value increased due to the increased presence of rGO. Although it has been studied ac-cording to the differences in ball diameters, it has been observed that the relative differences between the ball forging times affect the surface roughness changes in the samples. It was also observed that the hardness values were directly related to the ball forging times. On the other hand, the surface area of the forged matrix decreases in the presence of increasing rGO. While the hardness should increase after shot peening, cold deformation is not expected on the surface with the increase in the presence of rGO, and therefore, a decrease in the hardness value of the shot peened surface occurs. This is compatible with the residual stress results as well.

***Keywords*** − *AA1070, reduced graphene oxide, residual stress, stir casting, severe shot peening.*

## 1. Introduction

Graphite oxides as reinforcement material in different composite structures has become very popular in recent years (Naseer et al., 2019; Zhan et al., 2020a; Zheng et al. 2020b). Due to its high surface area and superior mechanical properties make the use of this structure for aluminium matrix composites much more interesting. It is also of great importance for applications in the automotive components where excessive motion is occurred, such as cylinder liners, crankshaft, tappets, pistons in order to increase the desired properties such as lightweight, surface hardness and mechanical strength in aluminium matrix composites (AMC) using a very small amount of reinforcement material (Naseer et al., 2019).

Due to the structural dissimilarity of graphene oxide in the aluminium matrix, there are difficulties in its homogeneous distribution in the matrix. For that reason, the studies on surface strengthening of graphene oxide added AMC have been carried out by using techniques such as stirred casting, liquid metal impregnation, sprayed deposition casting and so on (Toptan et al., 2010; Fadavi Boostani et al., 2018; Wu et al., 2019; Zheng et al., 2020a; Pehlivanlı & Pul, 2021). However, no study has been found on the graphene oxide-added AMCs with stirred casting so far using surface hardening processes like shot peening (Sun et al., 2018; Zhu et al., 2019; Rozhbiany & Jalal, 2019; Sasikumar et al., 2020)

With the shot peening process, the surface of the graphene oxide structured AMC will be hardened by a massive amount of high hardness and high-speed kinetic energy projectiles (Zhu et al., 2016; Zhan et al., 2018a; Zhan et al., 2018b). After shot peening, residual stress field and microstructure enhance its fatigue, surface hardness, resistance to stress corrosion cracks or high temperature oxidation resistance. However,

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residual stress distribution, microstructure and surface roughness will also differ depending on shot peening properties such as diameter of balls, Almen intensity, time and projectile material (Sasikumar et al., 2020; Avcu et al., 2021). In this study, the effect of shot peening with increasing rGO (0.25%, 0.5% and 1% wt.) on AA1070 composites were empirically investigated by scanning electron microscopy, electron diffraction scattering, x-ray diffraction, microhardness, residual stress and surface roughness.

## 2. Materials and Methods

### 2.1 Material

AA1070 was purchased from ISM Foreign Trade, Turkey and its chemical composition of the AA1070 alloy is presented in Table 1. The main reason for choosing the specified alloy is to examine the interaction of aluminum with the highest possible purity against rGO. In the synthesis of rGO, graphite (<20 μm powder), sulfuric acid ($H_2SO_4$.98%), sodium nitrate ($NaNO_3$), hydrogen peroxide ($H_2O_2$,30%) and potassium permanganate ($KMnO_4$, 98%) were obtained from Sigma Aldrich.

Table 1

Chemical composition of AA1070 alloy, % weight

| Element | Al | Cu | Fe | Mg | Mn | Si | Ti | V | Zn |
|---|---|---|---|---|---|---|---|---|---|
| % wt | rmn | 0.04 | 0.25 | 0.03 | 0.03 | 0.2 | 0.03 | 0.05 | 0.04 |

### 2.2 Experimental Procedure

Reduced graphene oxide was produced by the modified Hummers method (Ciğeroğlu et al., 2020). To explain shortly, 3 g of natural graphite was mixed with sodium nitrate (1.2 g) for 30 minutes in a beaker placed in an ice bath at 1°C, and 150 mL of sulfuric acid was then added. Afterwards, potassium permanganate (12 g) was added to oxidize the graphite. After the resulting solution was removed from the ice bath, it was subjected to magnetic stirring at 35°C for 90 minutes. So, it was observed that a rapid increase in temperature occurred with the addition of ultrapure water. The temperature was kept below 100°C. After these procedures, the solution, which is stirred for another 30 minutes, is diluted with 300 mL of ultrapure water and the reaction is terminated by adding 20 mL of 30% hydrogen peroxide. Finally, the mixture is washed sequentially with HCl and $H_2O$, then centrifuged at 4000 rpm for 10 minutes to separate the solid and dried under vacuum at 80°C overnight to yield GO powder. After obtaining GO powder, it was subjected to ascorbic acid with volume ratio of 1:1 and then heated for 30 min at 90°C. Finally, the aqueous solution was re-centrifuged at 8000rpm, washed three times with ethanol and water, respectively, and dried at 120°C for 60 min. in the oven. As it is seen in Figure 1, the presence of rGO is located close to 26.8° at the reflection plane (002) which was previously reported in Soomro et al. (2019) study. This peak also distinct rGO from GO as well.

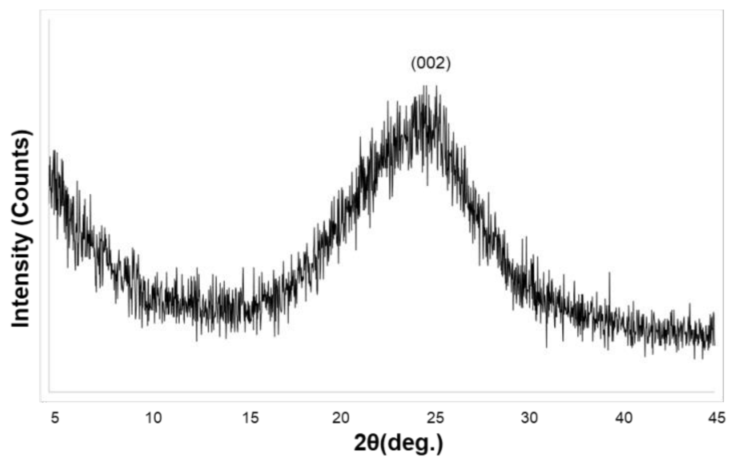



Figure 1. XRD analysis of synthesized rGO powder

Reduced graphene oxide with 0.25%, 0.5% and 1%wt. was separately added to the melted AA1070 alloy in a graphite crucible with a graphite probe, and after mixing at 4000 rpm for 5 minutes. Afterwards, a melted sample poured into the mold of a tubular shape of 10 mm in diameter and 10 cm in length and waited for cooling. Afterwards, each sample with a length of 10 mm was cut and made ready for structural and mechanical tests.

Severe shot peening process were applied with three Almen intensities of A10-12, A14-16 and A16-18 with other parameters remain constant. Severe shot peening treatment was conducted in accordance with MIL-13165 standard under the conditions shown in Table 2.

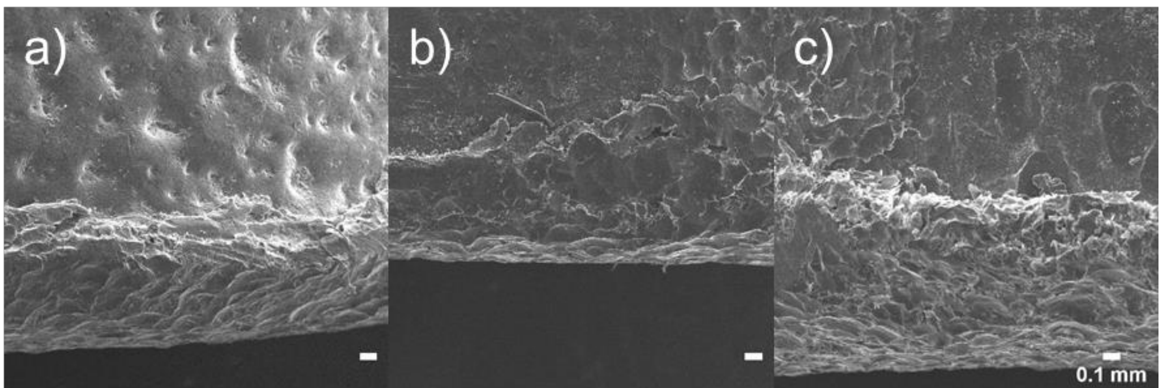
Table 2

Shot peening parameters

| Almen Intensity | Ball type and related hardness | Air pressure (bar) | Flow rate (kg/dk) | Saturation rate | Shot peening time (sn) |
|---|---|---|---|---|---|
| A10-12 | S110, 45-52 HRc | 4.5 | 5.0 | 100% | 35 |
| A14-16 | S170, 45-52 HRc | 5.5 | 5.5 | 100% | 31 |
| A16-18 | S230, 45-52 HRc | 5.5 | 5.0 | 100% | 42 |

**3. Results and Discusson**

The average shot peened layer under three different Almen intensity was illustrated in Figure 2. The shot peened layer is increased gradually from 0.74mm to 1.08mm. Almen intensity depends on the shot peening parameters. Ball or called projectile diameter, air pressure, saturation rate and shot peening time especially affect shot peening intensity. In this study, Almen intensity values were determined using A strips. As the Forging Intensity values increase, the shot peened layer thickness increases. As the Almen intensity increases, the kinetic energy value transferred to the forged material by the balls used in forging increases. Therefore, the forged or shot peened layer thickness increases.




| Average Shot Thickness (mm) of 0.5%wt. of rGO-AA1070 | | |
|---|---|---|
| 0.74 | 0.88 | 1.08 |

Figure 2. Average Shot Peened Microstructure of a)0.25%, b)0.5% and c) 1% wt. of rGO added AA1070 samples after shot peening

For selected 0.25%wt. of rGO added AA1070 samples, the surface roughness's with four different shot peening treatments were shown in Figure 3. The surface roughness of sample at unpeened condition was 2.8 and gradually is increased gradually from 7.5 to 9.2µm. Afterwards, the surface roughness suddenly decreases to 8.8 µm under A16-18 Almen intensity. At the A16-18 Almen intensity, the forging time was 42 seconds. By using S230 ball, the increase in this time causes the improvement of the surface quality. In the case of using S170 ball, since the applied air pressure value is the same, the impact speed was lower

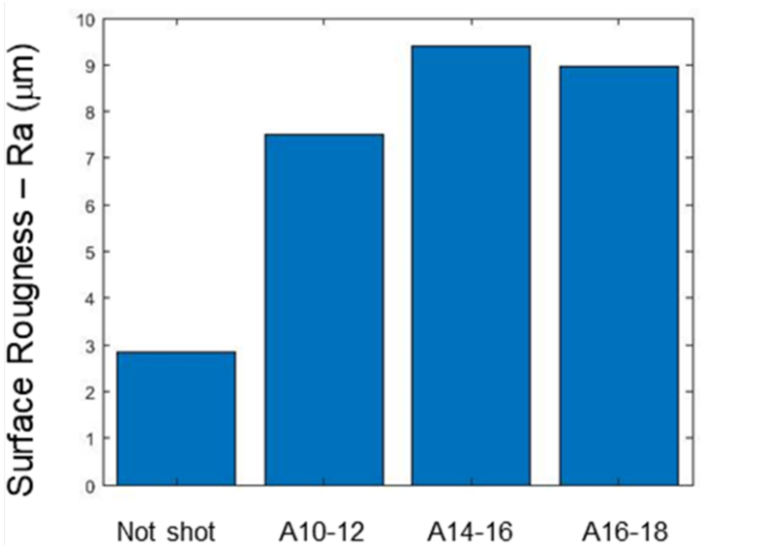



Figure 3. Average surface roughness of selected 0.25%wt. of rGO added AA1070 samples under different Almen intensity

because the mass of the S230 ball was higher. As a result, the surface quality was obtained better than the surface quality obtained under forging conditions using S170 ball diameter.

As seen in Figure 4., xrd analysis of pure and rGO added AA1070 matrix composite shows a single-phase structure of pure aluminium phase identification (JCPDS card no. 04-0787) which are present at 2θ equal to 38.47°, 44.74° and 65.13°, 78.23° and 82.43°. There is no trace of carbon in the Al matrix, so it is evidence that there is no second phase such as Al3C in the matrix. So, the rGO has not enough time to react with Al matrix during stir casting.

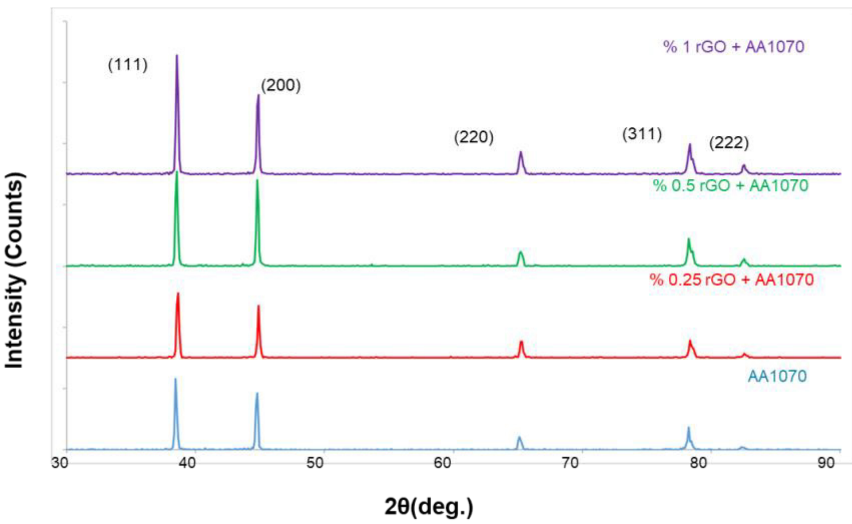



Figure 4. XRD analysis of pure and rGO added AA1070 composites

SEM images and SEM-EDS analysis of pure and rGO added AA1070 samples were shown in Figure 5. It is clearly seen that rGO particles were randomly distributed into Al matrix and the presence of rGO increases with increasing amount of rGO into the Al matrix. It is also seen at the EDS analysis that carbon is observed even at 0.25%wt. of rGO added AA1070 samples and gradually increases from 1.78 %wt to 8.88 %wt of carbon. Apart from AA1070 sample, rGO particles are clearly visible (marked as red circle) on the Al matrix which is compatible with the EDS analysis.

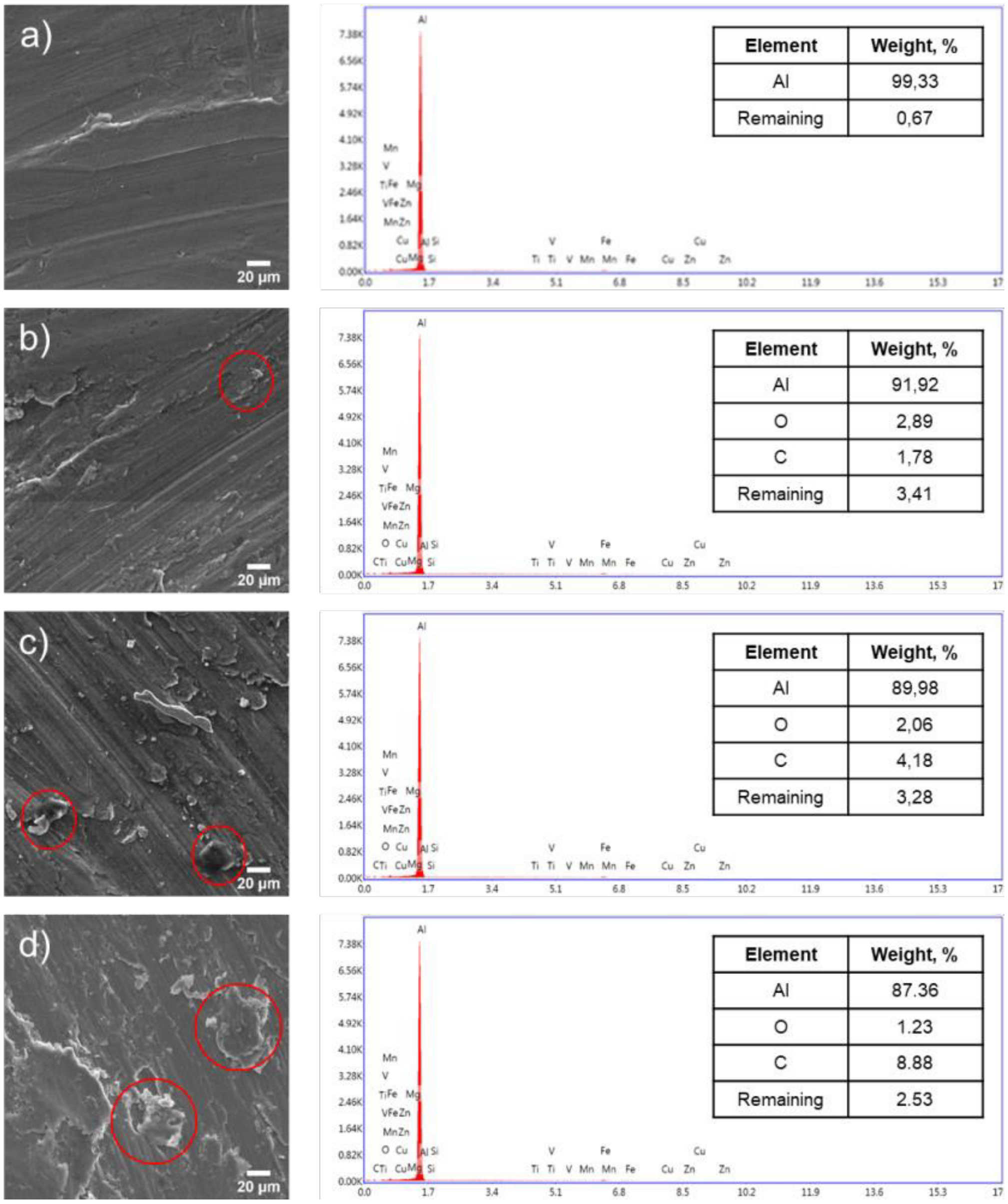



Figure 5. SEM and EDS analysis of pure and rGO added AA1070 samples

As it seen in Figure 6., the hardness values were decreased with increasing amount of rGO under various shot peening intensity. This observation can be concluded that even 0.25%wt. of rGO amount is still higher because it is not closer to pure AA1070 sample. It could be interpreted that rGO particles were agglomerated during the casting process and lower the hardness at the end. During the shot peening treatment, at least 99% of the surface is struck by the balls. However, as the amount of rGO added into the matrix material (AA1070) increases, the surface area of the forged material decreases. Essentially, it is the hardening that occurs during cold deformation of the surface of the shot peened material that reveals the increase in hardness. However, hardening of rGO should not be expected. Therefore, as the amount of rGO increases, the decrease in the hardness value of the shot peened surface is considered for this situation.

X-ray diffraction residual stress measurements was applied to all samples under different shot peening treatments as tabulated in Table 3. 0.25%wt. of rGO added AA1070 with increasing shot peening intensity decreases the residual stress which verify that surface area of rGO over 0.25% addition did not make any distinctive change against other samples. Residual stress results match exactly with the hardness values. This shows that the hardening effect is highly dependent on the residual stress as well.

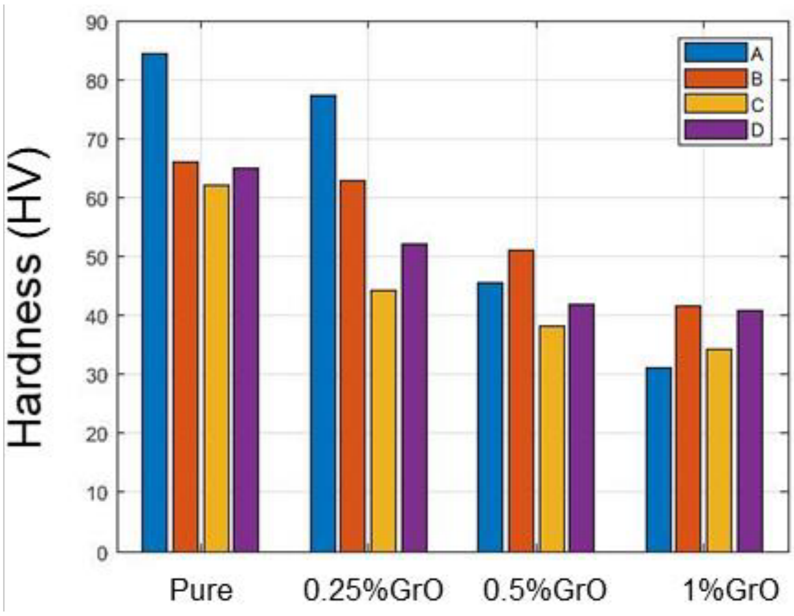



Figure 6. Microvickers hardness values of pure, 0.25%, 0.5% and 1% wt. of rGO added AA1070 samples under Almen intensity (A=not shot peened, B=A10-12, C=A14-16, D=A16-18)

Table 3

Residual stress of rGO added AA1070 samples under three different Almen intensity

| Residual Stress (MPa) | | | | |
|---|---|---|---|---|
| **Almen Intensity** | **Pure** | **0.25%wt. rGO** | **0.5%wt. rGO** | **1%wt. rGO** |
| A10-12 | -37.3±1.8 | -44.7±1.6 | -37.2±1.1 | -36.3±1.9 |
| A14-16 | -41.1±1.1 | -38.5±2.0 | -41.0±1.7 | -30.3±4.0 |
| A16-18 | -32.7±1.5 | -35.5±0.9 | -32.6±1.2 | -30.3±1.8 |

## 4. Conclusion

Pure and three different ratios (0.25, 0.5 and 1%wt.) of rGO added AA1070 composites were produced by stir casting and afterwards, severe shot peening applied for all samples. Under three different Almen intensity, the shot thickness and surface roughness increase up to A14-16 and then decreases. These phenomena are also observed at the hardness and residual stress values. Depending on the various Almen intensity and relative shot time differences, it can be interpreted that A14-16 Almen intensity is the optimum shot intensity value should be used for rGO added AA1070 samples. On the other hand, there is no trace of carbon reacted with Al matrix which is verified by xrd analysis. For SEM observation, rGO is randomly distributed into Al matrix and increases with increasing amount of rGO added into AA1070. Finally, residul stress of 0.25 %wt. of rGO added AA1070 was gradually decreased with incrasing Almen intensity while other samples show drastic change. This is a similar situation observed in the change of hardness and surface roughness due to the hardening phenomena observed in presence of rGO added into Al matrix.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**Author Contributions**

İremnur Bülbül: Provided the conception and design of the study, acquisition of data, analysis and interpretation of data, drafting of the article

Remzi Varol: Revised it critically for important intellectual content, and final approval of the version to be submitted

Mehmet Fahri Saraç: Provided the revised article critically for important intellectual content and gave final approval of the version to be submitted.

**Conflicts of Interest**

The authors declare no conflict of interest.

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