

## Effect of graphene nanoplatelets on the microstructure and mechanical behavior of erbium-modified Al-7.5Si-0.5Mg alloy

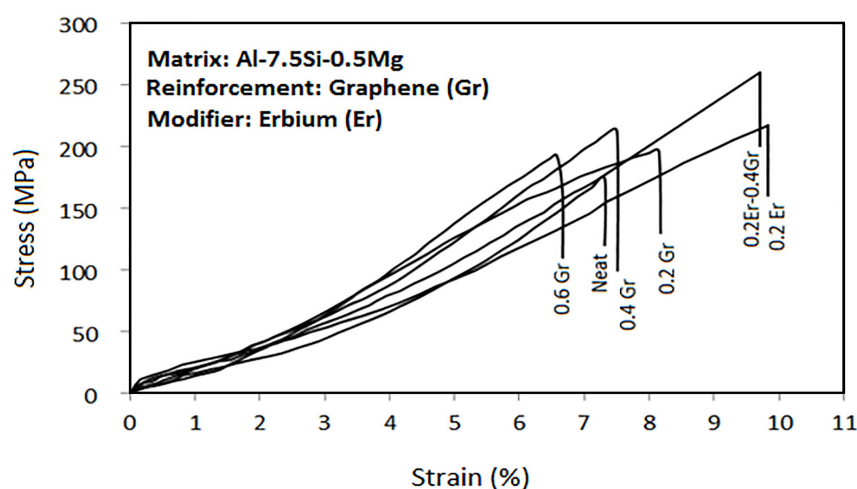
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### HIGHLIGHTS

- To study the effects of adding erbium and graphene (separately and in combination) on the mechanical properties of Al-7.5Si-0.5Mg alloy.
- The addition of erbium and graphene reduced the grain size of alpha-aluminum.
- The best tensile and wear properties were obtained for the sample containing 0.2 wt% erbium + 0.4 wt% graphene.

### GRAPHICAL ABSTRACT



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### ABSTRACT

In this study, the effects of adding erbium and graphene (separately and in combination) on the microstructure and mechanical properties of Al-7.5Si-0.5Mg alloy were investigated. Initially, the primary alloy was produced by casting in the form of ingots. To add graphene to the molten alloy, these particles were milled with aluminum powder for 3 h. Then 0.2, 0.4, and 0.6 wt% graphene, 0.2 wt% erbium, and finally, a combination of 0.2 wt% erbium with 0.4 wt% graphene were incorporated into the Al-7.5Si-0.5Mg alloy. The microstructure of the produced samples was examined using optical and electron microscopes, then the tensile and wear behaviors of the produced samples were examined. Finally, scanning electron microscopy (SEM) images were used to investigate the mechanisms of wear and failure. The results of this study showed that the addition of erbium and graphene modified the microstructure of the alloy and reduced the grain size of the alpha-aluminum. The best tensile and wear properties were obtained for the sample containing 0.2 wt% erbium + 0.4 wt% graphene. By adding this value to the Al-7.5Si-0.5Mg alloy, tensile strength and wear resistance were increased by 56% and 67%, respectively, compared to the control sample.

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## 1. Introduction

Metal-matrix composites are among the most important types of advanced materials, with a history of more than 50 years. These types of composites are used in many industries such as military, transportation, aircraft, and power transmission lines. Aluminum-matrix composites are a widely used type of metal-matrix composites. They have many applications due to their outstanding properties such as low density, high strength, excellent wear resistance, and good fatigue and corrosion resistance [1–3].

Al-Si alloys are one of the most widely used metallic materials in the automotive and aerospace industries due to their excellent castability, strength-to-weight ratio, and resistance to corrosive environments. The mechanical properties of Al-Si alloys are largely determined by the morphology of the eutectic Si. Coarse and needle-shaped Si particles usually act as the starting point for cracking, resulting in low ductility. As a result, modification of eutectic Si is an effective method to improve the mechanical properties of Al-Si alloys, especially for strength and ductility [4–6].

Graphene is considered a very desirable reinforcement for alloys due to its excellent mechanical, thermal, and electrical properties. Compared to other carbon materials, graphene has a sheet structure with a much higher surface ratio, which makes it an ideal reinforcement for composites [7–9].

Alipour *et al.* [10] prepared 7068 aluminum nano-composite reinforced by graphene nanosheets using a combination of ultrasonic and vortex casting methods, and they showed that the tensile and wear properties increased significantly by adding 0.5 wt% graphene. Yang *et al.* [11] showed that adding 0.54 wt% graphene increased the yield and tensile strengths of the aluminum sample by 116 and 45%, respectively. Lee *et al.* [12] showed that adding 0.25 wt% graphene to the aluminum matrix significantly increased the yield and tensile strength, although the ductility showed a slight decrease. Wang *et al.* [13] showed that the tensile strength of an aluminum matrix can be significantly improved (62%) by adding 5 wt% graphene. Meanwhile, Shin *et al.* [14,15] showed that the higher reinforcing efficiency of multi-layer graphene as compared to multi-layer carbon nanotubes can be mainly attributed to their higher specific surface area. In addition, Van *et al.* [16] obtained a 13% and 300% increase in tensile

and flexural strength, respectively, after adding 1 vol% graphene to an aluminum matrix. Lee *et al.* [17] used graphene oxide and carbon nanotubes in aluminum-based hybrid composites, which showed higher strength and reinforcing efficiency. Using graphene in an aluminum matrix, Yolshina *et al.* [13] achieved a 50% increase in tensile strength. Meanwhile, Jim *et al.* [18] fabricated crystalline graphene nanosheets using an electric charging process and achieved a 50% increase in tensile strength of an aluminum-matrix composite. Khodabakhshi *et al.* [19] observed a 100% increase in yield strength for the Al-2.2Mg alloy in the presence of graphene.

According to the literature, no research has been conducted on the mechanical properties of erbium-modified aluminum/graphene nanocomposites, which is the main reason for choosing this research.

## 2. Experimental

In this work, high-purity raw materials such as Al (99.7% purity), Mg (99.7% purity), Si (99.9% purity) as well as graphene nanosheets were used. Al-30% Er master alloy was also used as a modifier. Some characteristics of graphene are presented in Table 1.

**Table 1.** Some specifications of the graphene.

| Purity | Thickness | Specific area                           |
|--------|-----------|-----------------------------------------|
| 99.5%  | 2–18 nm   | 150–200 m <sup>2</sup> .g <sup>-1</sup> |

To fabricate the Al-7.5Si-0.5Mg alloy, in the first step, pure Al was melted using an induction furnace at 800 °C. Pure Si was added to the melt and mixed with a graphite stirrer to dissolve evenly. It should be noted, Si has a high melting point (1414 °C) and only dissolves in the melt. After adding Si to the melt, the crucible was placed in the furnace to increase the temperature to 800 °C. In the next step, Mg was added to the melt at 750 °C, and the crucible was returned to the furnace to increase the temperature. Casting was performed when the melt temperature reached 750 °C, and the slag was removed. The prepared ingots were melted again. After re-melting, different percentages of graphene (0.2, 0.4, and 0.6 wt%) were added to the melt and stirred with a graphite stirrer for 10 min. Samples containing 0.2 wt% erbium and other samples containing 0.2 wt% erbium + 0.4 wt% graphene were also produced. The graphene added to the melt in the first step was milled with pure Al powder for 4 h. The balls were made from hard Cr

steel, and the powder/ball weight ratio was 1/10. Casting was performed at 730 °C in a preheated mold.

To study the microstructure, the samples were cut in the form of a cylinder with a diameter and height of 1 cm. After the standard metallographic stage, samples were etched in 0.5% HF solution for 15 s. An Olympus optical microscope and a scanning electron microscope equipped with X-ray energy diffraction spectroscopy were employed. The average grain size of the structure was calculated using Image J software. The prepared samples were tested by an Instron Universal Tensile Test with a speed of 1 mm.min<sup>-1</sup>, and the stress-strain curves were obtained. A wear test was performed using an ECMA-C20604RS pin-on-disk device. The samples were prepared in the form of pins with a diameter of 6 mm and a length of 15 mm and placed under a load of 50 N on a rotating disk at a speed of 0.02 m.s<sup>-1</sup>. After tensile and wear tests, the fracture and worn surfaces of the samples were examined using a KYKY EM3900M scanning electron microscope.

### 3. Results and discussion

The ball-milling method was used to add the graphene into the molten alloy because the effect of the shear forces due to the ball impacts causes the graphene particles to be evenly distributed among the Al powders. It should be noted that the mixture was milled for 4 h to obtain a composite powder having 25 wt% graphene and 75 wt% Al. Fig. 1 shows the relatively uniform distribution of graphene particles among the Al powders, which prevents the formation of graphene clusters when the powder is added to the melt.

The optical microstructures of the samples are shown in Fig. 2. The samples illustrated are: the control sample without erbium or graphene addition, the sample containing 0.2 wt% erbium, the samples containing 0.2, 0.4, and 0.6 wt% graphene, and a composite sample containing 0.2 wt% erbium + 0.4 wt% graphene. The observed microstructure for all samples is a dendritic structure and consists of primary  $\alpha$ -Al along with  $\alpha$ -Si eutectic. As can be seen, the addition of erbium or graphene does affect the microstructure and grain size (Fig. 3).

It seems that the main reason for grain refinement in erbium-modified samples is due to constitutional supercooling as a result of the segregation of soluble elements in front of the solid-liquid interface [16,21]. This is because Er has a higher atomic radius than Al, and its solubility in Al is very low. Another mechanism may be due to the formation of an Al<sub>3</sub>Er intermetallic compound. The Al<sub>3</sub>Er with an FCC crystal structure is considered as a heterogeneous nucleation site for  $\alpha$ -Al (Al/Al<sub>3</sub>Er lattice parameter mismatch is 4%). The presence of the Al<sub>3</sub>Er phase can be seen in Fig. 4.

On the other hand, the addition of graphene can also refine the microstructure of the alloy, and the best refinement is seen for the 0.4 wt% graphene-loaded sample. At 0.6 wt% graphene addition, the effect is not significant, probably due to improper distribution of particles within the matrix.

The main reason for the refined structure due to the graphene addition can be related to the fact that graphene can act as  $\alpha$ -Al nucleation sites during the solidification of the alloy. On the other hand, these plates pin the grain boundaries and prevent grain growth. Graphene also increases the thermal conductivity of the melt and

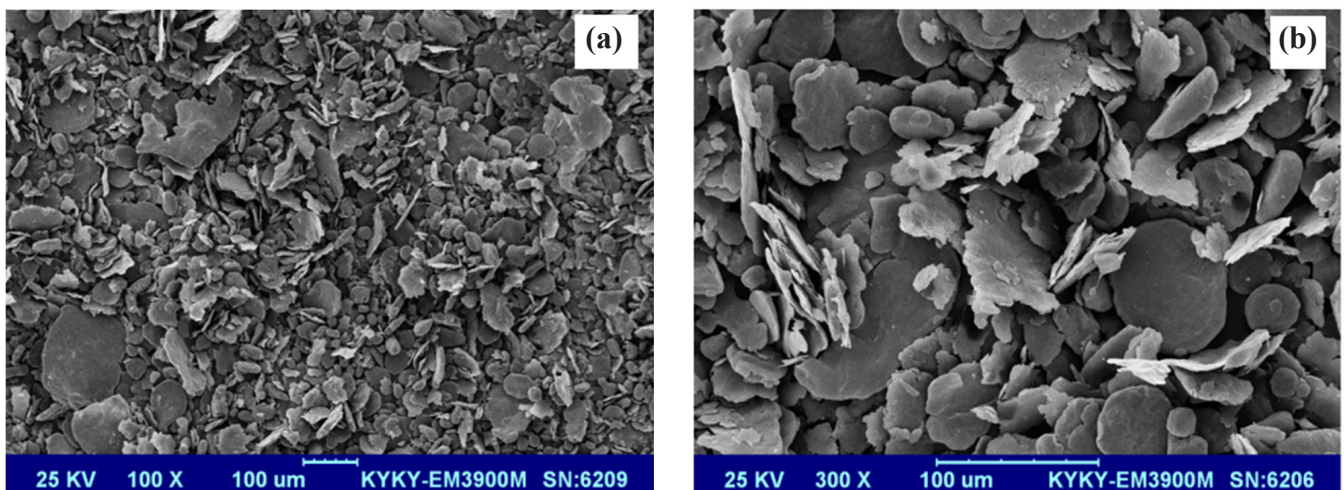
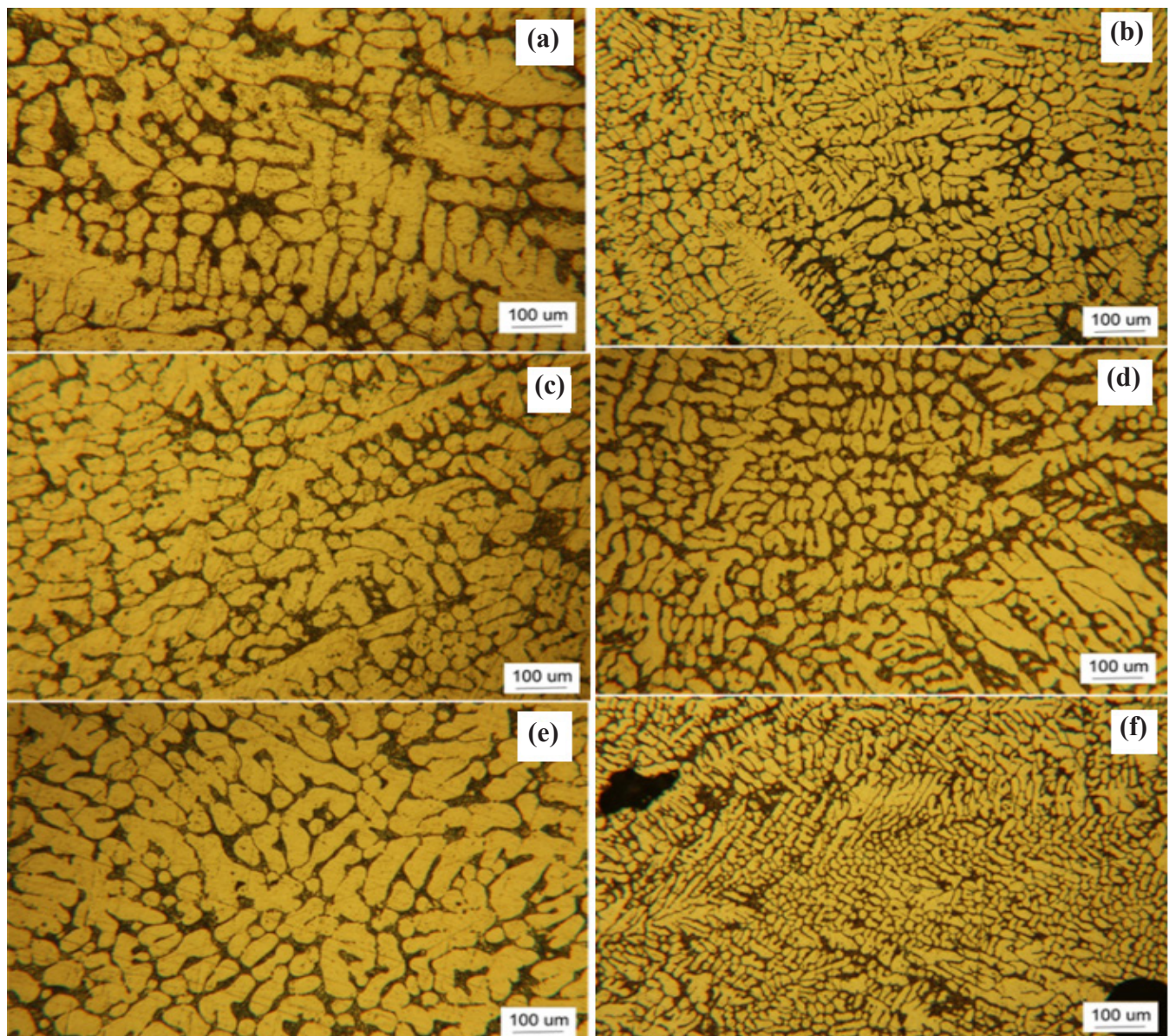


Fig. 1. SEM images from the ball-milled Al-graphene powder at (a) low magnification and (b) high magnification.



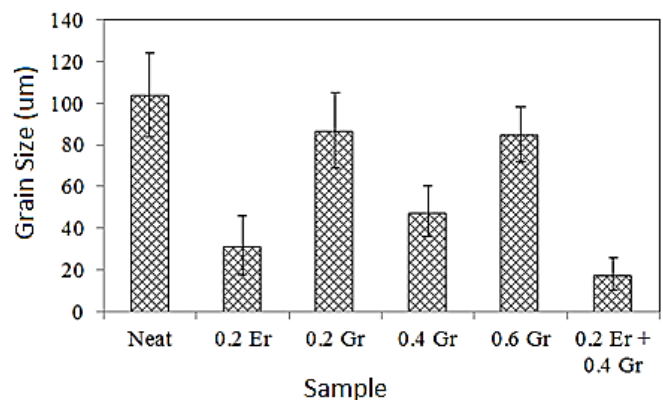
**Fig. 2.** Optical images from the microstructure of the (a) neat alloy, (b) 0.2 wt% Er-modified sample, (c) 0.4 wt% graphene reinforced sample, and (d) 0.2 wt% Er + 0.4 wt% graphene reinforced samples.

helps to cool the melt faster, and as a result, coarse  $\alpha$ -Al grains can be converted into fine coaxial grains. The results showed that when erbium and graphene were added simultaneously, the microstructure showed a significant refinement compared to the neat sample, which confirms that the simultaneous effect of the modifier and reinforcement on the microstructure is very remarkable.

The tensile behavior of the various samples produced in this study is shown in Fig. 5. As can be seen, the tensile behavior of the samples is similar, and all samples fail after reaching maximum tensile strength. In order to better compare the results, the tensile strength and ductility values were extracted from Fig. 5 and shown in Figs. 6 and 7.

The tensile strength of the neat sample is 170 MPa,

which is increased to 220 MPa (30% improvement) by adding 0.2 wt% Er. Among the graphene-reinforced samples, the highest strength can be seen for the 0.4



**Fig. 3.** The values of the average size of the  $\alpha$ -Al particles.

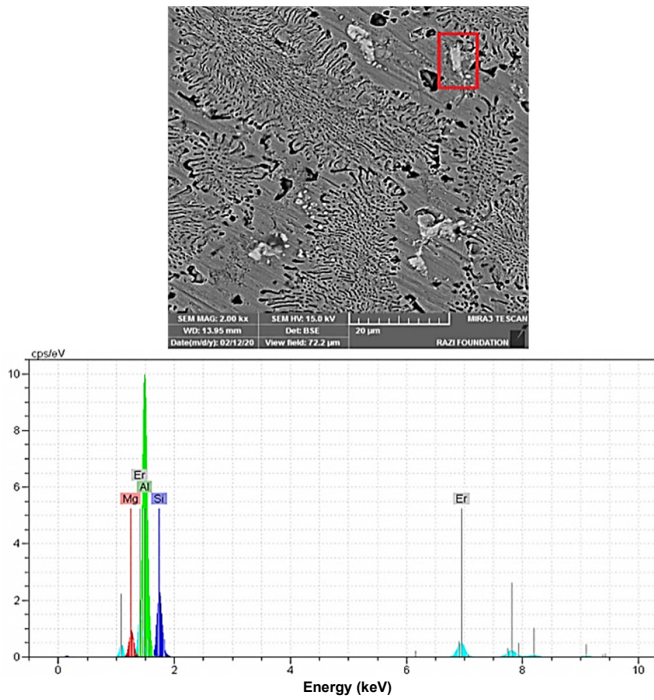


Fig. 4. Formation of the  $\text{Al}_3\text{Er}$  phase in the Er-modified sample.

wt% graphene addition, with a 26% improvement. When 0.2 wt% Er + 0.4 wt% graphene was added, the maximum tensile strength was obtained. In this case, the strength increases by 56% compared to the neat sample. Regarding the ductility trend, it is observed that the highest ductility is seen for the sample containing 0.2 wt% Er, and the addition of graphene has a very small effect on ductility. The main reason for the ductility improvement with the addition of erbium can be attributed to the change in Si morphology from a coarse-flaked shape to a fine-fibrous one, which has been discussed in our previous work [16].

The  $Q$  index is introduced (Eq. (1)) to better show the effect of adding graphene and erbium on tensile properties, which indicates the tensile properties based on both strength and ductility [23].

$$Q = \text{UTS} + 150 \log (\text{El}\%) \quad (1)$$

The values of the  $Q$  index for all samples are given in Table 2. As can be seen, the highest value of  $Q$  is related to the sample having 0.2 wt% Er + 0.4 wt% graphene.

The main reason for the improvement of the tensile properties due to the addition of erbium or graphene can be attributed to microstructure refinement (see Fig. 2). Graphene, on the other hand, acts as a barrier to the dislocation movement, thereby helping to improve the tensile properties.

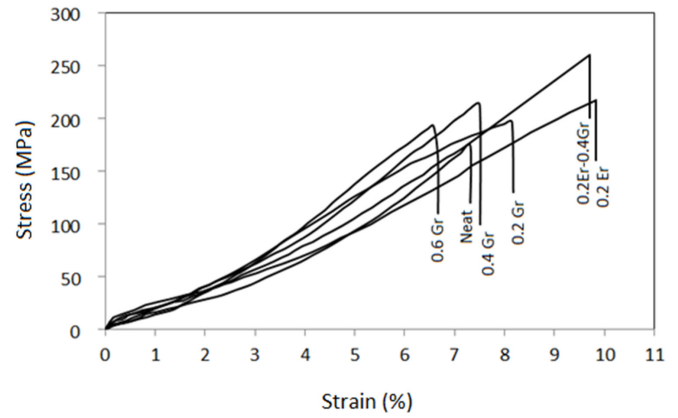


Fig. 5. The tensile behavior of the samples.

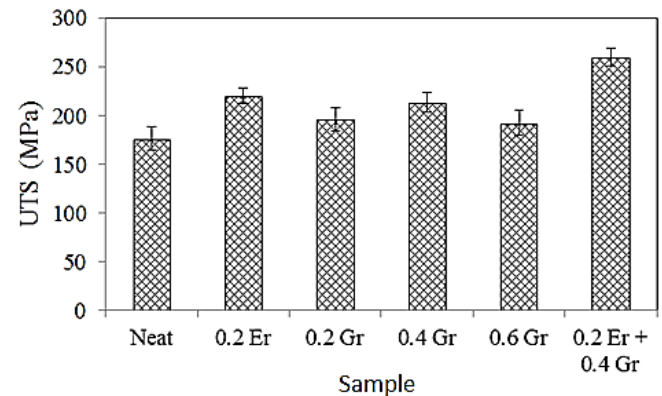


Fig. 6. The values of the tensile strength of the samples.

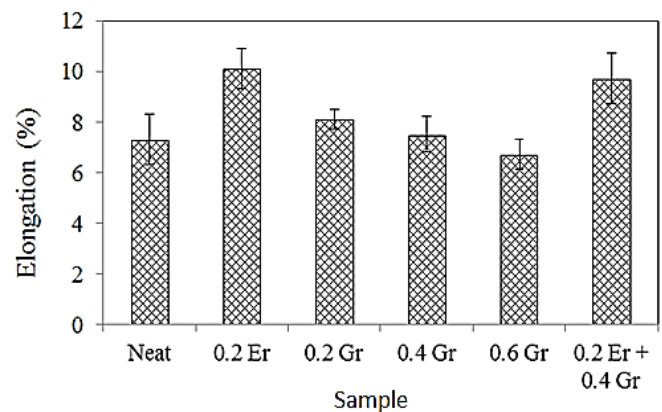


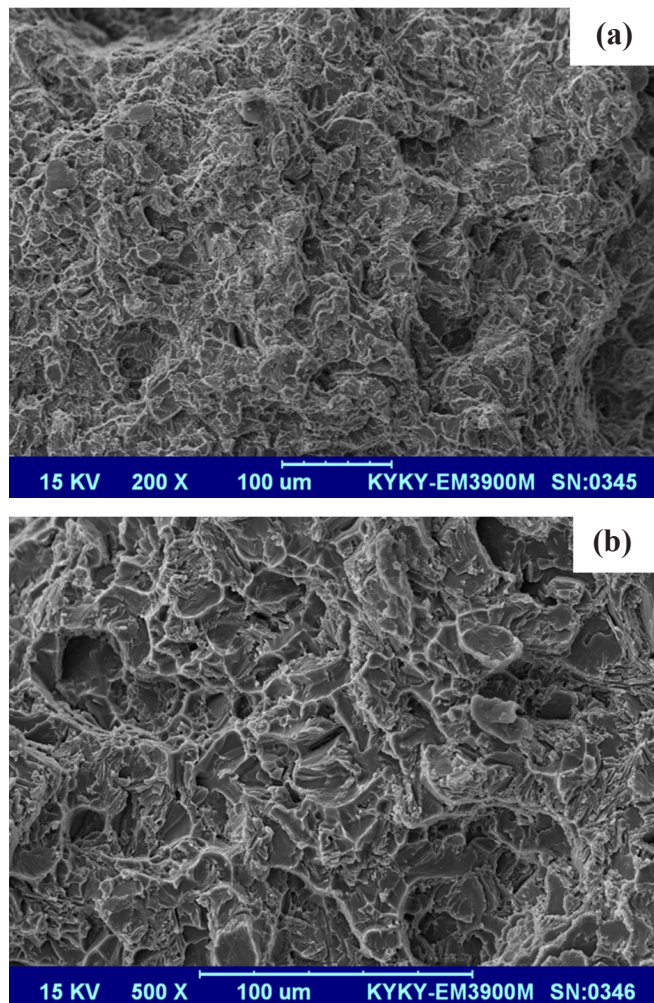
Fig. 7. The values of the elongation of the samples.

Table 2. The  $Q$  index values for the samples.

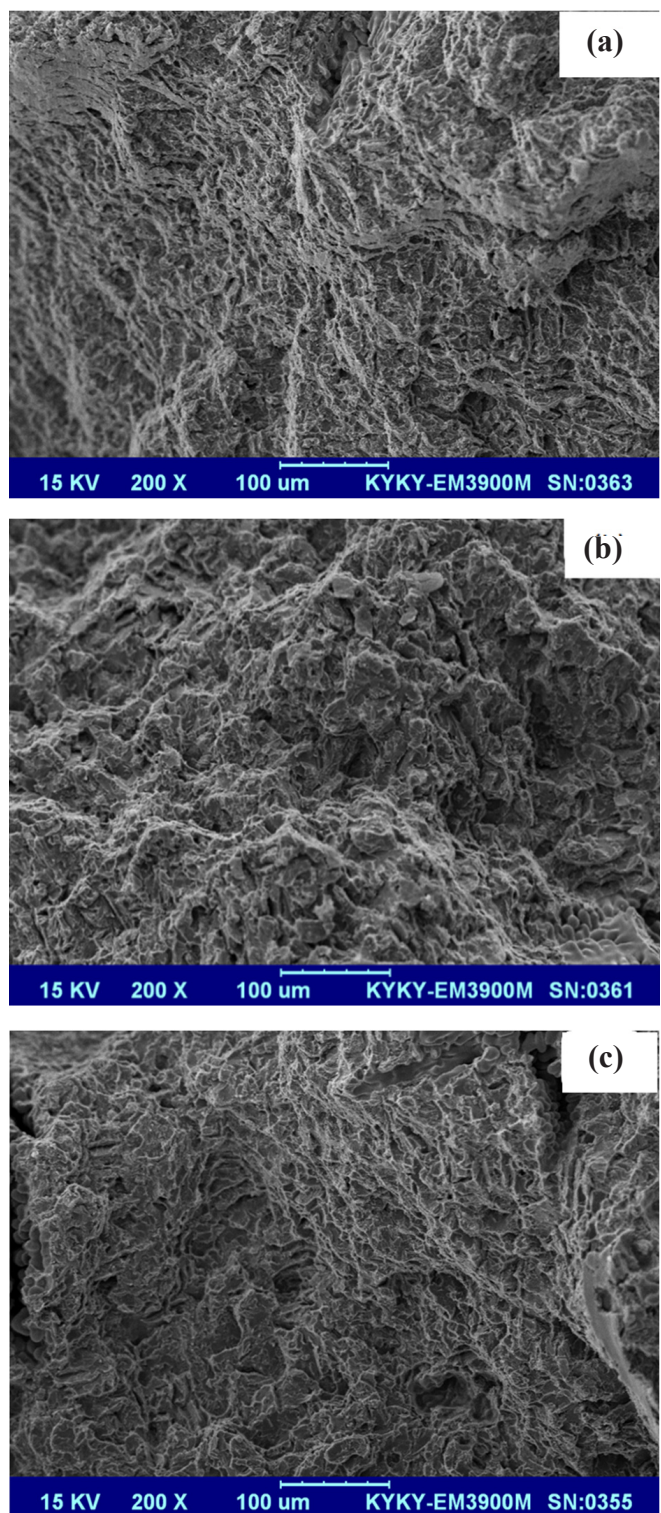
| Sample                        | $Q$ |
|-------------------------------|-----|
| Neat                          | 305 |
| 0.2 wt% Er                    | 370 |
| 0.2 wt% graphene              | 332 |
| 0.4 wt% graphene              | 344 |
| 0.6 wt% graphene              | 315 |
| 0.2 wt% Er + 0.4 wt% graphene | 408 |

Figs. 8 and 9 show the fracture surfaces of the samples. In Fig. 8, which shows the fracture surface of the neat sample, the cleavage surfaces (irregular surfaces) along with the tearing ridge pattern can be seen on the fracture surface. The size and area of the cleavage surfaces are reduced by adding 0.2 wt% erbium (Fig. 9a), and the tearing ridge pattern is visible in higher levels. Fig. 9b (fracture surface of the 0.4 wt% graphene-reinforced sample) shows a relatively brittle fracture pattern similar to that of the neat sample. Fig. 9c shows the fracture surface of the sample containing 0.2 wt% Er + 0.4 wt% graphene. It demonstrates a ductile failure pattern containing fine dimples. These images are in good agreement with the results obtained in Fig. 7.

Fig. 10 shows the weight loss of the samples exposed to the dry-sliding wear test. As can be seen, the structural modification of the base-alloy with erbium and graphene reduced the weight loss and improved its tribological performance. The lowest weight loss is observed for

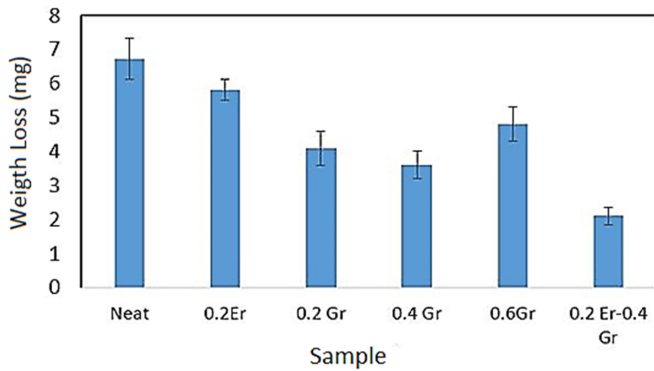


**Fig. 8.** The fracture surface of the neat sample at (a) low magnification, and (b) high magnification.



**Fig. 9.** The fracture surface of the (a) 0.2 wt% Er-modified sample, (b) 0.4 wt% graphene-reinforced sample, and (c) 0.2 wt% Er + 0.4 wt% graphene-reinforced sample.

the sample containing 0.2 wt% Er+0.4 wt% graphene, which shows the best wear performance between the samples. By adding 0.2 wt% graphene, weight loss is reduced by 14%, while adding 0.4 wt% graphene decreased the weight loss by 42%, compared to the neat sample. By adding 0.2 wt% Er+0.4 wt% graphene, the



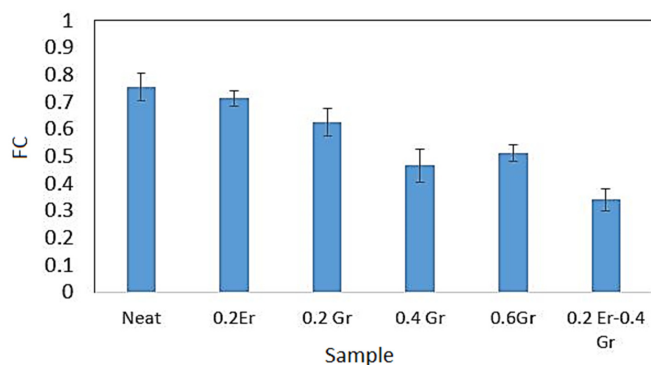
**Fig. 10.** The values of the weight loss of the samples under the wear test condition.

weight loss is decreased by 67% compared to the neat sample. This improvement in the wear performance can be attributed to the positive effect of erbium and graphene on  $\alpha$ -Al refinement as well as the modification of eutectic silicon.

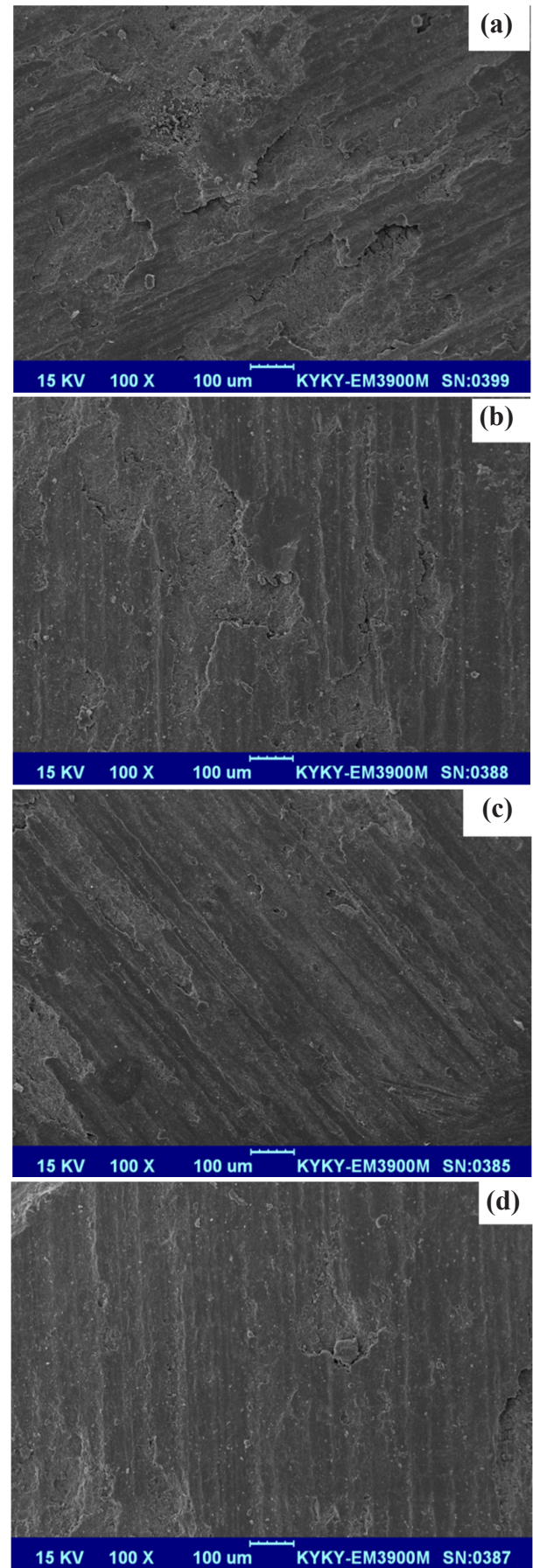
Fig. 11 shows the friction coefficient of the samples. As can be seen, the friction coefficient of the neat sample is 0.76, which is reduced by adding erbium and graphene. The lowest value of the friction coefficient (0.35) is related to the sample containing 0.2 wt% Er + 0.4 wt% graphene.

The improvement in wear properties in Er-modified samples can be attributed to microstructure refinement and modification of the eutectic Si. For the graphene-reinforced samples, the lubricating nature of graphene has a significant role in improving the wear properties. In the sample having 0.2 wt% Er + 0.4 wt% graphene, the simultaneous effect of erbium and graphene significantly improved the wear properties compared to the neat sample.

Fig. 12 shows the worn surfaces of the samples. For the neat sample, a big damage along with deep grooves can be seen on the worn surface, which confirms the existence of adhesive and abrasive mechanisms [24,



**Fig. 11.** The values of the friction coefficient (FC).



**Fig. 12.** The worn surfaces of the (a) neat alloy, (b) 0.2 wt% Er-modified sample, (c) 0.4 wt% graphene-reinforced sample, and (d) 0.2 wt% Er + 0.4 wt% graphene-reinforced sample.

25]. As can be seen, adding 0.2 wt% erbium or 0.4 wt% graphene reduces the surface ruggedness, and grooves with less depth are observed. In the sample containing 0.2 wt% Er + 0.4 wt% graphene, the surface is relatively smooth, and there is less-depth and finer grooves.

#### 4. Conclusions

In this study, the effect of adding erbium and graphene (separately and in combination) on the microstructure and mechanical properties of Al-7.5Si-0.5Mg alloy was investigated. The most important results of this work can be summarized in the following items:

1) The average grain size of the base alloy was significantly reduced via the erbium or graphene addition. The finest grains were observed in the sample containing 0.2 wt% Er + 0.4 wt% graphene.

2) Among the samples having only graphene, the highest tensile strength was observed at 0.4 wt% graphene (26% improvement over the neat sample).

3) The best tensile and wear properties were obtained for the sample in which erbium and graphene were added simultaneously.

4) When 0.2 wt% Er + 0.4 wt% graphene was added, the tensile strength increased by 56% compared to the neat sample.

5) The fracture surface of the sample containing 0.2 wt% Er + 0.4 wt% graphene demonstrated a ductile fracture pattern having fine dimples.

6) By adding 0.2 wt% Er + 0.4 wt% graphene, the wear resistance of the sample increased by 67% compared to the neat sample.

7) In general, the results of this study confirmed that the simultaneous addition of graphene and erbium had a significant effect on the microstructure and mechanical properties of aluminum cast alloys.

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