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Synthesis of Nickel nanoparticles on graphene oxide as a promising reinforcement for epoxy composites

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Abstract

Herein, Nickel-decorated graphene oxide nanoplatelets (Ni@GNs) were introduced as a promising reinforcement for epoxy composites. The morphology and structure of Ni@GNs were characterized by X-ray diffraction, atomic force microscope, and scanning electron microscope equipped with an energy-dispersive X-ray analyzer. This study sought to investigate the effects of Ni@GNs addition at different weight percentages (0.05, 0.1, 0.3, 0.5, and 0.7 wt%) on the tensile and dry-sliding wear properties of epoxy composites. Maximum enhancements in tensile strength (ie, 36%) and failure strain (ie, 56%) were observed for the specimen filled with 0.1 wt% Ni@GNs. In addition, by the introduction of 0.7 wt% Ni@GNs, a maximum enhancement of tensile modulus (ie, 39%) was achieved. The results of the wear tests demonstrated that the wear rate and friction coefficient of the epoxy matrix were decreased by 86% and 75%, respectively, by the addition of 0.3 wt% Ni@GNs. It was also found that the Ni@GNs/epoxy nanocomposite showed significant improvements in tensile and wear properties in comparison to the specimen filled with GNs.

KEYWORDS

epoxy, nanocomposite, Ni-decorated graphene oxide, tensile properties, wear properties

1 | INTRODUCTION

Epoxy resins due to their high mechanical properties are widely used in composite matrices. Recently, various types of nanoparticles such as SiO₂,^[1] Al₂O₃,^[2] CaCO₃,^[3,4] carbon nanotubes (CNTs),^[5,6] carbon nanofibers (CNFs),^[7] nanoclay,^[8,9] ZrO₂,^[10] TiO₂,^[11] and graphene^[12,13] have been investigated as fillers in an epoxy matrix. Among the nanoparticles, graphene nanoplatelets (GNs) have emerged as suitable candidates for reinforcement of epoxy due to its high surface area and exceptional mechanical properties. The mechanical behavior of GNs/epoxy composites has been investigated by several authors. Amirbeygi et al^[14] studied the effects of functionalized-GNs on the mechanical properties of epoxy nanocomposites. They found that the introduction of GNs increased the tensile strength and failure strain

at 0.05 wt%. Wang et al^[15] found that the incorporation of 1 wt% GNs enhanced the tensile strength of epoxy by 45%. Yao et al^[16] observed a remarkable enhancement in tensile strength and elongation for the 0.8 wt% GNs/epoxy nanocomposites. Ahmadi-Moghadam et al^[17] reported that the elastic modulus and fracture toughness of the 0.5 wt% GNs-loaded nanocomposites were improved 15% and 82%, respectively, as compared with the host epoxy matrix. In the work of Wang et al^[18] higher values of modulus and strength were achieved by increasing the loading of small GNs sizes (<1 μm) into the epoxy matrix.

In nanocomposites, the interfacial strength between the matrix and nanofillers plays a vital role in enhancing the mechanical properties.^[8,12] In order to enhance the matrix-GNs interfacial bonding and to improve the dispersion of GNs within the matrix, various approaches such as in-situ

polymerization,^[19] organic functionalization,^[20,21] and nanoparticle-decoration^[22] have been employed. Especially, several researchers have worked on the decoration of GNs with nanoparticles to enhance their distance in a matrix for preventing the formation of agglomerations. For example, Hou et al^[22] found that the decoration of nanodiamond (ND) onto the GNs prevented the aggregation of GNs in the epoxy matrix. In this work, the highest mechanical properties were obtained when GNs: ND (1:5 weight ratio) was used. Haeri et al^[23] investigated the thermal behavior of epoxy nanocomposites reinforced by SiO₂-GNs nanohybrid and noticeable enhancements in thermal resistance of SiO₂-GNs/epoxy composite were observed as compared to the GNs/epoxy specimen. Yu et al^[24] modified GNs by TiO₂ nanoparticles to improve the anticorrosion properties of epoxy coatings. Their results revealed that the TiO₂-GNs nanohybrid was more effective in enhancing the corrosion resistance of epoxy as compared to the GNs. The effect of SiO₂-GNs on the mechanical behavior of epoxy composites was investigated by Jiang et al,^[25] and it was found that the enhancement in mechanical properties of SiO₂-GNs/epoxy composite was much higher than that of the GNs/epoxy one.

In literature, based on our best knowledge, it was found no research work on the effects of Nickel-decorated graphene oxide nanoplatelets (Ni@GNs) on the mechanical properties of epoxy nanocomposites. In this work, various weight percentages of Ni@GNs were incorporated in an epoxy matrix and the tensile and wear behaviors of the resultant specimens were investigated.

2 | EXPERIMENTAL

2.1 | Materials

The GNs (6-10 layers) were provided by US-Research Nanomaterials Inc. According to the specifications of the

manufacturer, the GNs were 10 to 50 μm in diameter and 3.4 to 7 nm in thickness. Figure 1 shows scanning electron microscope (SEM) images of the as-received GNs at two different magnifications.

A medium-viscosity epoxy resin “KER 828” based on diglycidyl ether bisphenol-A was supplied by Kumho P&B Chemicals, Inc. The epoxy group content and dynamic viscosity of this resin are 5260 to 5420 mmol/kg and 12 to 14 Pa s, respectively. Nickel (II) chloride hexahydrate (NiCl₂·6H₂O), hydrazine hydrate (NH₂NH₂·H₂O), and ethylene glycol (CH₂OH)₂ were purchased from Sigma-Aldrich Co.

2.2 | Synthesis of the Ni@GNs

GNs of 0.5 g and NiCl₂·6H₂O of 4 g were incorporated into 60 mL ethylene glycol and the mixture was stirred for 1 hour using a magnetic stirrer. After this stage, 20 mL hydrazine hydrate + 3 g NaOH was added^[26] and the obtained mixture was refluxed for 7 hours at 110°C. The modified powers were separated, washed with ethanol, and dried at 80°C for 24 hours.

2.3 | Fabrication of the nanocomposites

The nanocomposite specimens reinforced by 0.05, 0.1, 0.3, 0.5, and 0.7 wt% Ni@GNs were fabricated as described below: the epoxy resin was mixed with the defined amounts of Ni@GNs using an overhead mechanical stirrer for 15 minutes. Then, the obtained mixture was sonicated for 30 minutes using a Ti-6Al-4V probe at 150 W (Ultrasonic Homogenizer, TOPSONICS Co., Iran). Next, as recommended by the manufacturer, the hardener was mixed with the mixture in a ratio of 10:100 (hardener:epoxy). Finally, the mixtures were poured into the pre-prepared silicone molds and cured for 24 hours at

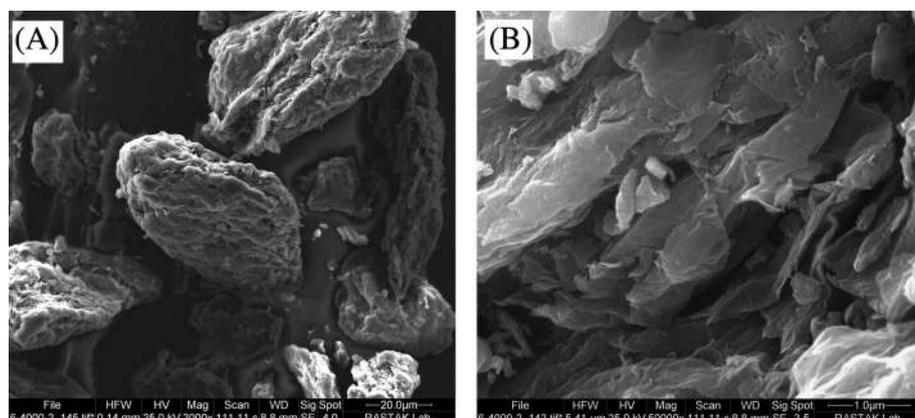


FIGURE 1 Scanning electron microscope images of the as-received graphene nanoplatelets at two different magnifications. A, Low magnification. B, High magnification

room temperature. For comparison, a 0.3 wt% GNs-loaded specimen was also fabricated.

2.4 | Mechanical tests

Tensile properties (modulus and strength) of the specimens were obtained according to the ASTM D638 standard. A Hounsfield: H25K universal apparatus was employed to run the tensile tests at a cross-head speed of 2 mm/min. Wear properties (wear rate and friction coefficient) of the specimens were measured using a pin-on-disk apparatus according to the ASTM G-99 standard. Wear rate (K) was calculated by the following equation:

$$K = \frac{\Delta m}{\rho LP} \quad (1)$$

where Δm , ρ , L , and P represent the weight loss, density, sliding distance, and normal load, respectively.

For all the specimens, an applied load of 10 N, a sliding distance of 1000 m, and a sliding speed of 0.5 m/s were utilized. A 52100 steel pin (64 HRC) was used as a slider. At least three specimens of each type of nanocomposites were tested and the averaged values were reported.

2.5 | Characterization

X-ray diffraction (XRD) signals of the GNs and Ni@GNs were extracted using a BRUKER-XRD (D8 ADVANCE) diffractometer. The measurements were done at a generated voltage of 40 kV using $\text{CuK}\alpha 1$. Morphology of the Ni@GNs was evaluated by an atomic force microscope (AFM) system (ARA Company, Iran). An FEI Quanta 200 SEM equipped with an energy-dispersive X-ray (EDX) spectroscopy was employed to demonstrate the Ni distribution on the GNs surface. Worn and fractured surfaces of the nanocomposites were evaluated using a KYKY 3039M SEM. The specimens were coated with a thin layer of gold prior to observation.

3 | RESULTS AND DISCUSSION

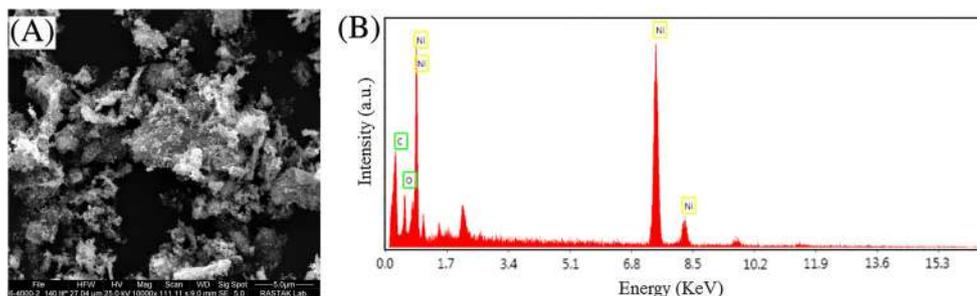
3.1 | Characterization of the Ni@GNs

Figure 2 depicts SEM image as well as EDX spectrum of the Ni@GNs. Obviously, the presence of Ni element on the surface of GNs can be observed. Figure 3 shows XRD patterns of the GNs and Ni@GNs. For the GNs, a sharp peak at around $2\theta = 10.5$ corresponds to the (001) basal plane. On the contrary, XRD pattern of the Ni@GNs indicates three prominent peaks assigned as Ni (111), Ni (220), and Ni (220), demonstrating the presence of FCC-Ni on the GNs surface. Compared to the GNs, the observed shift of the (001) basal plane peak in Ni@GNs is mainly due to the reduction of GNs in the presence of hydrazine hydrate. AFM image and height profile of the Ni@GNs are shown in Figure 4. As indicated in this figure, nanometric-Ni particles are formed on the surface of the GNs. In order to show the distribution of the decorated-Ni particles on the GNs surface, X-ray mapping analysis was conducted and the results are shown in Figure 5. According to this figure, a sought distribution of Ni particles can be observed.

3.2 | Results of the mechanical tests

Figure 6 depicts the variation of the tensile modulus and strength as a function of Ni@GNs content. According to the data in Figure 6, the tensile strength of the 0.1 wt% Ni@GNs/epoxy nanocomposite has been raised to a maximum value of 69.0 MPa, which shows a pronounced enhancement of 36% in comparison to that of the neat epoxy (50.9 MPa). This behavior is due to the enormous surface area of the Ni@GNs which increases their interaction with the epoxy matrix. As it is well documented in the literature, the reinforcing effects of GNs depend strongly on the load transfer capability from the matrix to the GNs.^[14] Hence, a stronger interfacial bonding leads to an effective load transfer, causing enhanced mechanical properties. The Ni-decoration of GNs improves the GNs-epoxy interfacial bonding. In fact, Ni

FIGURE 2 A, Scanning electron microscope image and B, Energy-dispersive X-ray spectrum of the Nickel-decorated graphene oxide nanoplatelets [Color figure can be viewed at wileyonlinelibrary.com]



nanoparticles act as interlocking agents between the GNs and epoxy matrix.

On further increasing the weight percentages of Ni@GNs beyond 0.1 wt%, the values of tensile strength are decreased. Especially, for the 0.7 wt% Ni@GNs/epoxy

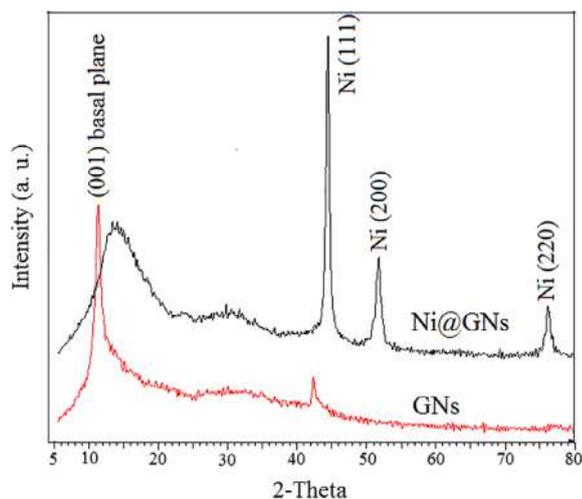
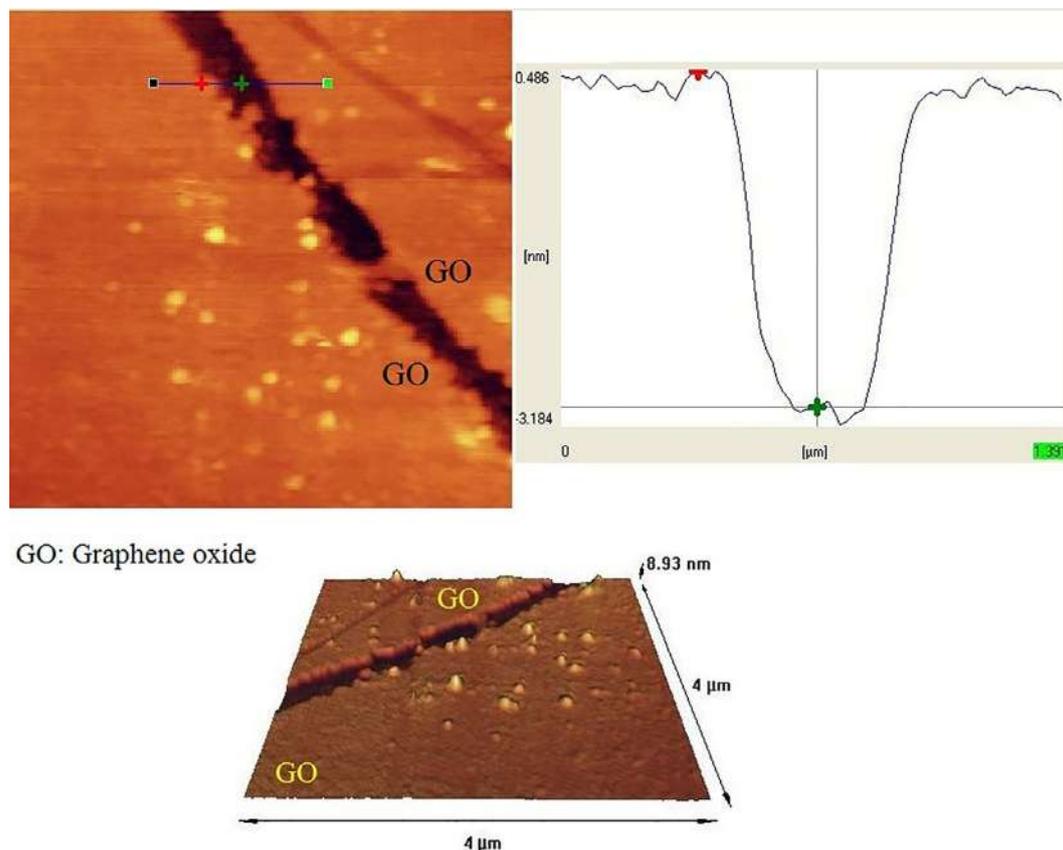


FIGURE 3 X-ray diffraction patterns of the graphene nanoplatelets and Nickel-decorated graphene oxide nanoplatelets [Color figure can be viewed at wileyonlinelibrary.com]

nanocomposite, the tensile strength is lower than that of the neat epoxy. The behavior can be explained in terms of the formation of agglomerates at higher contents of nanofillers. The agglomerates create local stress concentration zones in the matrix.^[13] A weak interface leads to a reduced load transfer capability, which causes declined tensile strength. Moreover, at higher nanofiller loadings, the viscosity of the mixture enhances and as a result, the possibility of air-trapping and formation of micro-voids is increased.

As can be observed in Figure 6, the highest tensile modulus is obtained for the addition of the 0.5 wt% Ni@GNs. At 0.5 wt% Ni@GNs, the tensile modulus is increased by 39%. This behavior is expected due to the higher modulus of Ni@GNs as compared to the epoxy. Moreover, at higher loading of Ni@GNs (ie, 0.7 wt%), the agglomerate size is enhanced. This leads to an easier molecular motion, resulting in reduced tensile modulus.

Figure 7 illustrates the variation of tensile failure strain against the Ni@GNs content. From Figure 7, the failure strain is firstly increased and then declined by increasing the Ni@GNs content. The greatest increase in the value of failure strain is attributed to the 1 wt% Ni@GNs/epoxy composite. For this specimen, a 54%



GO: Graphene oxide

FIGURE 4 Atomic force microscope image and height profile of the Nickel-decorated graphene oxide nanoplatelets [Color figure can be viewed at wileyonlinelibrary.com]

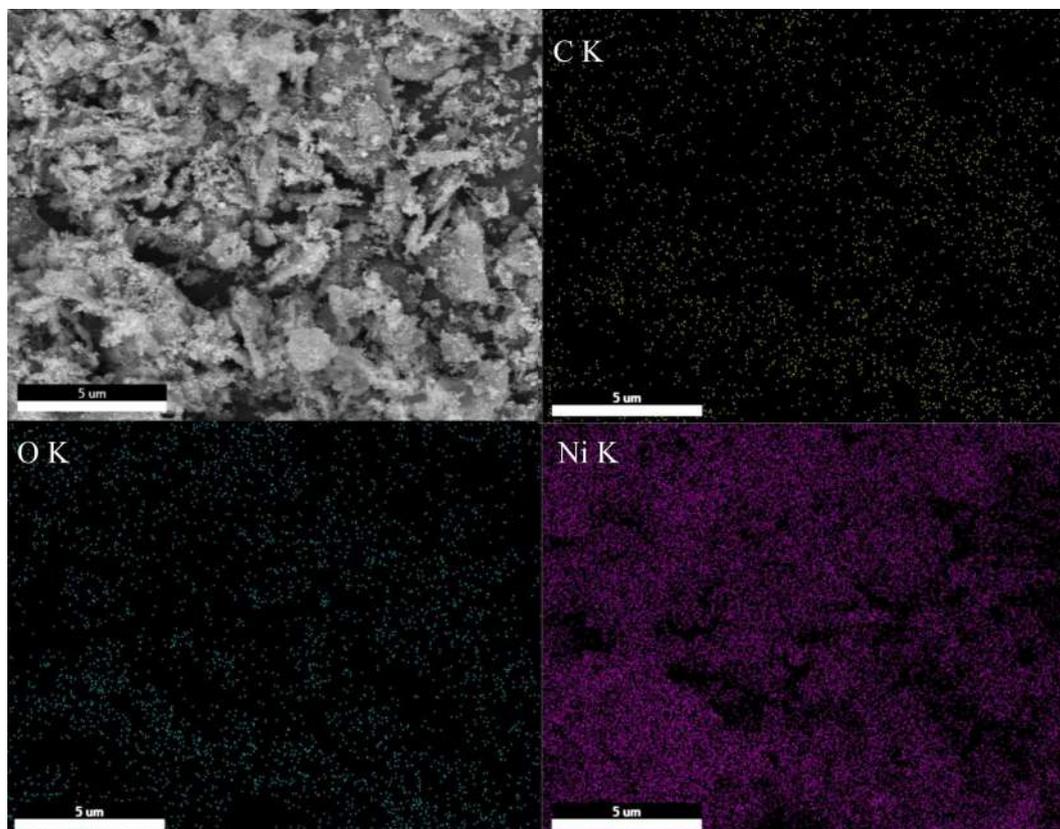


FIGURE 5 X-ray mapping analysis of the Nickel-decorated graphene oxide nanoplatelets [Color figure can be viewed at wileyonlinelibrary.com]

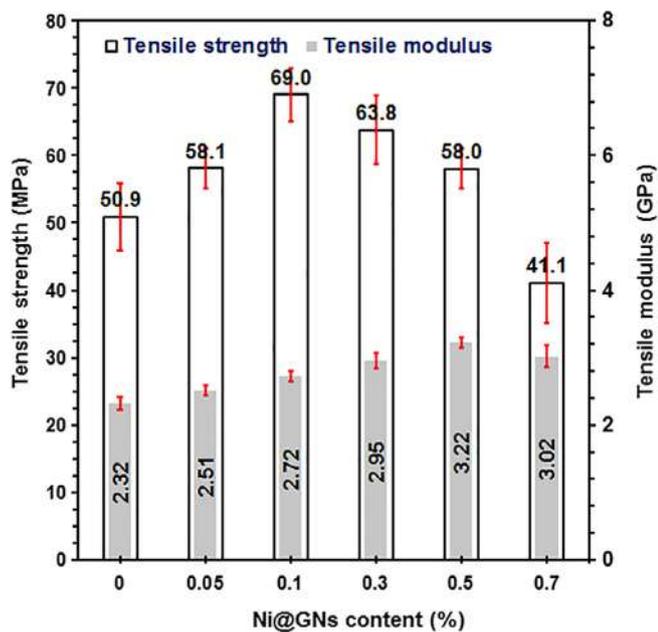


FIGURE 6 Tensile modulus and strength vs Nickel-decorated graphene oxide nanoplatelets content [Color figure can be viewed at wileyonlinelibrary.com]

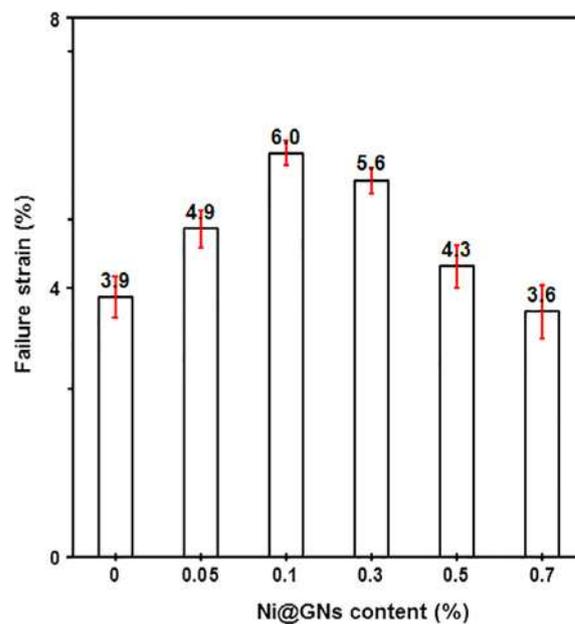


FIGURE 7 Tensile failure strain vs Nickel-decorated graphene oxide nanoplatelets content [Color figure can be viewed at wileyonlinelibrary.com]

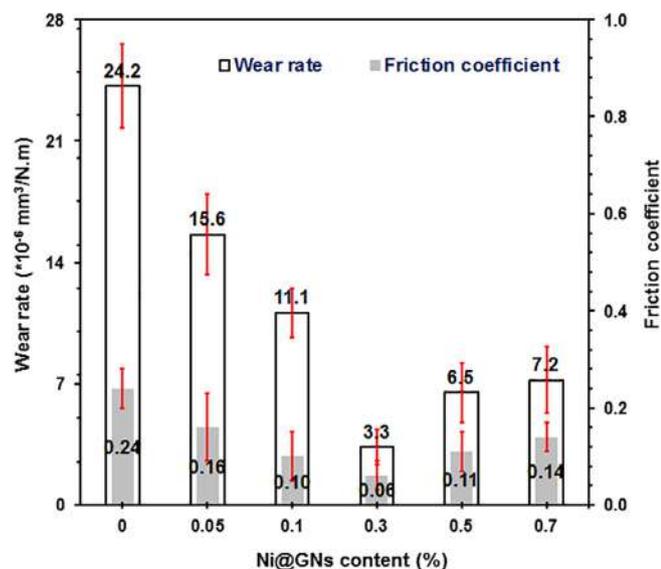


FIGURE 8 Variation of the wear rate and friction coefficient of the specimens as a function of Nickel-decorated graphene oxide nanoplatelets content [Color figure can be viewed at wileyonlinelibrary.com]

enhancement in failure strain is achieved compared to the neat epoxy. The improvement in failure strain by adding the Ni@GNs to the epoxy matrix is mainly due to this fact that when the crack tip reaches the Ni@GNs in the matrix, they alter the direction of crack growth by deflection mechanism and as a result, brittle failure of the matrix is prevented. Similar to the tensile strength, formation of the agglomerates is the main reason for decreasing the failure strain at higher Ni@GNs contents.

Figure 8 depicts the variation of the wear rate and friction coefficient of the specimens as a function of Ni@GNs content. The wear rate and friction coefficient show a descending trend within the addition of 0.3 wt% Ni@GNs, and more Ni@GNs addition leads to an increase in the wear rate and friction coefficient of the specimens. It can be seen that the 0.3 wt% Ni@GNs/epoxy composite possesses the lowest wear rate and friction coefficient, which can decrease by 73% and 75% compared to the neat epoxy, respectively. These results can

be explained by the following issues: (a) Graphene is a lubricant material. Hence, the specimen lubricity is increased with the addition of Ni@GNs, (b) the addition of Ni@GNs increases the stiffness and strength of the matrix, and (c) Ni@GNs particles increase the thermal conductivity of the matrix, helping to reduce the slider-specimen interfacial temperature. As a result, the adhesive friction between the two components is decreased. The presence of the agglomerates at higher Ni@GNs loadings acts as stress concentration zones and increases the wear rate and friction coefficient of the specimens, as can be seen in Figure 8.

3.3 | Effect of GNs modification with Ni on mechanical properties

Table 1 summarized the mechanical properties of the epoxy nanocomposites reinforced by 0.3 wt% GNs or Ni@GNs. One can clearly see that the mechanical properties, such as tensile modulus, tensile strength, failure strain, wear rate, and friction coefficient of the 0.3 wt% Ni@GNs/epoxy are higher than those of the 0.3 wt% GNs/epoxy composite. This shows that the reinforcing effects of Ni@GNs are much better than utilizing GNs alone. The positive effects of nanoparticle-decorated GNs on the mechanical properties of polymers are also reported by other authors.^[25,27] This behavior is mainly due to enhanced interfacial adhesive strength between the GNs and epoxy matrix after surface decoration of GNs with Ni nanoparticles. In addition, Ni nanoparticles act as barriers between GNs and inhibit the aggregation of GNs within the matrix.

3.4 | Evaluation of the fracture and worn surfaces

In order to evaluate the failure mechanisms, SEM analysis was conducted on the fracture and worn surfaces of the specimens. Figure 9 illustrates the representative SEM micrographs of the fracture surface of the neat

TABLE 1 Mechanical properties of the epoxy nanocomposites reinforced by 0.3 wt% GNs or Ni@GNs

Specimen	Tensile modulus (GPa)	Tensile strength (MPa)	Failure strain (%)	Wear rate ($\times 10^{-6}$ mm ³ /N m)	Friction coefficient
0.3 wt% GNs/epoxy	2.69 (0.14 ^a)	52.40 (4.10)	4.10 (0.32)	15.60 (1.38)	0.15 (0.05)
0.3 wt% Ni@GNs/epoxy	2.95 (0.11)	63.80 (5.20)	5.60 (0.19)	3.30 (1.04)	0.06 (0.03)

Abbreviations: GNs, graphene nanoplatelets; Ni@GNs, Nickel-decorated graphene oxide nanoplatelets.

^aThe numbers are the SD.

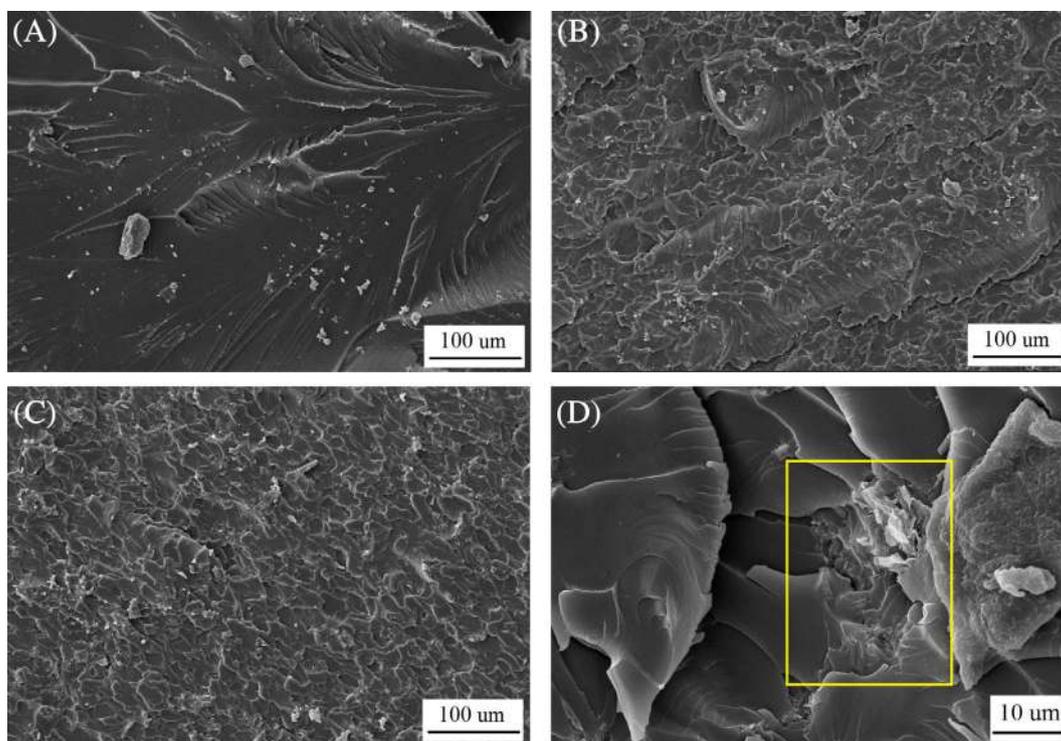


FIGURE 9 SEM micrographs of the fracture surface of the (A) neat epoxy, (B) 0.3 wt% graphene nanoplatelets/epoxy, (C) 0.3 wt% Ni@GNs/epoxy, and (D) the presence of agglomerates in fracture surface of the 0.7 wt% Nickel-decorated graphene oxide nanoplatelets/epoxy composite [Color figure can be viewed at wileyonlinelibrary.com]

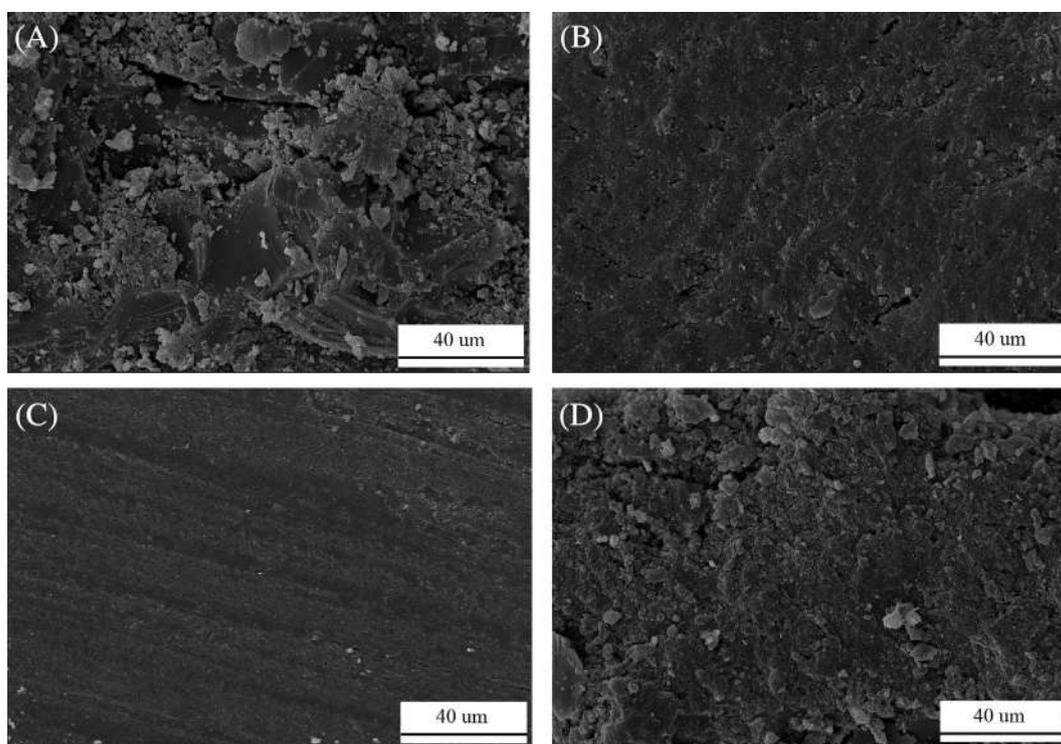


FIGURE 10 SEM micrographs from the worn surface of the (A) neat epoxy, (B) 0.3 wt% graphene nanoplatelets/epoxy, (C) 0.3 wt% Nickel-decorated graphene oxide nanoplatelets (Ni@GNs)/epoxy, and (D) 0.7 wt% Ni@GNs/epoxy

epoxy and epoxy nanocomposites reinforced by 0.3 wt% GNs or Ni@GNs. From Figure 9A, a smooth and nearly featureless fracture surface is observable for the neat epoxy, demonstrating the brittle failure.^[28] Compared with that of neat epoxy, the fracture surfaces become progressively rough with the addition of 0.3 wt% nanofillers in the matrix, as indicated in Figure 9B,C. Such a feature of the fracture surface represents the crack deflection mechanism. Furthermore, almost no remarkable change is seen in the fracture surfaces of the GNs/epoxy and Ni@GNs/epoxy composites (Figure 9B,C). The presence of agglomerates can be observed on the fracture surface of the 0.7 wt% Ni@GNs/epoxy composite (Figure 9D). Such an observation can verify the declined mechanical properties of the specimens at higher loadings of nanofillers.

SEM micrographs from the worn surface of the neat epoxy, 0.3 wt% GNs/epoxy, 0.3 wt% Ni@GNs/epoxy, and 0.7 wt% Ni@GNs/epoxy composites are shown in Figure 10. It can be seen from Figure 10A that there is a severe wear associated with unevenly eroded zones on the worn surfaces of the neat epoxy, implying the low wear resistance. On the contrary, the 0.3 wt% Ni@GNs/epoxy composite exhibits a relatively homogeneous worn surface with shallow scratches, demonstrating a high wear resistance. Clearly, the positive effect of Ni-decoration onto the GNs surface can be confirmed by comparing the worn surfaces of the GNs/epoxy and Ni@GNs/epoxy composites (Figure 10B,C). It can be also seen that for the 0.7 wt% Ni@GNs/epoxy, the matrix damage has noticeably increased as compared to the specimen filled with 0.3 wt% Ni@GNs (Figure 10D) due to the nonuniform distribution of the nanofillers at higher loadings. All these findings are in agreement with the wear test results.

4 | CONCLUSIONS

In this work, tensile and wear properties of the Ni@GNs/epoxy composites were evaluated. Results indicated that by incorporation of 0.1 wt% Ni@GNs in an epoxy matrix, the tensile strength and failure strain were enhanced significantly compared to the neat epoxy (ie, 36% and 56% increase, respectively). Embedding 0.7 wt% Ni@GNs in the epoxy matrix increased the tensile modulus by 39%. The wear rate and friction coefficient of the 0.3 wt% Ni@GNs/epoxy composite were 86 and 75% lower, respectively, than that of the neat epoxy. Totally, the Ni@GNs/epoxy nanocomposite demonstrated better tensile and wear properties when compared to the GNs/epoxy nanocomposite.

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