



RESEARCH ARTICLE – MATERIAL SCIENCE (MISCELLANEOUS)

## Enhancing the Tribological Characteristics of Epoxy Composites by the Use of Three-Dimensional Carbon Fibers and Cobalt Oxide Nanowires

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Article Info.	Abstract
<p><i>Article history:</i></p> <p>Received 16 February 2024</p> <p>Accepted 07 April 2024</p> <p>Publishing 30 June 2024</p>	<p>In this paper, the results of our research were presented into the sliding wear behaviors of reinforced epoxy composites made with 3D networked carbon fibers ornamented with nanowires of cobalt oxides (NWRs@CFs). Composites are made using the cast-in-place method. Under different normal loads and sliding velocities, these composites' wear and friction coefficient performances are assessed. Tests were performed at 400 rpm spindle speed, 10, 20, and 30 N normal load, and a duration of 300 seconds. Pure epoxy exhibits wear rates of 0.449, 0.481, and <math>0.501 \times 10^{-5} \text{ mm}^3/\text{Nm}</math> at 10, 20, and 30N. Conversely, 3D CFs/epoxy composites exhibit lower wear rates (0.334, 0.360, and <math>0.390 \times 10^{-5} \text{ mm}^3/\text{Nm}</math>) at the same pressure. The epoxy composites of NWRs@CFs wear less, measuring 2.1, 3.5, and <math>0.4 \times 10^{-5} \text{ mm}^3/\text{Nm}</math> under applied loads. The effects of speed on the tribological characteristics were also studied, including the friction coefficients for 400, 800, and 600 rpm at 10 N. Pure epoxy has friction coefficients of 0.449, 0.494, and <math>0.552\mu</math> at 400, 800, and 600 rpm. In comparison, three-dimensional carbon fibers and epoxy composites show reduced wear rates (0.334, 0.376, and <math>0.304 \mu</math>) under identical loads. Epoxy composites of NWRs@CFs have friction coefficients of 0.261, 0.304, and <math>0.332\mu</math>. Pure epoxy has high wear, indicating less friction resistance.</p>

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**Keywords:** Tribological Performance; Epoxy Composites; 3D Carbon Fibers; Cobalt Oxide Nanowires.

### 1. Introduction

Epoxy is used for flooring and airport runway repairs because of its inexpensive cost, strong thermal stability, chemical and humidity resistance, adhesive and mechanical performance, and wear resistance. Tribological applications were severely limited by cured epoxy's low wear resistance. Fillers including silica, alumina, clay, and graphene oxide were utilized to solve this problem [1], graphene nano-platelets [2] and multi-wall carbon nanotube [3], short carbon fibers (CF) [4], Polytetrafluoroethylene (PTFE) [5], fly ash [6], and crumb rubber is added to the epoxy matrix, and exhibit improved tribological behavior [7-9]. Additionally, the influence of environmental pollutant crumb rubber on the dry sliding wear behavior of epoxy composites has also been investigated [9, 10].

Research has been conducted on nano ceramic reinforced epoxy composites to assess their ability to enhance the wear resistance of composites. Multiple research projects have shown the efficacy of integrating ceramic particles and nanoparticles into epoxy resin composites. Wear in polymer composites is found to decrease with increasing amounts of nano-silica material, according to research. Polymer composites with nano silica particles had 32.61% better wear performance [11]. Research reveals that epoxy composites containing microsilia particles minimize wear and increase impact energy and hardness [11, 12]. The research examined how SiC affects epoxy resin composite wear resistance and found that a friction-resistant ceramic layer increased it. This makes polymers suitable for abrasive linkages and joints. The study also found that SiC % in polymer composites lowered wear rate [13]. Epoxy composites using  $\text{Al}_2\text{O}_3$  nanoparticles and graphite have improved wear rate and tribological performance.  $\text{Al}_2\text{O}_3$  nanoparticles in epoxy/ $\text{Al}_2\text{O}_3$ /Gr nanocomposites reduce wear and friction, according to studies.

Nomenclature & Symbols			
3D CFs	Three-Dimensional Carbon Felt	$\mu$	Coefficient of Friction
NWRs	Nanowires	$\text{Co}_3\text{O}_4$	Cobalt Oxide
CF	Carbon Fibers	PTFE	Polytetrafluoroethylene
CF/EP	Carbon Fiber/Epoxy	$W_s$	Specific Wear Rate

The composites with 1 wt.%  $\text{Al}_2\text{O}_3$  exhibited the lowest specific wear rate of  $0.7 \times 10^{-4} \text{ mm}^3/\text{Nm}$ , 65% lower than the unfilled epoxy [14]. The research also found that increasing  $\text{Al}_2\text{O}_3$  and graphite particles reduced wear and friction. Nanocomposites showed reduced wear and friction even with minimal loads [14]. A larger concentration of nanoparticles may only slightly reduce the wear rate in reinforced epoxy composites using nanoceramics. For instance, increasing nanoparticle concentration may only slightly reduce wear. Additionally, producing epoxy composites using nanoceramics is difficult. Optimizing material characteristics requires homogeneous nanoparticle dispersion in the epoxy matrix, which may be difficult [15].

High modulus, corrosion and fatigue resistance, and tensile strength are all benefits of carbon fiber reinforced polymers [16, 17]. The transportation, marine engineering, bridge construction, naval architecture, and aerospace industries utilize it. Carbon fiber reinforced polymer is used in friction materials because of its low coefficient of friction, wear rate, and self-lubrication [18]. Carbon composites are widely used yet difficult and expensive to prepare for aircraft brakes. Carbon fiber/epoxy (CF/EP) composites are widely used because they are easy to produce [19, 20]. The resin matrix maintains fibers together in traditional woven carbon fiber/epoxy laminates, causing interlaminar delamination. Interlaminar delamination and inadequate impact damage tolerance wear down friction-sensitive materials quickly. Increased interlaminar strength and impact damage tolerance of laminated plates may make friction materials more abrasion resistant. Nanoparticles, 3D network carbon felts, fiber-matrix adhesion, and matrix toughness strengthen composites [21].

The random distribution of nano-carbon components such as graphene and carbon nanotubes in an epoxy matrix poses a challenge to the progress of using graphene-epoxy composites in the field of tribology. Utilizing three-dimensional network carbon materials, such as graphene aerogel, is a viable approach to completely harness the anisotropic features of graphene. This method enhances epoxy composites and reduces wear rates. Studies demonstrate that including graphene and its derivatives, namely graphene aerogels with a three-dimensional carbon structure, can greatly improve the mechanical and tribological properties of epoxy composites [22]. The production of carbon aerogels (graphene aerogels) can be costly, resulting in a substantial rise in the overall manufacturing expenses of epoxy composites. The cost issue might serve as a constraint for the extensive implementation of industrial applications [21].

Carbon felt foam is a carbon-fiber material characterized by its robust three-dimensional cross-linked structure. It is widely esteemed for its exceptional mechanical robustness, conductivity, low weight, high porosity, and affordable price. Carbon fiber felts have low thermal conductivity as a result of their fibrous porosity structure and strong heat resistance at the interfaces between fibers, which is produced by the noticeable gaps between the surrounding composites. These carbon fiber felts also demonstrate excellent damage tolerance owing to the presence of reinforced fibers [19].

The objective of this study was to investigate the impact of 3D carbon felt and cobalt oxide nano wires on the wear characteristics of polymer composites filled with particles. Cobalt nanowires were synthesized using a hydrothermal method. The composites were assessed under dry conditions using a disc-on-disc setup.

## 2. Experimental Work

### 2.1. Preparation of $\text{Co}_3\text{O}_4$ nanostructure

Hydrothermal and calcination were used to make  $\text{Co}_3\text{O}_4$  nanostructures. With vigorous stirring, 0.8 g  $\text{Co}(\text{NO}_3)_2$ , 0.4 g  $\text{NH}_4\text{F}$ , and 3 g  $\text{CO}(\text{NH}_2)_2$  were dissolved in 50 mL deionized water to make the precursor solution. The solution was chemically activated and placed in a 100ml Teflon-lined stainless-steel autoclave at 160 °C for 5 hours. After naturally cooling to ambient temperature, the sample was washed with deionized water and ethanol and vacuum-dried overnight at 60 °C. To form the oxide phase, the  $\text{Co}_3\text{O}_4$  precursor was annealed at 650 °C in air for 1 hour at 2 °C/min.

### 2.2. Hybrid epoxy composite preparation

The 3D nano $\text{Co}_3\text{O}_4$  carbon felt foam/epoxy composites were fabricated using the cast-in-place method. Initially, a total of 100 grams of epoxy resin and 50 grams of curing agent were vigorously mixed using mechanical stirring while maintaining a vacuum to eliminate any trapped air bubbles. Fig. 1 illustrates the procedure of pouring the epoxy liquid into the 3D carbon felt foam at a temperature of 35 degrees Celsius and allowing it to solidify for a duration of 24 hours. Subsequently, the treated composites were transformed into typical samples to assess their tribological performance.

### 2.3. Materials

Nantong Synthetic Materials Co., Ltd. provided the epoxy resin (E44) and the curing agent (diethanolamine). We sourced the cobalt (II) nitrate from the Chinese company W&Q Fine Chemicals CO., LTD. The Chinese company Fairsky Industrial Co., Limited provided the ammonium fluoride. Co., Ltd. of Taian Guangyuan International Trade supplied the urea. Pure 5mm thick CF foam was received from SGL Carbon Se Co., Ltd., with a density of  $1.79 \text{ g/cm}^3$  and individual felt fiber diameters measuring 23  $\mu\text{m}$ , as shown in Fig. 2.

### 2.4. Characterization and testing

3D carbon felt-Nano cobalt oxide/epoxy composites and nano  $\text{Co}_3\text{O}_4$  morphology were studied using field-emission scanning electron microscopy (Inspect F 50 FEI-SEM Eindhoven, The Netherlands).

A tribological test was conducted to determine the coefficient of friction and wear resistance in accordance with ASTM G-99 using the MMW-1A vertical friction and wear testing equipment (Beijing United Test Co. Ltd., China). It was spinning at 400 revolutions per minute with a force of 10 Newtons.

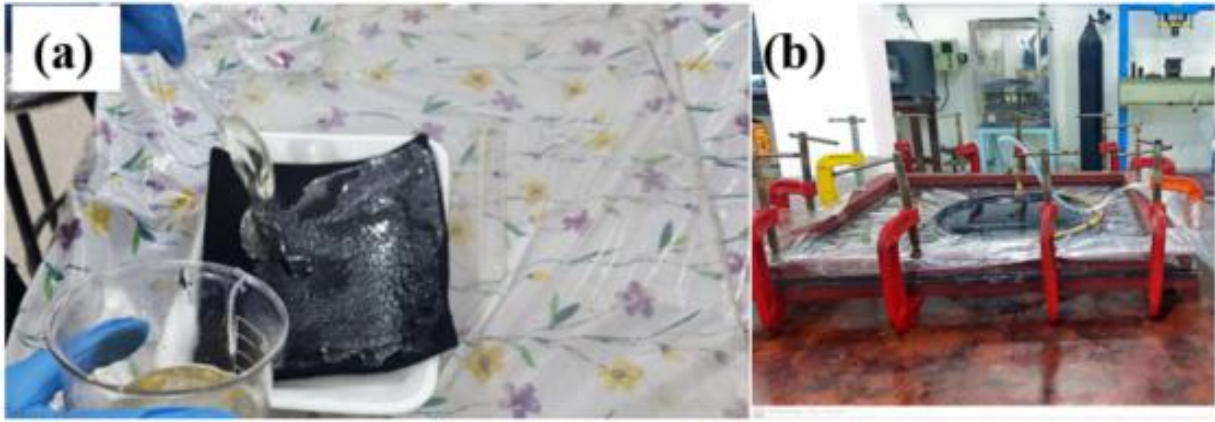


Fig 1. A process of manufacturing 3D carbon felt foam/epoxy composites by the cast-in-place technique; (a) casting epoxy in 3D carbon fibers, and (b) putting 3D carbon foam/epoxy within a vacuum bag to remove the bubbles.

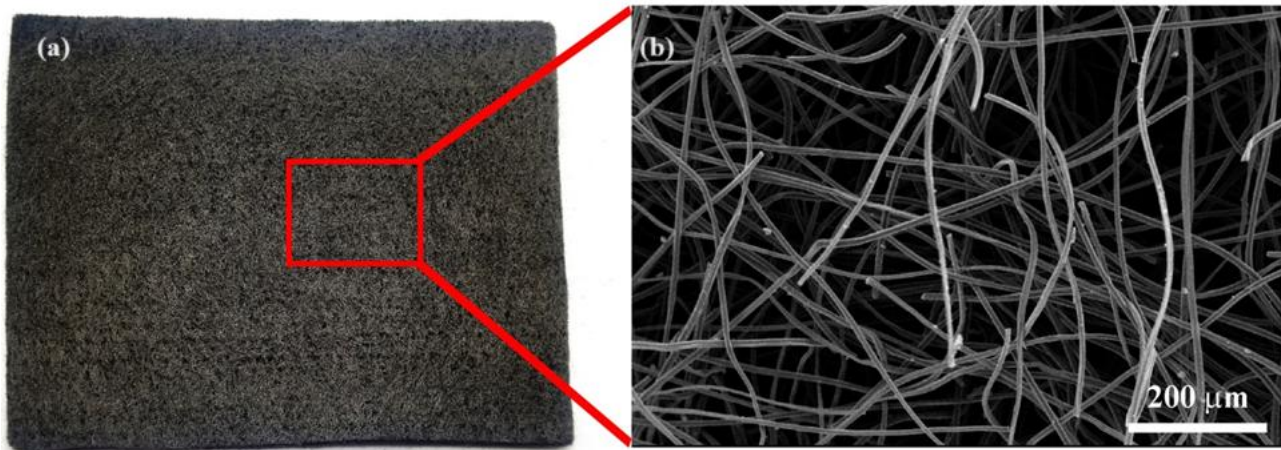


Fig. 2. (a) An optical image of a three-dimensional network made of carbon felt, and (b) scanning electron micrographs depicting the three-dimensional structure of carbon felt

### 2.5. Tribological tests

The coefficient of friction and wear resistance test was performed using the vertical friction and wear testing instrument MMW-1A (Beijing United Test Co. Ltd. - China). The composite was first sliced into a disk with a diameter of about 50 mm prior to testing. In the abrasion tests, SiC was used as the counter-face material, and epoxy composite pins were ground with a grit size of 4000, resulting in a roughness of about 0.03 $\mu$ m. As illustrated in Fig. 3, the pin specimen is paired with silicon carbide (SiC) grinding paper, which acts as the counter-face material in the wear testing setup. Using a balance scale, the mass of each sample was determined both before and after the wear testing process. After making sure the pin samples were perpendicular to the grinding paper, they were carefully put in the specimen holder to ensure they met the necessary contacting conditions. The filing down was completed. The three speeds (400, 600, and 800 rpm) and the applied force (10, 20, and 30 N) were all adjusted to their corresponding values. For each material composition, we took three samples and averaged the mass loss data to get the specified wear rate (Ws). The sample was taken out of the abrasion test, rinsed with ethyl alcohol to remove any wear debris, and then dried before the final mass was measured. Following is an analysis of the mass loss of polymeric specimens using Eq. (1):

$$\Delta m = m_i - m_f \tag{1}$$

In this context,  $\Delta m$  denotes the specimen's mass loss,  $m_i$  denotes its initial mass, and  $m_f$  denotes its final mass after abrasion testing. The quantity of wear was transformed from mass loss to volumetric loss, and the ratio of mass loss to the materials' predicted density is given by Eq. (2) as ( $\Delta V$ ).

$$\text{Volumetric loss } (\Delta V) = \left( \frac{\text{Mass loss, (g)}}{\text{Density, g.cm}^{-3}} \right) \times 1000 (\text{mm}^3) \tag{2}$$

Through the utilization of Eq. (3), the calculated data obtained from the wear test enables the determination of the specific wear rate Ws.

$$W_s = \left( \frac{\text{Volumetric loss } (\Delta V)}{F_n L} \right) \frac{\text{mm}^3}{\text{N.m}} \tag{3}$$

where L is the abrading distance in meters,  $\Delta V$  is the volumetric loss in  $\text{mm}^3$  and  $F_n$  is the applied load in newtons.



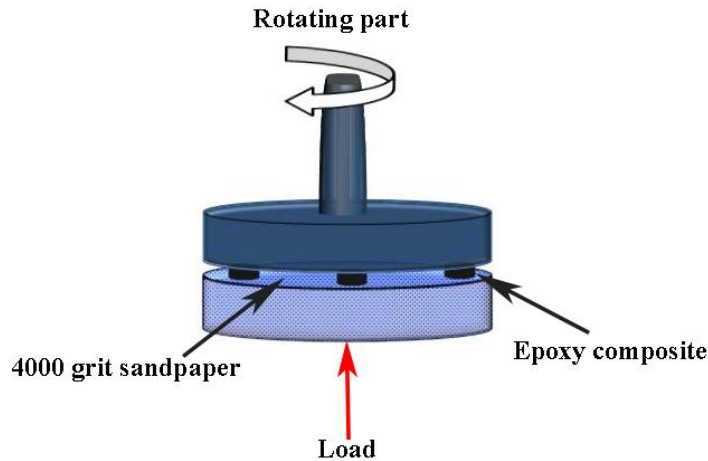


Fig. 3. Schematic of pin sample abrasion against SiC paper-bonded disk

### 3. Results and Discussion

#### 3.1. Morphological studies

The technique of hydrothermal growth was employed to fabricate nanowires (NWRs) of  $\text{Co}_3\text{O}_4$  on a carbon felt foam substrate. Cobalt oxide nanoarrays with unique morphology were produced on a carbon felt foam substrate at hydrothermal temperatures ( $130^\circ\text{C}$ ). Fig. 2 displays scanning electron microscope (SEM) pictures of pure carbon fibers and  $\text{Co}_3\text{O}_4$  nanowires on carbon fibers. Notably, carbon felt has a smooth, clear surface (Fig. 4a). Under the conditions of a temperature of  $130^\circ\text{C}$ , the growth conditions underwent a transformation, resulting in the formation of nanowires (NWRs) that displayed unique characteristics (Fig. 4b).

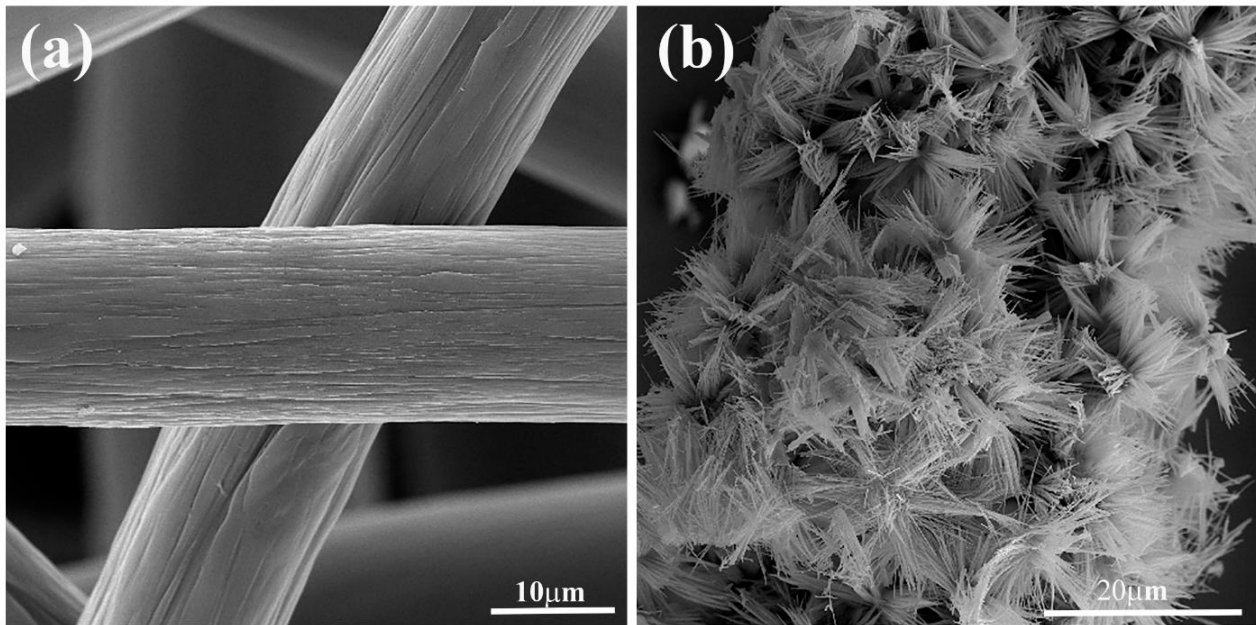


Fig. 4. Scanning electron micrographs depicting the structure of carbon felt and nanowire of  $\text{Co}_3\text{O}_4$  on carbon felt. (a) carbon felt and (b) nanowire  $\text{Co}_3\text{O}_4$ @ carbon felt

#### 3.2. Reinforcing analysis

To evaluate the impact of adding 9.2 wt.% of 3D carbon felt foam with cobalt oxide nanowires on the friction and wear characteristics of 3D CFs-NW $\text{Co}_3\text{O}_4$ /epoxy composites, an investigation was performed to measure the quality of the interface between the fillers and the epoxy matrix. The evaluation was conducted utilizing scanning electron microscopy (SEM). Fig. 5a illustrates that the carbon fibers lacking  $\text{Co}_3\text{O}_4$  nanowires have a feeble contact with the epoxy matrix. Moreover, Fig. 5b illustrates the enhanced bonding between the carbon felt and the surface of the carbon fibers in the epoxy matrix. Improved adhesion reduces the likelihood of wear mechanisms like abrasion and delamination, resulting in a material that is more durable and has an extended lifespan.



Fig. 5. Displays the fracture surface of the 3D CFs/epoxy composites, with and without  $\text{Co}_3\text{O}_4$  nanowire; (a) Three-dimensional carbon fiber/epoxy composites, (b) Carbon nanowires incorporated into carbon fiber/epoxy composites

### 3.3. Tribological test

The study examined the wear and friction properties of 3D carbon fibers (CFs) reinforced with  $\text{NWC}_3\text{O}_4$  nanoparticles in an epoxy matrix. The specimens underwent testing under identical environmental circumstances, with test settings set at a spindle speed of 200 rpm, a normal load of 10, 20 and 30 N, and a duration of 300 seconds.

The wear rates of pure epoxy are  $6.5, 7.8, \text{ and } 8.5 \times 10^{-5} \text{ mm}^3/\text{Nm}$  under normal load of 10, 20, and 30N, respectively. Conversely, 3D CFs/epoxy composites have reduced rates of wear, measuring  $4.2, 5.1, \text{ and } 6.4 \times 10^{-5} \text{ mm}^3/\text{Nm}$  under identical pressures. It is worth mentioning that the epoxy composites of NWRs@CFs demonstrate significantly lower rates of wear, specifically measuring  $2.1, 3.5, \text{ and } 4.1 \times 10^{-5} \text{ mm}^3/\text{Nm}$  under the corresponding applied normal load.

Fig. 6 illustrates the fluctuation in friction coefficient that occurs regarding the usual loads of 10, 20, and 30 N while the samples speed remains constant at 400 revolutions per minute. It has been shown that the friction coefficient of pure epoxy is 0.449, 0.481, and  $0.501 \mu$  when subjected to pressures of 10, 20, and 30N, respectively. In contrast, three-dimensional carbon fibers and epoxy composites exhibit a lower friction coefficient, with measurements of 0.334, 0.360, and  $0.3903 \mu$  when subjected to identical normal loads. It is important to note that the epoxy composites of NWRs@CFs exhibit much-reduced rates of wear. Specifically, the friction coefficient measured 0.261, 0.29216, and  $0.316 \mu$  when subjected to the respective applied loads. The elevated wear rate found in pure epoxy signifies a deficiency in wear resistance. In contrast, the samples that were strengthened, particularly the NWRs@CFs epoxy composites, exhibit a significant decrease in wear rates and coefficient of fraction, indicating that the nanowires on carbon fibers successfully prevent wear in the epoxy matrix. Incorporating cobalt oxide nanowires onto carbon fibers in 3D CFs/epoxy composites leads to a reduction in wear rate by impeding shear deformation under sliding conditions.

Fig. 7 shows the wear rate-composite coefficient-spindle speed relationship. The wear rate as a function of slide speed (400 to 800 rpm) was determined for 300 seconds at a 10 N normal load. Strong fiber-matrix interfaces often reduce friction and wear. This is because loads are transferred more effectively, and stress is distributed more evenly. The material's capacity to resist deformation and to endure external pressures is improved because of the strong link that exists between the fibers and the matrix. Under these conditions, the matrix transfers the applied load to the reinforcing fibers in an incredibly efficient manner. As a result, the material's stress is distributed more uniformly, leading to less friction and wear in certain regions. The fibers and matrix can't slide or separate from one another due to the strong interface, which adds to the material's overall structural stability. There are a few variables that affect the relationship between wear rate and coefficient of friction at higher speeds (RPM) under dry sliding conditions without external lubrication. For example, the amount of heat energy produced by friction increases in direct proportion to the spindle velocity. Rising temperatures have a domino effect on wear rates and coefficients of friction because they cause material hardness to decrease, chemical reactivity to increase, and surface property changes to occur.

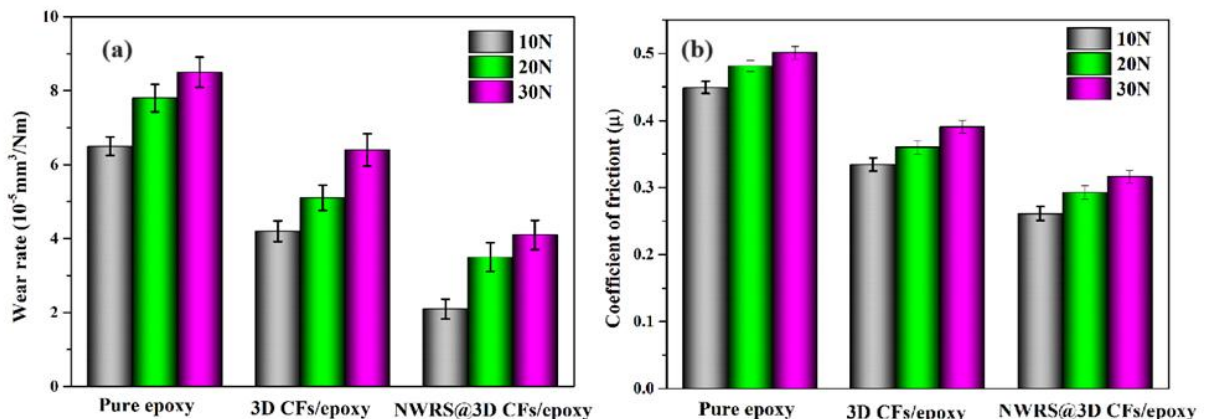


Fig. 6. Wear rate and friction coefficient as a function of type of fillers at different normal loads; (a) wear rate, and (b) friction coefficient

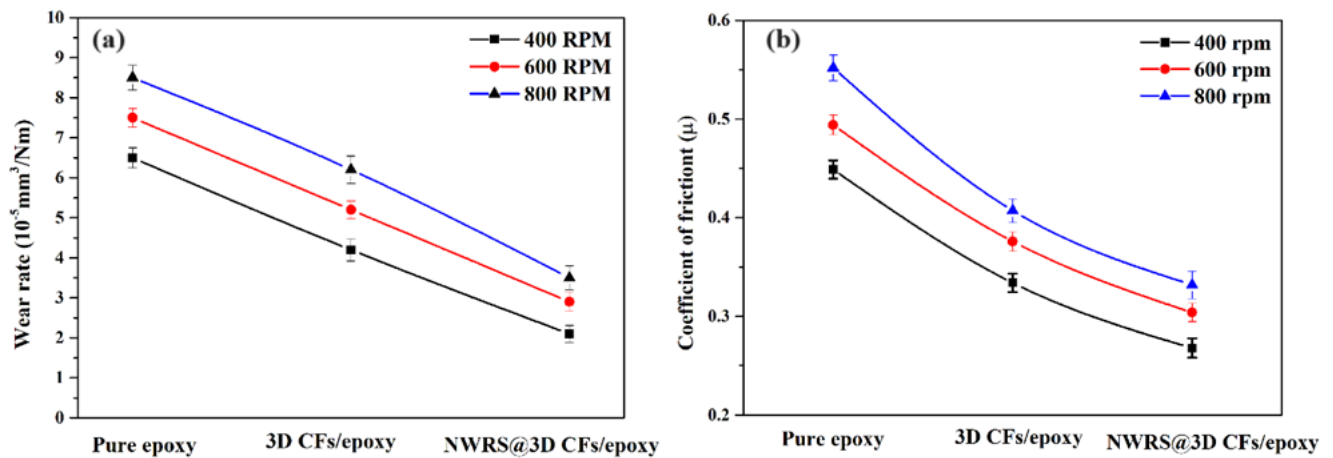


Fig. 7. Wear rate and friction coefficient as a function of type of fillers at different spindle speed (400, 600,800 RPM); (a) wear rate, and (b) friction coefficient

#### 4. Conclusion

The sliding wear behavior of reinforced epoxy composites was investigated, utilizing 3D networked carbon fibers and cobalt oxide nanowires. Manufacturing of cast-in-place composites is carried out. These composites are put through a series of tests to determine their wear and friction coefficient performance under a variety of typical loads and sliding speed. The tests were conducted at a 200 rpm spindle speed, with normal loads of 10, 20, and 30 N for 300 seconds. Pure epoxy exhibits wear rates of 0.449, 0.481, and  $0.501 \times 10^{-5} \text{ mm}^3/\text{Nm}$  at normal loads 10, 20, and 30N, respectively. Conversely, 3D CFs/epoxy composites exhibit reduced wear rates of (0.334, 0.360, and  $0.390 \times 10^{-5} \text{ mm}^3/\text{Nm}$ ) at the same loads. NWRs@CFs epoxy composites show lower wear rates, measuring 2.1, 3.5, and  $0.4 \times 10^{-5} \text{ mm}^3/\text{Nm}$  under the same loads. The friction coefficients for 10, 20, and 30 N loads at 400 rpm are as follows. Pure epoxy has friction coefficients of 0.449, 0.481, and  $0.501\mu$  at loads of 10, 20, and 30 N. Under identical loads, three-dimensional carbon fibers and epoxy composites have friction coefficients of (0.334, 0.360, and  $0.3903 \mu$ ). NWRs@CFs epoxy composites have friction coefficients of 0.261, 0.29216, and  $0.316\mu$  under the loads. High wear indicates little friction in pure epoxy. In addition, the wear rate increased with an increase in sample speed.

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