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DRY SLIDING FRICTION and WEAR PROPERTIES of $CaCO_3$ NANOPARTICLE FILLED EPOXY/CARBON FIBER COMPOSITES

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ABSTRACT

Metal/Fiber-reinforced polymer (FRP) composite joints with lower coefficients of friction are increasingly replaced metal-metal couples in a variety of fields. The wear performance of metal/FRP tribo-contacts becomes a key design parameter for their service life, and the improvement in the wear performance of metal-FRP friction pairs is needed to extend their applications. In this paper, sliding friction and wear characteristics of carbon fiber (CF) reinforced epoxy composites against metallic counterparts were investigated. Tests were performed on a ball-on-disk tester at a constant normal load and velocity against chromium steel under dry ambient. Moreover, calcium carbonate ($CaCO_3$) nano reinforcements were introduced into Epoxy/CF composites to improve their wear performance. The coefficient of friction (65%) and the specific wear rate (75%) were drastically reduced with the addition of $CaCO_3$ nano reinforcements. Worn surfaces were analyzed by scanning electron microscopy (SEM) to evaluate the wear mechanisms. It was concluded that the abrasion dominated wear mechanism of the neat Epoxy/CF composites transformed into adhesion for the multi-scale composites with the addition of cubic $CaCO_3$ nanoparticles, which is responsible for the increased wear performance of neat Epoxy/CF composites. This impact was most likely attributed to two main factors: “nano $CaCO_3$ particles facilitate sliding” and “act as a solid lubricant”.

Keywords: Carbon fiber**,** nano $CaCO_3$, Polymer matrix composite, Sliding wear, Wear mechanism

$CACO_3$ NANOPARTİKÜL DOLGULU EPOKSİ/KARBON FİBER KOMPOZİTLERİN KURU SÜRTÜNME ve AŞINMA ÖZELLİKLERİ

ÖZET

Düşük sürtünme katsayılı metal/fiber takviyeli polimer (FRP) kompozit bağlantılar, çeşitli alanlarda giderek metal-metal çiftlerin yerini almaktadır. Metal/FRP çiftlerinin aşınma performansı servis ömürlerine etki eden anahtar bir tasarım parametresi haline gelmiştir ve metal/FRP sürtünme çiftlerinin kullanım alanlarının genişletilmesi için aşınma performansının iyileştirilmesi gerekmektedir. Bu makalede, karbon fiber takviyeli epoksi kompozitlerin metalik karşıt yüzeylere karşı kayma sürtünmesi ve aşınma özellikleri araştırılmıştır. Testler, sabit bir normal yükte ve hızda kuru ortam altında krom çeliğe karşı ball-on-disk test cihazı üzerinde gerçekleştirilmiştir. Ayrıca, Epoksi/CF kompozitlere aşınma performanslarını artırmak için kalsiyum karbonat ($CaCO_3$) nano takviyeleri eklenmiştir. $CaCO_3$ nano takviyelerin eklenmesiyle sürtünme katsayısı (%65) ve özgül aşınma oranı (%75) önemli ölçüde azaltılmıştır. Aşınma mekanizmalarının değerlendirilmesi için aşınmış yüzeyler taramalı elektron mikroskopisi (SEM) ile analiz edilmiştir. Elde edilen sonuçlar neticesinde, yalın Epoksi/CF kompozitlerde ana aşınma mekanizması olarak gözlemlenen abrazif aşınma, kübik $CaCO_3$ nano takviyelerin ilavesiyle üretilen çok-ölçekli kompozitlerde adhezif aşınma mekanizmasına dönüşmüş ve aşınma mekanizmasındaki dönüşüm yalın kompozitlerin aşınma performansının artmasında temel rolü oynamıştır. Bu etki: “$CaCO_3$ partiküllerin kaymayı mümkün kılması” ve “katı yağlayıcı görevi görmesi” olmak üzere büyük oranda iki temel faktöre dayandırılmıştır:

Anahtar kelimeler: Karbon fiber, nano $CaCO_3$, Polimer matris kompozit, Kayma aşınması, Aşınma mekanizması

1. INTRODUCTION

It is well known that the coefficients of friction (~0.7-1.0) are comparatively high for the steel-steel contacts [1]. Recently, metal-fiber reinforced polymer (FRP) pairs that have lower coefficients of friction are coupled together in advanced industrial applications such as automotive, aircraft, aerospace, and electrical components [2]. Carbon fiber reinforced epoxy composite

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materials are replacing traditional metallic materials in automotive, aerospace, marine, defense, and various industrial fields due to their high weight resistance rates [3]. Although it has many advantages compared to metallic materials, it may be necessary to improve the tribological properties of fiber-reinforced composites for specific applications. The improvement of the tribological performance of fiber-reinforced composites is through enhancing the physical, thermal, and mechanical properties [4]. It is prominent to introduce micro- and nano-scale particles as additives into the matrix, which is one of the methods used in improving the mechanical and tribological performance of composite materials recently [5]. The hardness, modulus, and strength of the matrix can be enhanced by adding ceramic-based nano reinforcements with high wear performance [6]. In addition to reinforcing the matrix, forming wear debris containing nano reinforcements is a useful strategy, which reduces friction between the peer surfaces. In recent years, studies examining especially nano-sized reinforcements have been increasing. Carbon nanotube [7, 8], graphene [3, 9, 10], and clay [11, 12] are among the most researched inorganic nano additives. Recently, nano-$CaCO_3$, which is of interest among inorganic fillers, is used in the polymer applications due to its low cost [13] as well as reinforcing and hardening effects [14].

In various studies, it was stated that the introduction of nano-sized $CaCO_3$ to different composites generally improved their mechanical performance. $CaCO_3$ nanofillers significantly increased impact resistance in polypropylene composites [15, 16]. In the case of CaCO3, nanofillers were introduced to the epoxy matrix, compressive strength, elasticity modulus, flexural modulus, impact strength, and thermal stability [17-19] have been improved, particularly with low concentrations. In studies on glass [20] and carbon [18] fiber-reinforced composites, it was stated that the $CaCO_3$ additive also positively improved the impact strength, thermal stability, fracture toughness, and bending properties of the composites. The wear resistance of thermoplastic polymer composites such as polyether-ether-ketone (PEEK) [21], poly-tetra-fluoro-ethylene (PTFE) [22], acrylonitrile-butadiene-styrene (ABS) [23], polyamide 66 (PA66) [24] and polycarbonate (PC) [25] with $CaCO_3$ additive has increased in dry sliding conditions, thereby improving their tribological properties.

However, there is very limited information on the tribological properties of $CaCO_3$ nanoparticle filled thermoset polymers. Therefore, it is imperative to investigate the effects of $CaCO_3$ reinforcements on the tribological behaviour of $CaCO_3$ nanoparticle filled epoxy and their carbon fiber composites. In this paper, the tribological properties of Epoxy/CF multi-scale composites modified with various amounts of $CaCO_3$ nanoparticles were investigated. Wear tests were performed on a ball-on-disc wear tester under different test conditions. In the observed wear mechanisms based on nanofiller content and test, parameters were interpreted with SEM images captured from wear surfaces.

2. MATERIAL and METHOD

2.1. Materials

Epoxy (MGS-L160) and curing agent (MGS-M160) as matrix material were commercially supplied from Momentive Hexion Inc. $CaCO_3$ nanoparticles (98% purity) with cubic morphology and 100 nm size were purchased from the Chengdu Kelong Chemical Co., Ltd. company. Plain weave carbon fiber fabric with 300g/m^2 areal density was obtained from the DowAksa company.

2.2. Production of Multi-Scale Composites

The production scheme of nano-$CaCO_3$ modified multi-scale Epoxy/CF composite laminates is given in Fig. 1. The Vacuum-Assisted Resin Infusion Molding (VARIM) process was used to produce the multi-scale laminate plates consisting of ten plies of carbon fabrics, and the procedure in similar studies previously conducted in the literature has been applied [26]. First, the epoxy matrix was modified with $CaCO_3$ nanoparticles. The particles were mixed in acetone with an ultra sonicator for 15 minutes to prevent agglomeration. Following, the epoxy resin was introduced into the mixture, and the resultant mixture was further stirred with epoxy for another 30 min. To evaporate acetone, the solution was kept in a vacuum oven at 70 °C for 24 hours. After the evaporation process, the curing agent was added into the mixture in accordance with the manufacturer's recommendations, and the mixture is mixed mechanically for 5 min. To minimize the air bubbles formed during the mixing process, the mixture was degassed in a vacuum oven at 70 °C for 15 minutes, and degassing was performed. During the production of multi-scale carbon fiber reinforced composite material, the stainless steel mold was heated up to 70 °C to reduce the viscosity of the matrix polymer. The prepared epoxy matrix is impregnated with fiber-reinforced composite layers. The samples cured at 70 °C for 1 h and post-cured at 120 °C for 4 h. Through this production process, 1wt%, 3wt%, and 5wt% CaCO3 nano-reinforced Epoxy-CF multi-scale laminates were obtained. Also, the production of epoxy composite material without nanoparticle additives was carried out by similar processes.

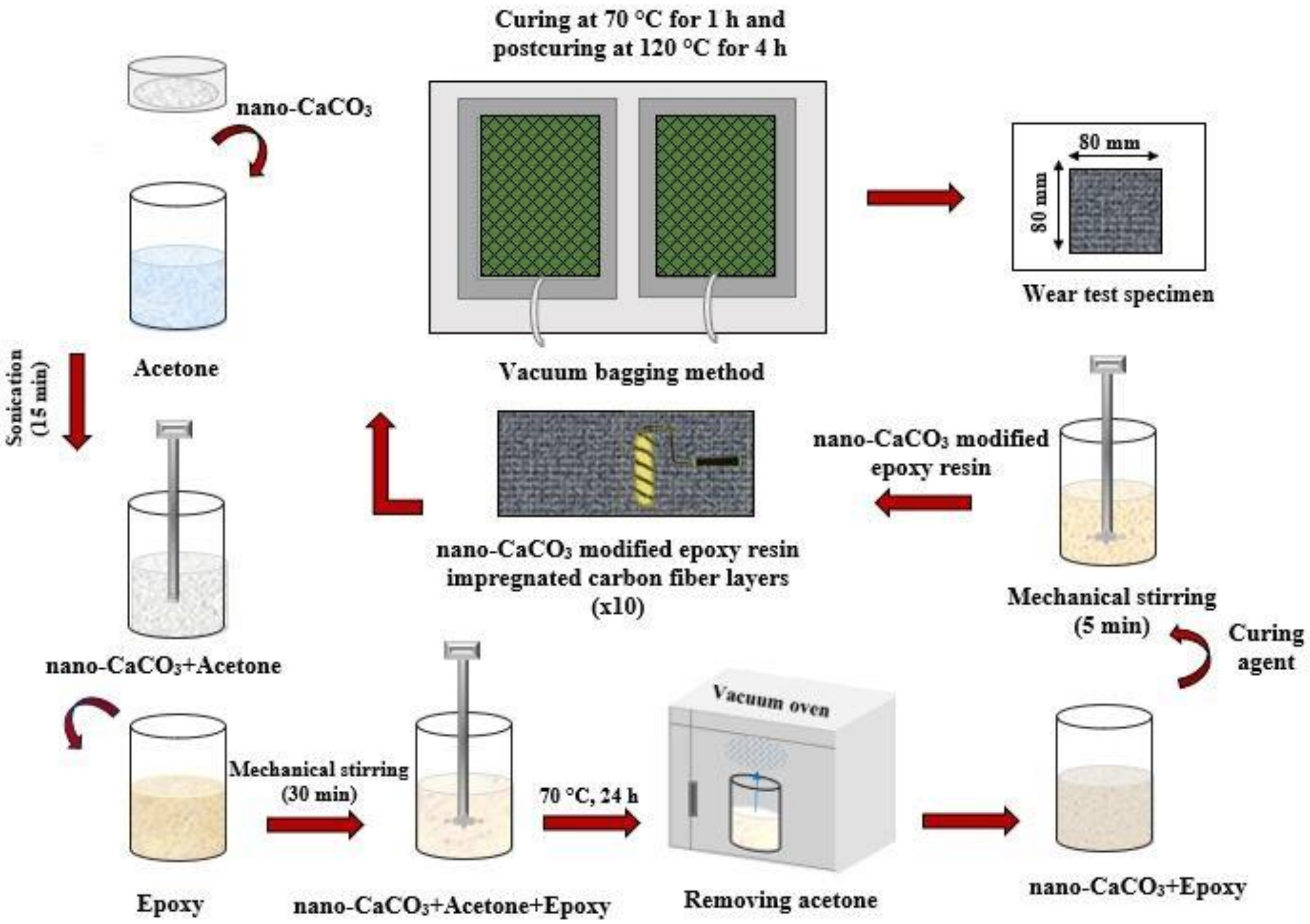

Figure 1. Production scheme of composite materials

2.3. Friction and Wear Tests

The tribological performance of the samples was determined under dry sliding conditions with the ball-on-disc wear tester (ASTM-G99) shown in Fig. 2. As an abrasive ball, a 6 mm diameter 62 HRC hardness chrome steel ball is used. The friction and wear tests were performed at ambient temperature under a load of 10 N at a sliding velocity of 0.75 m/s and a sliding distance of 2700 m.

After the experiments, the weights of the samples were measured with the help of precision scales, the weight losses were determined and Eq. 1 was used in the wear rate calculation.

$$Ws = \frac{\Delta m}{\rho . F_N . L} mm^3/Nm \quad (1)$$

Here, L represents the sliding distance (m), F_N applied load (N), ρ sample density (gr/mm^3), and Δ_m the weight loss (gr) of the wearing sample.

The morphologies of the worn surfaces of the samples were analyzed using a Zeiss LS 10 scanning electron microscope (SEM).

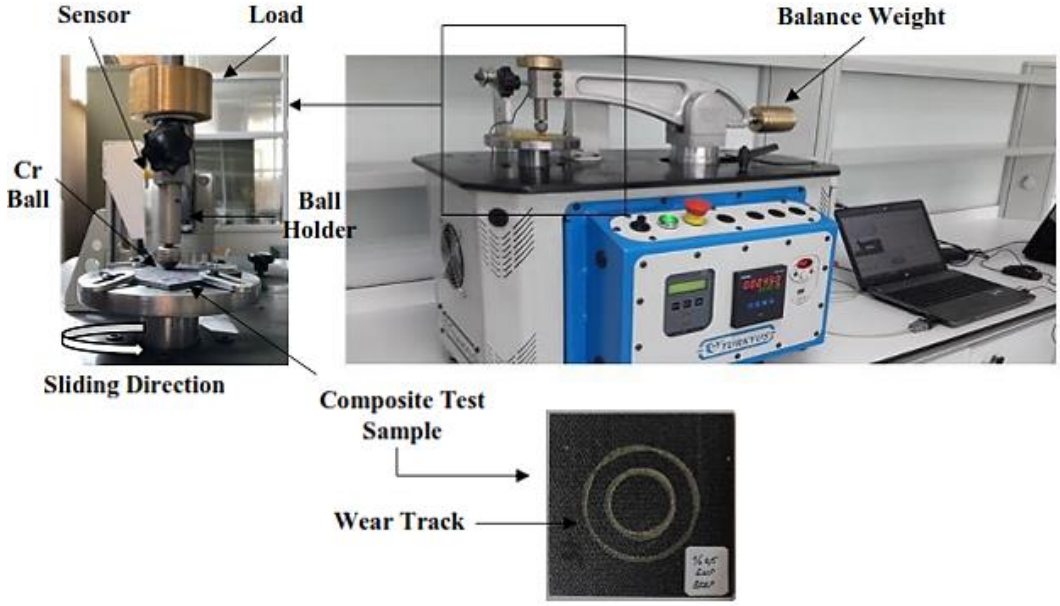

Figure 2. Ball-on-disc wear tester

3. RESULTS and DISCUSSION

3.1. Dry Sliding Wear Tests

Figure 3 represents the variation of characteristic coefficients of friction to time. It is observed that the neat Epoxy/CF (Fig. 3.a) and 5wt% nano-$CaCO_3$ modified multi-scale composite samples (Fig. 3.d) composites exhibit steady wear performance after a short initial running-in period. However, the 5% nanofiller content provided a serious stabilization in the coefficient of friction curve. In the case of 1wt% (Fig 3.b) and 3wt% nano-$CaCO_3$ (Fig. 3.c) modified multi-scale composites, reaching the steady-wear stage took longer initial periods. Steady-wear stage data are essential in interpreting the friction behaviour of the material and characterizing the long-term properties of the system. [27]. The steel ball, which contacts the surface in dry sliding wear, is initially embedded in the surface and forms a wear track during the running-in period. During the running-in period, the coefficient of friction depends on parameters such as hardness, surface roughness, sliding speed, and applied load. In this study, wear tests were performed under the same sliding velocity and load. In general, therefore, these findings suggest that the most crucial factor in the transition to the stable wear zone is the surface morphology of the composites. Composites are not standardized materials, and the epoxy layer on their surfaces may differ from each other after vacuuming during the production phase. It is attributed to the fact that the Epoxy /CF composite is in the stable wear zone from the start.

In all cases, the addition of $CaCO_3$ nanofillers facilitates sliding due to its friction-reducing properties [28], which is effective in the transition from the running-in zone to the stable wear zone. For the multi-scale composites with 5wt% nano-$CaCO_3$, both the short running-in period and the stabilization in the coefficient of friction curve in the steady wear stage reveal the positive impacts of the nano-$CaCO_3$ additive.

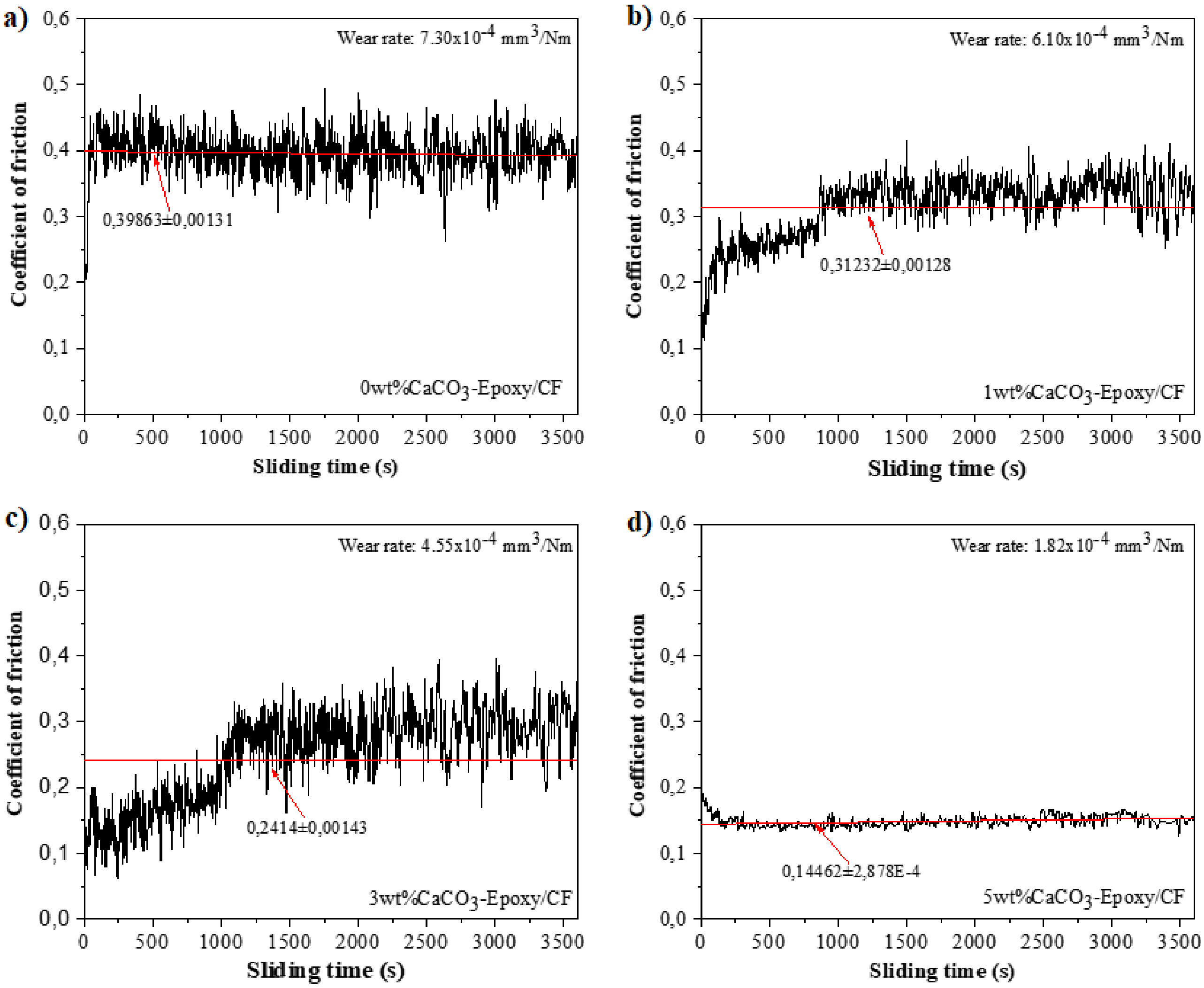

Figure 3. Variation of the coefficient of friction of the **a)** 0wt% $CaCO_3$-Epoxy/CF **b)** 1wt% $CaCO_3$-Epoxy/CF **c)** 3wt% $CaCO_3$-Epoxy/CF and **d)** 5wt% $CaCO_3$-Epoxy/CF composites with sliding time.

The correlations of the coefficient of friction and wear rates of composites with increasing nano-$CaCO_3$ content are given in Fig. 4. Both coefficients of friction and wear rates of multi-scale composites show a decreasing trend with increasing weight percentage.

The mean coefficient of friction of the neat Epoxy/CF composite is calculated as 0.4. Compared to the neat samples, dramatic reductions are achieved for the multi-scale composites. The lowest coefficient of friction is calculated as 0.14 for the multi-scale composites containing 5 wt% nano-$CaCO_3$, which is approximately 65% lower than that of the neat composite laminates (Fig. 4.a). The wear rate of the neat Epoxy/CF composite is calculated as $7.30*10^{-4}$ mm^3/Nm. The lowest wear rate is calculated as $1.82*10^{-4}$ mm^3/Nm for the multi-scale composites containing 5 wt% nano-$CaCO_3$, which is approximately 75% lower than that of the neat composite laminates (Fig. 4.b). The change in tribological properties is thought to be caused by the change in the mechanical properties of the composites with the nano-$CaCO_3$ additive [26] and internal structures such as the homogeneous dispersion of nano-$CaCO_3$ in the nanocomposites [29]. Here, the nano-$CaCO_3$ additive with good dispersing properties [28, 30] has two important impacts on the wear performance of Epoxy/CF composite laminates. These are, with its antiwear and friction-reducing properties [28], nano-$CaCO_3$ facilitates sliding and provides lubrication under dry sliding conditions with its solid lubricant feature. [31, 32].

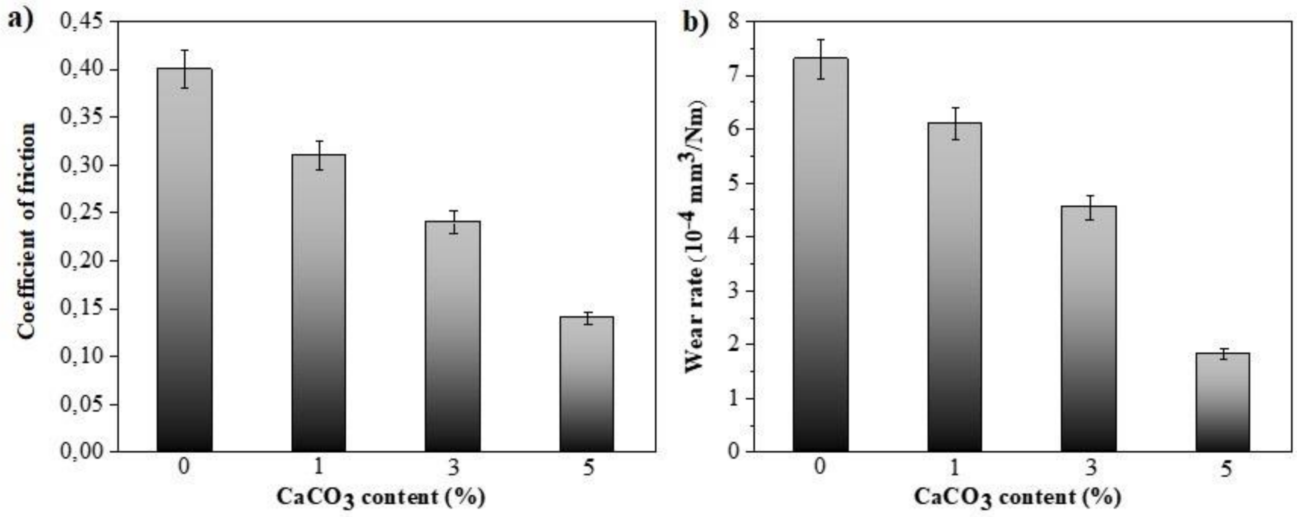

Figure 4. Effect of nano-$CaCO_3$ content on **a)** coefficient of friction and **b)** specific wear rate of the Epoxy/CF composites

SEM images of neat Epoxy/CF and nano-$CaCO_3$-Epoxy/CF composites are given in Fig. 5. Abrasive wear, which is the primary wear mechanism for the neat Epoxy/CF composite, has removed the epoxy matrix locally from the surface as the sliding continues (Fig 5.a). Fiber breakages are observed in those regions, and micro-crack formations and wear debris are seen where the matrix is not completely removed (Fig. 6.b). In the case of multi-scale composite samples with 1%wt nano-$CaCO_3$ content, both abrasive and adhesive wear types are observed. (Fig 5.c). Matrix cracks are seen on the surface with abrasive wear. The matrix cracks were observed close to the worn surface; no fiber damage was obtained. $CaCO_3$ nanofillers cover the matrix region, allowing the matrix to protect the fibers in areas where abrasive wear is active. (Fig. 5.d). In the adhesive wear zones, wear debris are plastered on the abrasive ball surface as the sliding continues. The adhesive wear mechanism is effective in 3wt% (Fig. 5.e) and 5wt% nano-$CaCO_3$ modified Epoxy/CF (Fig. 5.g) multi-scale composites. As the abrasive ball continues to slide on the contact surface, the temperature at the contact point increases with the effect of friction. Ceramic nanoparticles with high thermal conductivity can increase the thermal conductivity of the composite into which they are added. Increased thermal conductivity can contribute to a reduction in the contact area temperature during sliding [5]. He et al. stated that thermal properties improved up to 4wt% nanofiller content, a decrease in the thermal properties occurred with the increasing $CaCO_3$ content due to the agglomeration. Possible agglomerations at high nanofiller contents do not limit molecular mobility, so the decomposition temperature decreases [33]. The reduction in thermal conductivity caused by possible agglomerations in 3 wt% nano-$CaCO_3$-Epoxy/CF composites restricts the homogeneous dissipation of the heat generated at the contact point during wear, contact point temperature increases. High frictional heat and contact zone temperature causes thermal softening in the matrix [34]. Significant plastic deformation zones appear with the effect of thermal softening (Fig. 5.f) In their study where the mechanical properties of CaCO3 reinforced carbon fiber composites were examined, Eskizeybek et al. achieved the highest mechanical performance with the addition of 2wt% nanofiller content. After 2% additive, there were decreases in properties [26].

Possible reductions in mechanical properties in 5 wt% nano-$CaCO_3$-Epoxy/CF composites facilitate deformation during adhesive wear and subsequent adhesion of the deformed particles to the surface. Thus, marked sliding tracks (Fig. 5.g) and scratches (Fig 5.h) are observed on the wear surfaces.

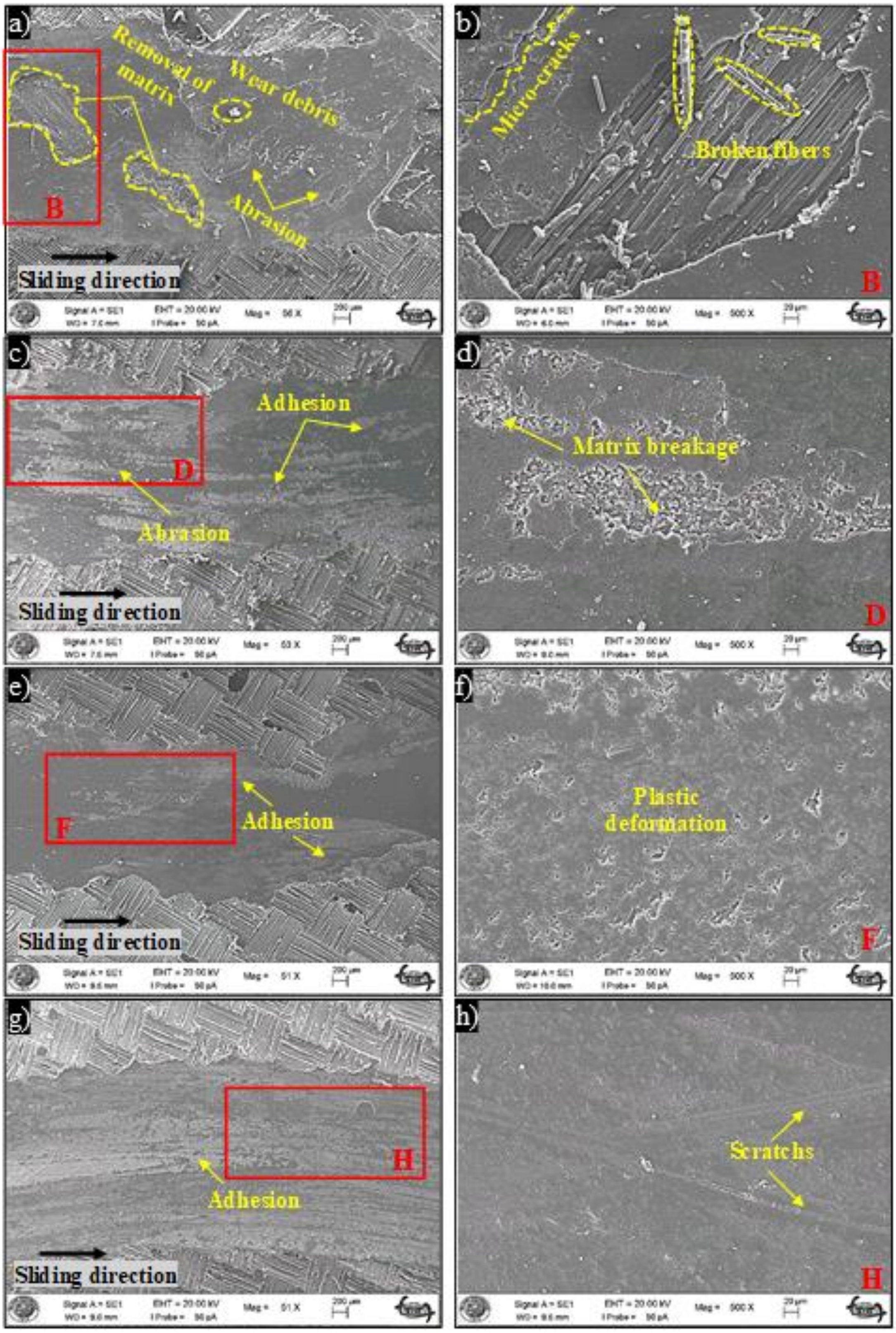

Figure 5. SEM images of worn surfaces of the **a)-b)** 0wt% $CaCO_3$-Epoxy/CF, **c)-d)** 1wt% $CaCO_3$-Epoxy/CF, **e)-f)** 3wt% $CaCO_3$-Epoxy/CF, **g)- h)** 5wt% $CaCO_3$-Epoxy/CF composites

The possible wear mechanism that occurred is given schematically in Fig. 6, Figure 6.a) represents dry sliding wear and wear track formation. The primary mechanism for the neat Epoxy/CF composites is abrasion, as given in Figure 6.b) The incompatibility and weak bonding between the fiber-matrix allow the matrix to be easily removed from the surface under the influence of abrasion. Besides, micro-cracks can be seen on the surface. In areas where the matrix is removed, breaks may occur in the fibers along with the matrix. The fibers and matrix, which are broken down under the effect of the sliding that continues after breaking, appear as wear debris on the surface. In nano-$CaCO_3$-Epoxy/CF composites, adhesion wear dominates the contact surfaces. During adhesion wear, it is easier for particles to break off from the surface and then re-plastered to the surface, due to the effect of agglomerations and mechanical properties that increase as the additive rate increases. The debris that forms a film layer by plastering on the surface makes it easy to slide with the solid lubricant effect of $CaCO_3$.

Sliding tracks and scratches are observed on the surface in line with wear. Thermal properties worsening in increasing additive ratios cause the heat generated by the effect of friction at the contact point not to be distributed homogeneously. The contact point temperature increases. Increased temperature causes thermal softening of the polymer, and softening causes plastic deformations.

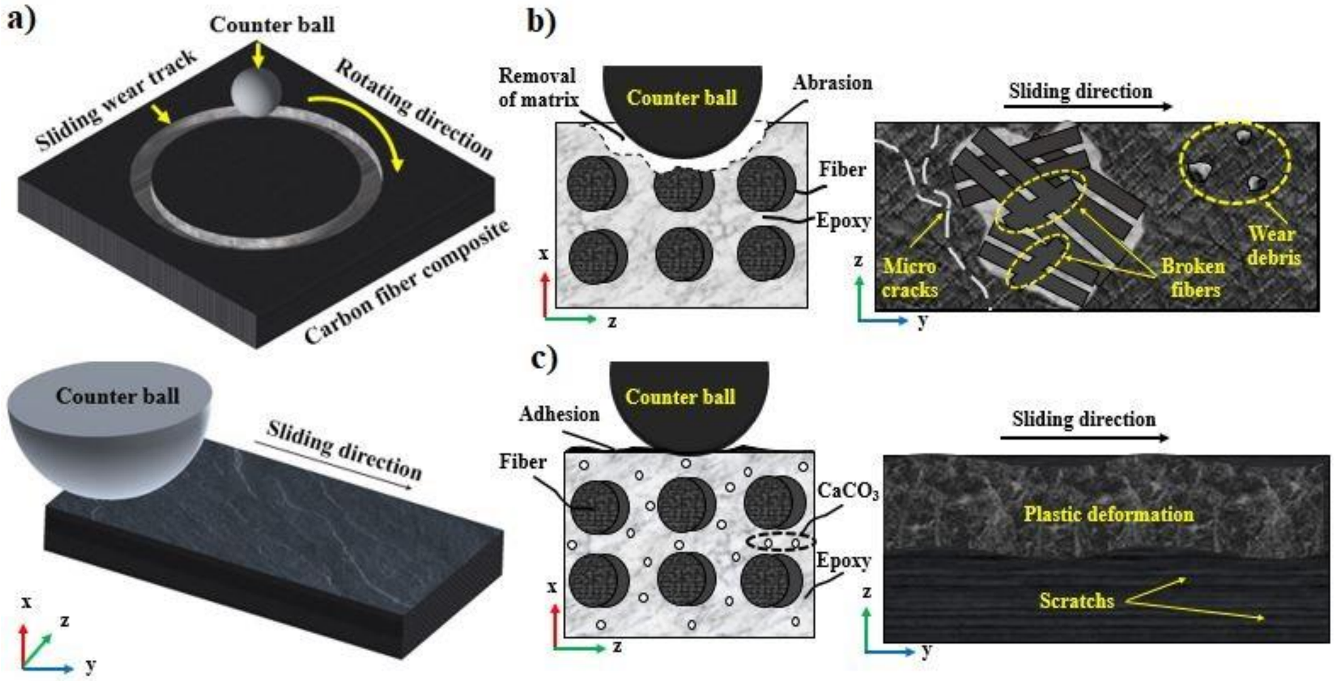

Figure 6. Schematic illustration of dry sliding wear mechanism
a) schematic of ball-on-disk wear test **b)** neat Epoxy/CF and **c)** multi-scale $CaCO_3$-Epoxy/CF

4. CONCLUSIONS

In this work, the effect of nano-$CaCO_3$ particles on the tribological properties of carbon fiber reinforced epoxy composites were studied. The experimental data reveals the following conclusions:

- The dispersion of nano $CaCO_3$ in the matrix improves friction and wear performance of Epoxy/CF composites.
- It is clear, for all nano-$CaCO_3$-Epoxy/CF composites, that the coefficient of friction and specific wear rate decreases with an increase in the percentage of nano-$CaCO_3$-Epoxy/CF.
- The nano-$CaCO_3$ particle reinforcement was reduced the mean coefficients of friction and wear rates of Epoxy/CF composites by 22.5%-65% and 16%-75%, respectively.
- The 5 wt% nano-$CaCO_3$ content dramatically reduced the mean coefficients of friction and wear rate by 65% and 75%, respectively.

- Several wear mechanisms such as abrasion, ploughing, crack formation, the fracturing of fiber and matrix, adhesion, and plastic deformation were observed. Abrasion and adhesion are the primary wear mechanism for neat Epoxy/CF and nano-$CaCO_3$-Epoxy/CF composites, respectively.

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