Tribological behavior of epoxy nanocomposites under corrosive environment: effect of high-performance boron nitride nanoplatelet

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ABSTRACT: This paper sheds light on the impact of corrosive service conditions on the mechanical and tribological performances of epoxy nanocomposites containing 0.5 wt.% hexagonal boron nitride (BN) nanoplatelets as fillers. The composite samples were aged in 10 wt% H_2SO_4 solutions by immersion for 504 hours. Variation in mechanical and tribological behaviors during immersion was investigated using tensile and ball-on disk tests. The ball-on disk tests were performed at room temperature under a 10 N constant load and 0.8 m/s speed for 3000 seconds. The high-performance BN nanoplatelets improved neat Epoxy's tensile strength, toughness, and wear performance by 17%, 27%, and 56%, respectively. The aging results revealed that H_2SO_4 exposure deteriorates the mechanical performance of all epoxy composite samples, even though BN/Epoxy nanocomposites exhibited better resistance to the corrosive environment. In contrast, the tribological performance increased with the acidic solution exposure that acts as a lubricant and forms a corrosive protective layer after the immersion of 504 hours.

Key Words: Corrosive environment, ball-on disk, epoxy, hexagonal boron nitride, sulphuric acid, wear.

1. INTRODUCTION

Thermoset polymers are a promising material solution for different engineering applications. Epoxy is the most preferred thermoset group polymer in various industrial usages such as automotive, marine, aerospace, transports, engineering structures, and adhesive bonded joint due to their low cost, low friction coefficient, low shrinkage upon cure, good adhesion to different material types, high mechanical performance, and corrosion resistance [1,2]. However, aggressive service conditions like temperature, humidity, UV radiations, acid, alkaline, saline atmospheres have a destructive effect on the mechanical and wear behavior of polymer systems [3,4]. Epoxy-based structural parts/epoxy coatings, especially in petroleum products, aggressive liquids, and hazardous chemical waste transport pipes-storage containers, can be exposed to acidic conditions throughout their service life [5]. The literature studies revealed that the exposure of the acidic solution causes the degradation of epoxy systems [6]. As a result, the mechanical and wear performances of the epoxy resin decreased in the acid solution with increasing pH ratio and immersion time through chemical reactions between the filler and the acid solution in the epoxy compound [3]. On the other hand, modifying the polymer resin with nanoreinforcements can prevent deterioration and improve their service performances. In the last several years, many researchers have reported the nano reinforcements such as Si_3N_4, SiC, ZnO, Al_2O_3, TiO_2, SiO_2 MnO_2, $CaCO_3$, graphene, halloysite significantly improve the mechanical and tribological properties of polymer composites [7–16].

Lately, boron nitride (BN) nano reinforcement has allured concern owing to their low density, good thermal properties, high resistance against acids and aqueous environment, low friction coefficient, and their potential industrial area in manufacturing polymer composites [17,18]. Many studies have demonstrated that the BN particle reinforcement is a preferable process to enhance the mechanical performances of epoxy-based

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polymer composites [18,19]. Ulus et al. designed a set of epoxy nanocomposites with superior tensile strength by utilizing the different weight ratios of BN particles as nanofillers [20]. Kaybal et al. reported the improved machinability of fiber-reinforced composite with BN nano reinforcement due to the excellent thermal properties in the drilling process [21].

On the other hand, by including nano BN fillers in epoxy resins, tribological properties also can be improved under dry friction conditions [22]. Usage capability in the tribological application of epoxy polymer is directly related to their enhanced mechanical/wear properties and minimized friction coefficients. Düzcükoğlu et al. examined the wear performances of epoxy resin reinforced with BN and multi-walled carbon nanotubes (MWCNTs). Their test results showed that the BN modification was improved wear resistance and reduced both working temperatures and friction coefficient [23]. Yu et al. investigated the tribological performance of epoxy coatings modified with functionalized BN particles having cubic and hexagonal geometric shapes. They reported that the cubic-shaped BN modification resulted in better wear resistance thanks to the superior hardness properties, whereas the hexagonal counterpart showed better friction reducing performance due to its crystalline structure [17]. To date, there has been no scientific evidence on the tribological properties of BN doped nanocomposites under harsh conditions such as acidic aqueous solutions that are active on wear behavior and friction coefficiency.

This study investigates the influences of acidic conditions on both mechanical performance and wear behaviors of epoxy polymer composite material modified with nano boron nitride (BN) nanoplatelets using the tensile and ball-on-disk wear tests. The neat Epoxy samples and BN/Epoxy nanocomposites are immersed in the sulphuric acid solution at room temperature and kept for 504 hours. The changes in the properties of the composite samples are determined after every 168 hours of immersion and compared with each other. The wear tracks of both neat Epoxy and BN/Epoxy nanocomposites are examined using optical microscopy to identify the acidic aging-induced damage mechanisms.

2. MATERIAL and EXPERIMENTAL METHOD

MGS L160 lamination resin and MGS H160 curing agent are used as the epoxy matrix to produce composites. While undoped epoxy and its hardener are directly mixed and poured into the test molds to manufacture samples, a different stirring process detailed in our previous study [13] is designed for the 0.5% by weight boron nitride nanoplatelet doped epoxy tensile and wear samples. The homogeneous distribution of BN nanoparticles in Epoxy is achieved with the help of a tipped sonicator. The amount of BN nanoparticles is taken as 0.5% of the total weight of the epoxy system. The additive rate is defined as 0.5%, which is the optimum ratio that improves the friction and wear properties of the epoxy polymer in the literature [22]. The BN doped epoxy solution is blended with a sonicator for 20 minutes at 5-minute intervals. Then, the solution is mixed in a mechanical stirrer with a 25% curing agent. The BN reinforced epoxy mixture is poured into the test molds is initially cured at room temperature for 24 hours, then at 80 °C for 15 hours (Figure 1).

Tensile tests of samples are carried out with crosshead speed at 2 mm/min following the ASTM D638 standard [20]. The tests are repeated five times with one-week (168 hours) intervals after aging until 504 hours. On the other hand, the wear tests are carried out in the ball-on-disc wear tester according to the ASTM G99 standard (Figure 2) [13]. Chrome steel balls, which have a diameter of 6 mm and a propertied 62 HRC hardness, are utilized as an abrasive material in the wear device. The wear test parameters are determined according to the preliminary test results performed in our previous tribological studies on epoxy-based polymer composites [11–13]. The tests are carried out under a 10 N load at room temperature, at 0.8 m/s speed, for 3000 seconds. After the tests, the weight loss of the samples is determined by measuring with a precision balance. Equation 1 has been used in the wear rate calculation [24].

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

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

10 wt% H_2SO_4 solution is prepared by adding distilled water to examine the effects of an acidic environment on the mechanical and wear properties of neat Epoxy and nano BN reinforced epoxy composites. It can be noted that H_2SO_4 is one of the most destructive environmental conditions [25], especially for polymer materials; therefore, it was chosen as an acidic solution to investigate the aging performance of the epoxy-based composites under acidic attack in this study. In the literature, epoxy composites exposed to 5% H_2SO_4 solution aged up to six weeks [26]. In this study, the concentration of the solution is determined as 10 wt% (twice the average concentration in literature) to simulate the accelerated aging conditions, and the aging period was determined as three weeks (504 hours) since the materials deteriorated significantly during the one-month aging period in the preliminary studies. Tensile and wear test samples prepared following the standards are aged in acid solution. Tests are carried out primarily in the dry environment under the conditions specified in the standards. After the samples are aged 168, 336, and 504 hours in an acidic environment, mechanical and wear tests are repeated five times, and the data are compared with the control samples (dry samples).

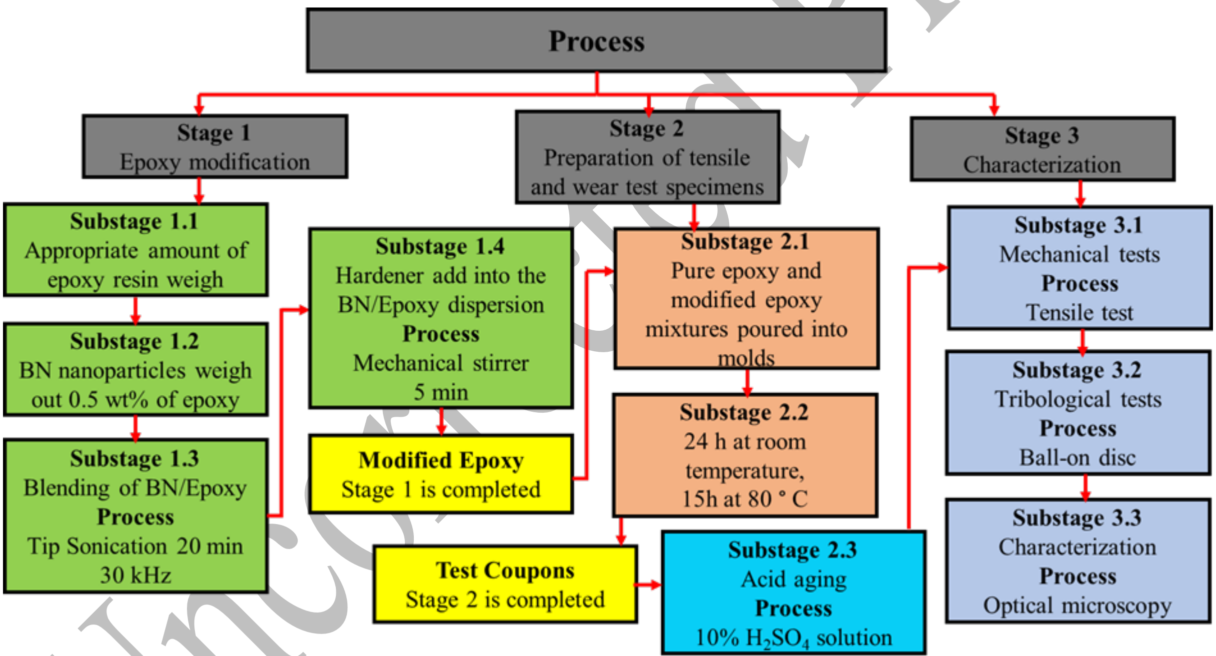

Figure 1. Production and testing methodology of BN reinforced epoxy composites [27]

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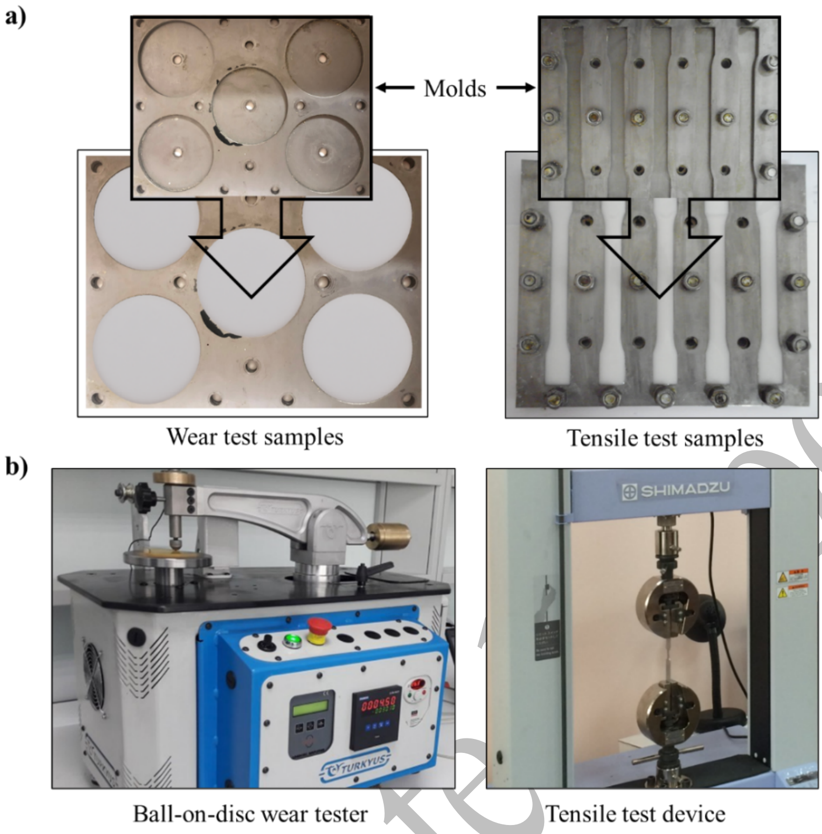

Figure 2. a) Tensile-wear test molds and samples, **b)** Ball-on-disc wear tester and tensile test device

3. RESULTS and DISCUSSION

3.1. Mechanical Characterization

Tensile strength-strain curves obtained from tensile tests of composites are given in Figure 3. As expected, an increase in the strength and strain of nanocomposite samples is observed compared to unmodified epoxy samples. While unmodified Epoxy has a tensile strength of 65±1.95 MPa under normal conditions, the strength increased to 78±2.34 MPa with a 0.5% nano BN addition, and a 17% improvement is achieved. Moreover, the toughness of neat Epoxy is enhanced by 27% with the reinforcement of 0.5 wt.% BN nanoparticles. The homogeneous dispersion of BN nanoplatelets created broad bonding surfaces in the epoxy matrix and, through sufficient wetting and strong adhesion between Epoxy and the filler, improved the mechanical properties. BN nanoplatelets form radical groups in the epoxy polymer with their hexagonal structures in plates, creating adhesion surfaces with strong bonds through these groups [28]. BN nanoplatelets can also bridge the micro-cracks during loading and increase the load-bearing capacity of the epoxy resin during tensile tests. Correspondingly strain at the break of epoxy increase with BN modification by approximately 5% [20]. Furthermore, the slope of the curves before arriving at the maximum tensile strength is relevant to the rigidity modulus. Nanocomposite samples have a higher rigidity modulus than unmodified samples, both nonaged and all aging cases. These findings demonstrate that the nano BN doping to the epoxy matrix enhances its mechanical performance.

Figure 4 represents the acid solutions' effect on the tensile performance of unmodified Epoxy, and nano BN reinforced epoxy composites versus immersion times. Figure 4.a, b, and c show tensile strength, strain at

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break, and toughness values of neat Epoxy and BN modified epoxy nanocomposites, respectively. It can be seen from Figure 4.a that the tensile strength, strain, and toughness of the composite samples have continuously decreased with the drastic effect of acidic aging. Although the acidic conditions resulted in the degradation of mechanical properties of all composites, the tensile performance of BN modified epoxy nanocomposite materials after aging is better than its unmodified counterpart. At the end of the 168, 336, and 504 hours of immersion, a drop of 15%, 35%, and 50% have occurred in the tensile strength values of neat epoxy samples aged in the acidic environment, respectively. The tensile strength of BN epoxy nanocomposites also diminished by 15%, 32%, and 49% at the end of the 168, 336, and 504 hours of immersion, respectively. Toughness values of both neat Epoxy and BN nanocomposites reduced at approximately similar rates to the strength values until the end of 306 hours. After 306 hours of immersion, the toughnesses of the pure Epoxy and BN/Epoxy samples are decreased by about 57% and 30%, respectively. However, at the end of the 504th hour, BN nanoplatelets started to lose their protective properties, and the toughnesses of unmodified Epoxy and nanocomposite counterparts were reduced by 68% and 65%, respectively. It can be concluded that the BN modification is effective in decelerating the degradation of the epoxy matrix for a specific immersion time. This absorbed solution can deteriorate microstructure by weakness the polymeric chains and leading to oxidation in the chains of epoxy resin, as mentioned in the literature [29,30]. The epoxy matrix can be swelled with the acidic solution absorption, then stress-induced micro-cracks can occur and decreased tensile load-carrying capacity. Additionally, during the corrosive solution-based aging process, the water and corrosive fluids can be lead to the combined action, which causes the matrix expansion and forming of pits. Then blisters can arise and expand by the swelling effect with the growing aging time [31]. On the other hand, the reduction in strain values after acidic solution exposure is related to the decline in the molecular mobility of network chains in the matrix, thus causing embrittlement of the composites [32].

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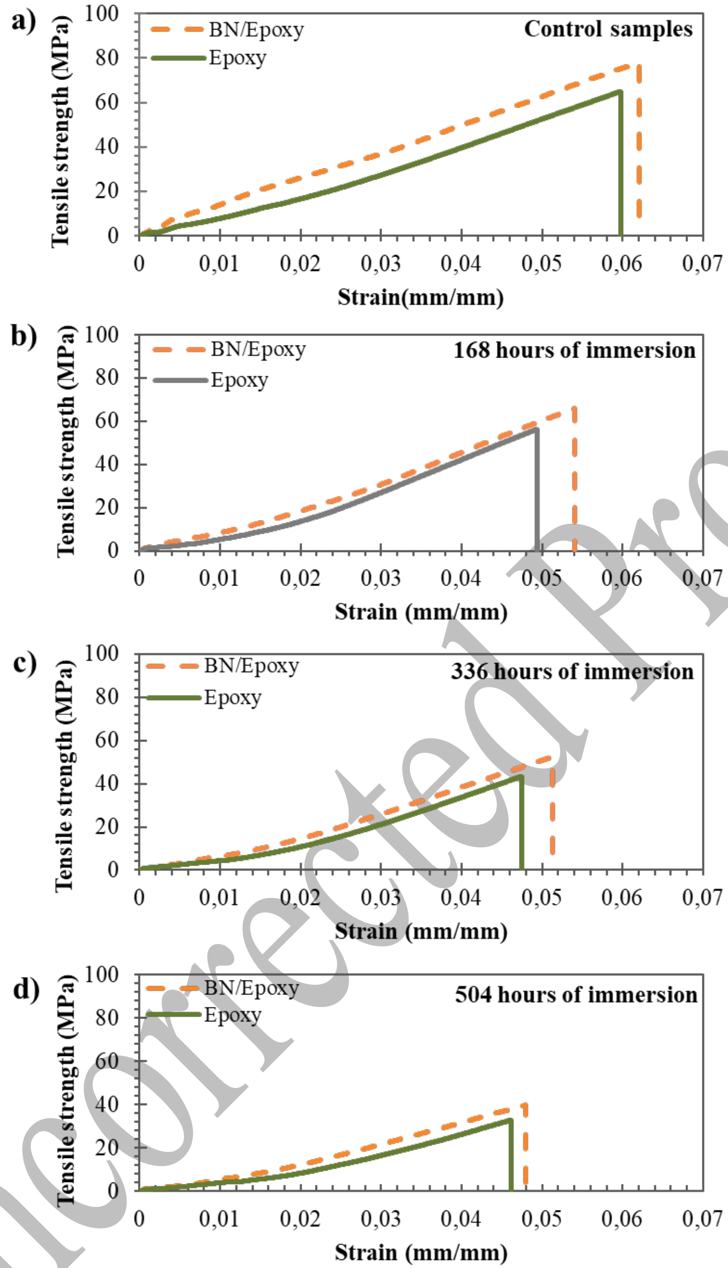

Figure 3. Tensile strength-strain curves of neat Epoxy and BN/Epoxy samples according to increasing immersion times

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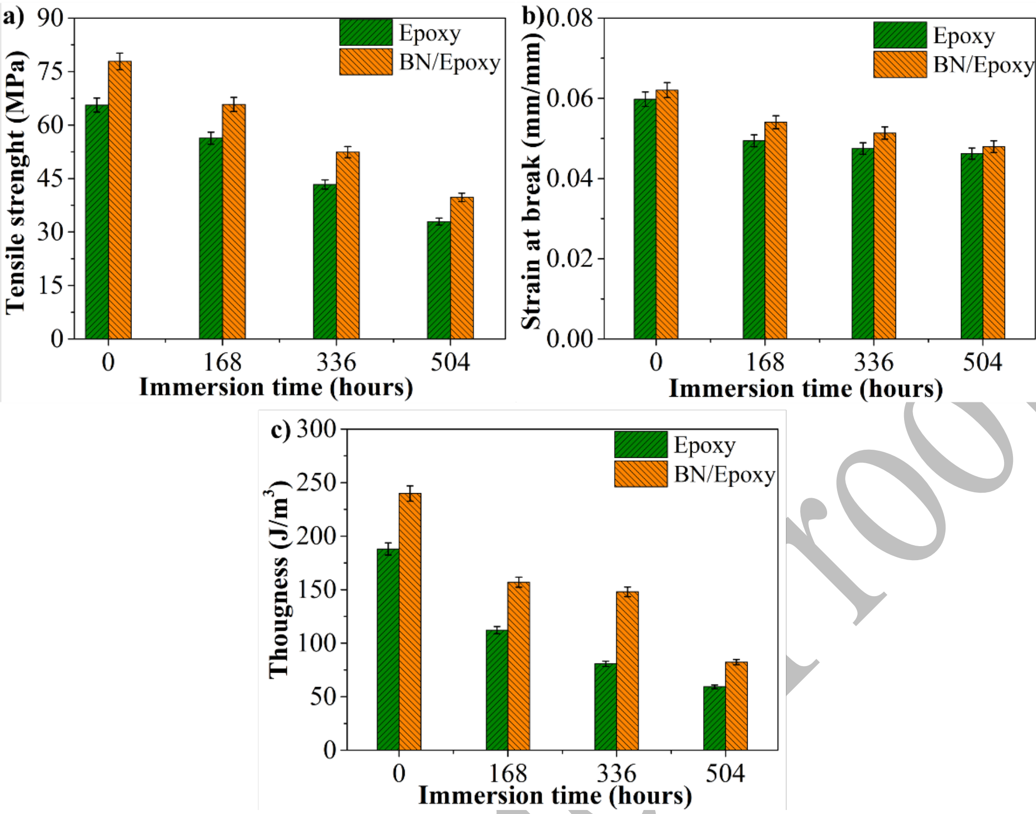

Figure 4. Tensile test results of neat epoxy and BN/Epoxy nanocomposites according to immersion times **a)** tensile strength **b)** strain at break **c)** toughness (error bars represent the standard deviation)

3.2. Tribological Characterization

Figure 5 shows the friction coefficient variation at 0.8 m/s of speed under 10 N loading depending on the sliding distance. It is found that the friction coefficient raised in all composites with the rise in the sliding distance. It is known that the neat epoxy sample has high polymer strength and a high friction coefficient at room temperature. While the friction coefficient of neat Epoxy (Figure 5.a) under dry conditions was 0.36, it decreased to 0.25 with BN nano reinforcement (Figure 5.b). The nano BN modification was influential in stabilizing the friction coefficient curve and reducing the friction coefficient and wear rate. The low shear strength, high lubrication, and load-bearing capacities of BN nanoplatelets play a vital role in reducing the friction coefficient and increasing the wear resistance of BN-reinforced epoxy nanocomposites. Additionally, one of the most critical factors is the bond strength between epoxy and BN nanoplatelets [33]. On the other hand, during the wear tests, the contact surface heat due to friction affects the mechanical properties and the tribological performance [23]. BN fillers with high thermal conductivity create thermal conductive networks and paths within the polymer, raising the composites' thermal conductivity [34]. While the rise in temperature causes softening and an increase in the contact area and friction coefficient, BN reinforcement improves the thermal conductivity of Epoxy and prevents the composites' softening and degradation by reducing the interface temperature [35]. BN additive enhances thermal conductivity and provides heat removal from the contact area [23]. Thus, the friction coefficient diminishes with the temperature at the contact point. Besides, boron nitride has a solid lubricating feature [36] and has a crystal structure similar to carbon. This lubricating property plays an influential role in reducing the friction coefficient of Epoxy, the weight loss, and the wear rate, resulting in improved tribological performance.

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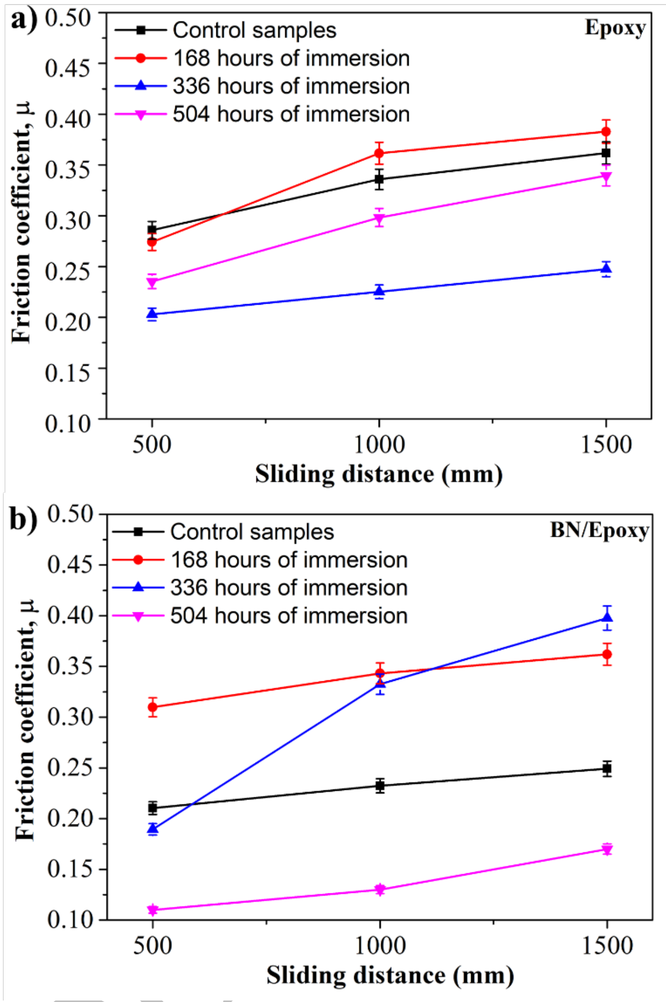

Figure 5. Variation of friction coefficient of **a)** neat Epoxy and **b)** BN/Epoxy nanocomposites depending on the sliding distance

The friction coefficient (Figure 6.a) and wear rate (Figure 6.b) of neat Epoxy and BN/Epoxy nanocomposites aged in acid solution increased after the immersion of 168 hours. It is known that the friction coefficient mainly depends on the surface roughness properties and the interface temperature at the contact area, whereas the wear rate is relevant to the counterface type and molecular structure of the polymer [37]. Based on the literature, the increase of friction coefficient and wear rate after the immersion of 168 hours can be associated with the increasing surface roughness on contact surfaces of samples. At the end of the 504 hours of immersion, both friction coefficient and wear rate values of Epoxy and BN/Epoxy samples are seriously reduced compared to the dry environment values. There is a 5.5% and 20% reduction in friction coefficient and wear rate of neat Epoxy, respectively. In the case of 504 hours immersion, the friction coefficient and wear rate of nanocomposites declined by 32% and 34%, respectively. It is thought that the corrosion layer that occurs on the surface of the composites with increasing immersion time acts as a protective layer, declining the contact temperature, thus improving the friction and wear properties of the composites. In addition to the corrosive layer, the solid lubricant property and high thermal conductivity of hexagonal boron nitride particles further reduced both friction and wear rate of BN/Epoxy composites compared to neat Epoxy.

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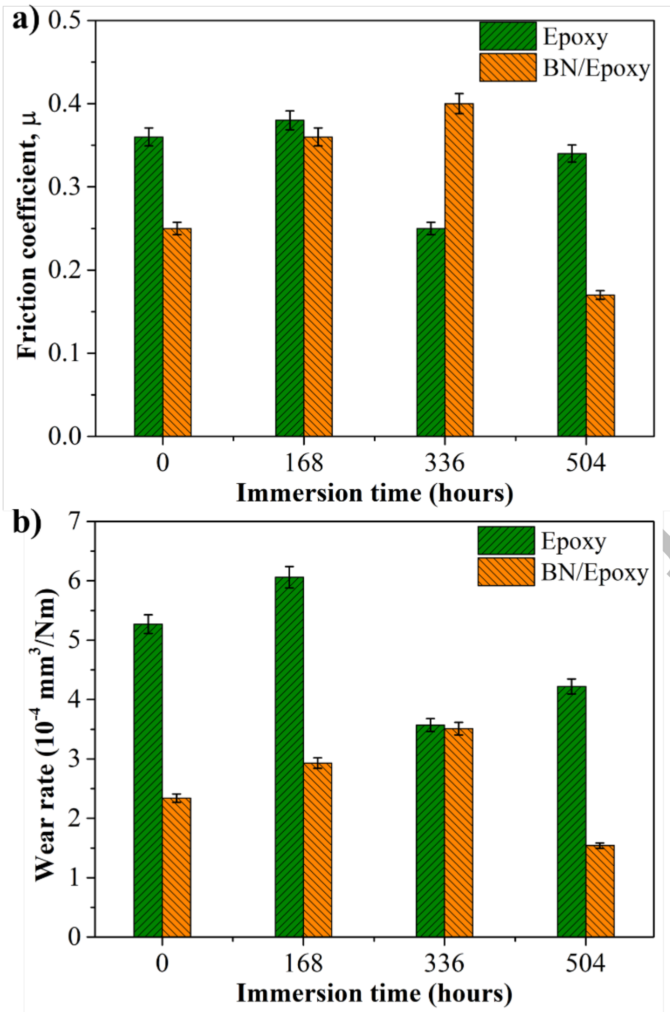

Figure 6. Dry sliding wear test results of neat Epoxy and BN/Epoxy nanocomposites according to immersion times **a)** friction coefficient **b)** wear rate (error bars represent the standard deviation)

To further investigate the tribological properties and the effect of acidic aging, the worn-out surface morphologies of samples are analyzed via microscopy, as given in Figure 7. The main wear mechanisms of the reference and nano-doped samples under dry conditions can be regarded as abrasive wear and micro-ploughing. It can be clearly seen in Figure 7.a that during the wear test, long micro-cracks, deep and wide pits occurred in the neat epoxy samples, while shallow pits, short and tiny micro-cracks formed in the BN/Epoxy nanocomposites (Figure 7.b). The wear track is also more expansive compared to BN/Epoxy nanocomposites. In the case of BN doped, fewer cracks and pits ensue in the samples thanks to its higher hardness and load-bearing capacities. After 504 hours of acidic aging, the primary wear mechanism of both samples turned into plastic deformation, adhesive, and fatigue wear (Fig 7.c-d). The change in wear mechanism after aging is attributed to the corrosive layer formed on the contact surface. This corrosion layer has a lubricating effect on the contacted surfaces, reducing the friction coefficient and the wear rate. As the sliding of the adhesive ball on the contact surface continues, corrosion products are removed from the surface by both fatigue wear and adhesive wear. In addition, the narrow wear track, as seen in Figure 7.d, shows that the BN nanoplatelets with high thermal conductivity and their lubricating behavior are decreased further the wear of nanocomposite samples. Also, the

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abrasive and adhesive wear in neat Epoxy manifested a severe degree, whereas in BN/Epoxy nanocomposites was mild.

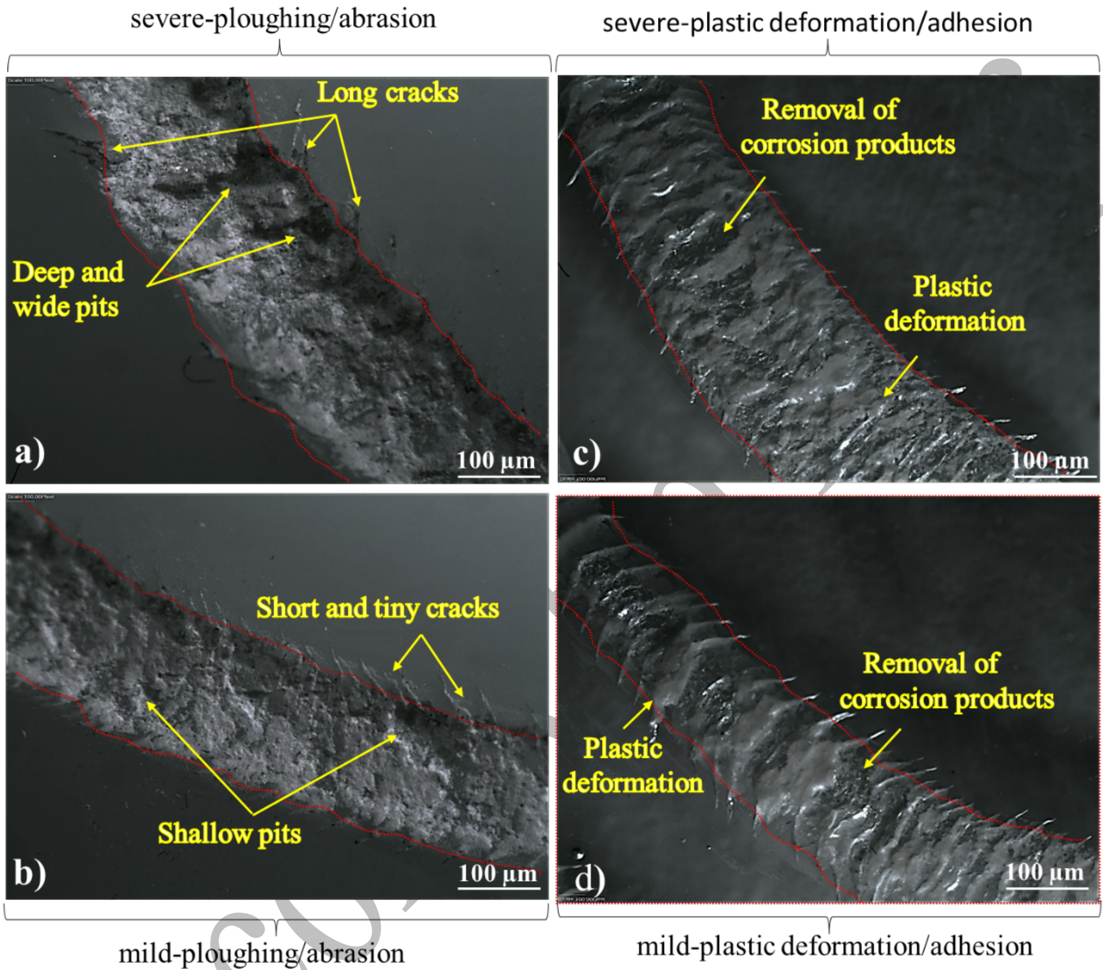

Figure 7. Worn-out surface morphologies of epoxy samples **a)** neat Epoxy before immersion **b)** BN/Epoxy nanocomposites before immersion **c)** neat Epoxy after acidic solution exposure for 504 hours **d)** BN/Epoxy nanocomposites after acidic solution exposure for 504 hours

On the basis of the experimental results, a rational wear mechanism is proposed for shedding light on the effect of acidic aging on the tribological behavior of neat Epoxy and BN reinforced Epoxy nanocomposites, as illustrated in Figure 8. The wear mechanism of the neat Epoxy has mainly occurred as micro-ploughing and abrasive wear, while it is raised in BN/Epoxy nanocomposites as plastic deformation, adhesive, and fatigue wear. It can be revealed that the BN nanoplatelets enhanced the wear resistance by prohibiting the crack propagation in the resin and close contact between the nanocomposite samples and the steel ball. Besides, the high thermal conductivity of the BN reinforcing is effective for the removal of the friction heat on the contact surface by homogenous and quick heat dissipation. On the other hand, corrosion products can accumulate on the contact surface and form a corrosive layer during the acidic exposure, which shows a lubrication effect, decreasing the

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contact surface temperature and friction coefficient. Firstly, this corrosive layer is removed from the surface, then the wear progress to the thickness of the polymer, and a lower wear depth is observed in the aged samples compared with their unaged parts, which results in a low wear rate and high wear resistance. Moreover, BN nanoplatelets can be extended the diffusion path of the acidic solution and reduce the absorbed solution amount, hence decelerating the epoxy resin's deterioration. Consequently, the synergistic effect of BN nanoplatelets and acidic solution enhances the tribological performance of epoxy under acidic conditions after 504 hours of immersion.

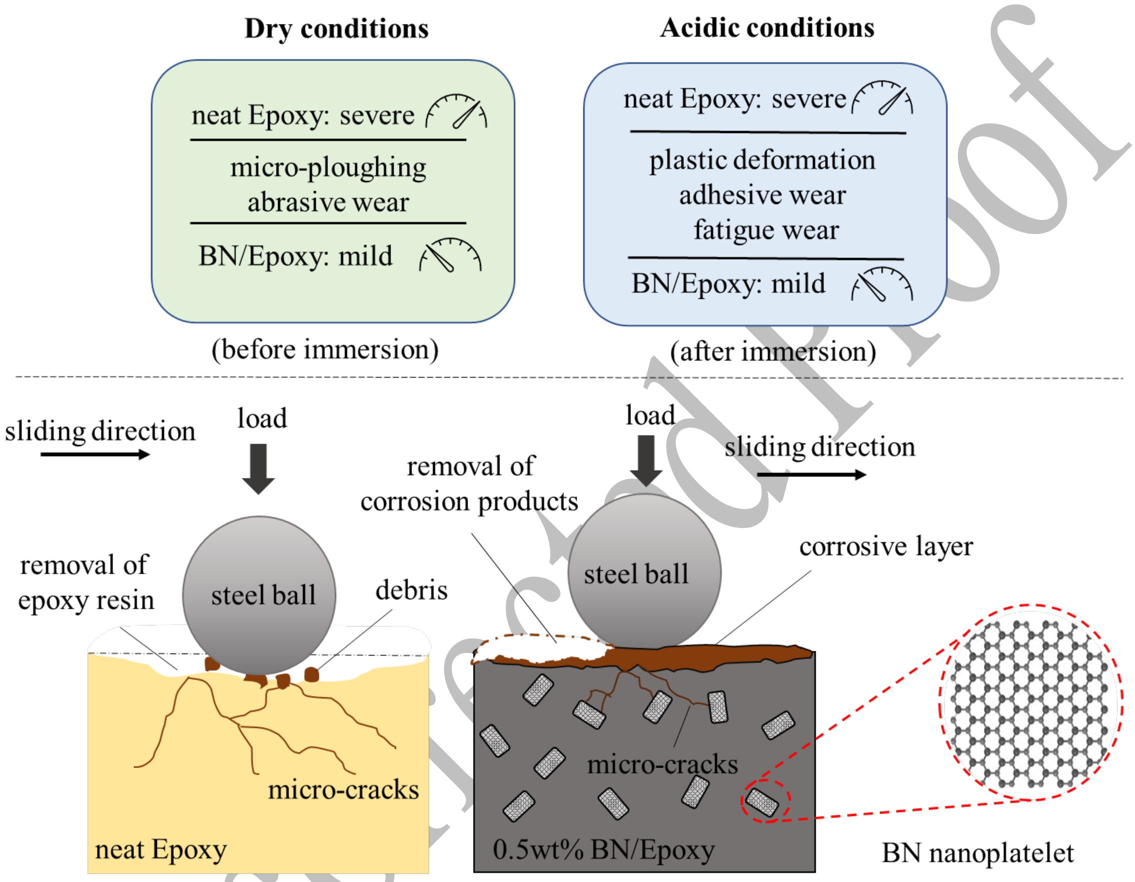

Figure 8. Schematic illustration showing the wear mechanism of neat Epoxy and BN/Epoxy nanocomposites under dry and acidic conditions

CONCLUSION

The present research aimed to report the effect of BN reinforcement on the mechanical and tribological performance of epoxy resin under dry ambient and acidic conditions. Compared to the unreinforced Epoxy, BN modified epoxy nanocomposites showed higher mechanical performance and wear resistance thanks to higher hardness, load-bearing capacity, thermal conductivity, and lubricity of BN nanoplatelets. The acidic aging reduced the mechanical performance of both neat Epoxy and BN/Epoxy nanocomposites through deterioration of the microstructure and swelling of the resin. However, BN modified Epoxy showed better performance than the neat Epoxy because of the strong interaction and mechanical interlock between the Epoxy and BN nanoplatelets. In contrast to the mechanical performance, the tribological performance of neat Epoxy samples with and without BN addition is improved by the acidic solution after the immersion of 504 hours. The acidic solution can be created a corrosive protective layer on the contact surface of the samples, which removed the friction-induced

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heat quickly, acted as a lubricant, and reduced the friction coefficient and wear rate. In addition to the lubricity of the corrosive layer on the surface, BN nanoplatelets further decreased the friction coefficient and wear rate of the composited compared with the neat Epoxy with their lubrication properties. Finally, a correlation between mechanical and tribological properties was found, which BN reinforcing improved the performance of neat Epoxy under dry and acidic conditions.

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