Enhancement of Underwater Tribological Properties of Hybrid PTFE/Nomex Fabric/Epoxy Resin Multilayer Composites by Mixed Graphite and MoS$_2$ Fillers

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ABSTRACT: This work studied the effect of mixed graphite and MoS$_2$ fillers on tribological properties of hybrid polytetrafluoroethylene (PTFE)/Nomex fabric/epoxy multilayer composites under water lubrication. A ring-on-block tribometer was used to perform the tribological test under dry sliding and water lubrication conditions. The worn surface was analyzed by scanning electron microscopy and optical microscopy. The results showed mixed fillers with 2.5 wt % graphite and 5 wt % MoS$_2$ had the best underwater tribological properties with the lowest friction coefficient (COF) of 0.067 and the lowest wear amount of 1.7 mg. Mixed fillers optimize epoxy resin properties, thereby increasing shore hardness, reducing water absorption, and improving wear resistance. This study also explained the reasons of the wear amount was higher in water than in dry sliding.

1. INTRODUCTION

Hybrid fabric-reinforced resin matrix composites are the self-lubricated materials that have excellent tribological and mechanical properties, which are widely used in aviation, aerospace, shipbuilding, and so on. In shipbuilding, fabric resin composites can be used to make water-lubricated stern bearings in power systems. In hybrid PTFE/Nomex fabrics, the PTFE fibers exhibit excellent self-lubrication, and Nomex fibers have high binding strength with adhesive resin. Because the adhesion between PTFE fibers and resins is poor, and PTFE fibers are easily worn, it is necessary to improve the tribological properties of hybrid PTFE/Nomex fabric epoxy resin multilayer composites.

Various fillers are used to improve the tribological properties of hybrid fabric resin composites. Professor Zhang and his coworkers at the Lanzhou Institute of Chemical Physics have performed a significant amount of work on the effect of different fillers on fabric composites. Ren studied the tribological properties of hybrid PTFE/Nomex fabric/phenolic composites and used WS$_2$, MWCNTs-GO hybrids fillers, and air-plasma treatment to improve the dry sliding antiwear property. Zhang used ZnO Nanoparticles, carbon nanotubes, TiO$_2$ and SiO$_2$ nanoparticles, and CuS nanoparticles to improve the tribological properties of PTFE/Kevlar fabric/phenolic composites. Yang et al. used Mo$_2$C, Polyfluoro Wax, ZrB$_2$, and hexagonal boron nitride fillers as well as the air-plasma treatment to improve the high-temperature tribological behaviors of hybrid PTFE/Nomex fabric/phenolic composites. Su et al. used ZnO, nano-TiO$_2$, Synfluo 180XF wax, and nano-SiO$_2$ fillers to improve the friction and wear of hybrid glass/PTFE fabric/phenolic resin composites. The results showed an improvement of the wear resistance and the friction coefficient (COF) reduction of the fabric composites under dry sliding. Yuan et al. used amino silane and polydopamine to improve the tribological and mechanical properties of hybrid fabric composites by improving the interfacial adhesion between hybrid Nomex/PTFE fabric surfaces and resin materials. Some combined fillers are also used to improve the tribological behavior of fabric composites. Li et al. studied the influence of combining fillers with nano-SiC and submicron-WS$_2$ as well as combining nano-Si$_3$N$_4$ and submicron-WS$_2$ under dry sliding conditions; the results showed that the combined fillers exhibited the best comprehensive performance compared to the single fillers.

Some studies are devoted to using MoS$_2$ and graphite fillers to improve the tribological properties of the fabric-reinforced resin matrix composite. Zhang et al. studied the effect of MoS$_2$ and graphite fillers in fabric/phenolic resin composites, the results showed that graphite fillers were effective in reducing the wear resistance. Li et al. studied the...
wear reduction of graphite and MoS$_2$ in epoxy composite coatings for switch slide baseplates, and the results showed that the graphite reduced the friction coefficient and wear rate, MoS$_2$ reduced the wear rate effectively but had little effect on friction coefficient reducing, and the mixed graphite and MoS$_2$ had the best effect on reducing the COF and wear amount.

However, Zhang$^{25}$ had not referred to the filler effect under water lubrication and the effect of combined fillers. Moreover, different from the research of Li,$^{26}$ the properties of epoxy composite coatings on the A36 steel were not similar as the PTFE/Nomex fabric/epoxy resin multilayer composites; adding graphite and MoS$_2$ fillers exhibit different performance. Additionally, the underwater properties of the hybrid fabric epoxy resin composite were important. The two above studies did not relate to the underwater tribological properties of PTFE/Nomex fabric epoxy resin multilayer composites.

The main effect of fillers is to reconcile the adhesive resin performance and lubricates the surface of materials. Fillers can improve the comprehensive performance. However, there are few studies related to the influence of fillers on comprehensive performance under water lubrication. In this study, first, multilayer hybrid fiber—resin composites are used; second, the underwater properties are studied; third, the influence of combined fillers and its optimum ratio was studied. Finally, the filler influence mechanism on the underwater multilayer fabric resin composite was revealed.

### 2. RESULTS AND DISCUSSION

#### 2.1. Tribological Test Results under Different Fillers.

Figure 1 shows that Composite C with single 5 wt % graphite fillers has the lowest COF, Composite A with single 5 wt % MoS$_2$ has the highest COF, and Composite B with mixed 2.5 wt % graphite and 2.5 wt % MoS$_2$ fillers has the middle COF. Graphite fillers improve thermal stability, which is conductive to COF reduction of Composite B and Composite C. In Figure 1, the COF range is smooth under dry sliding, but all the COF values have a fluctuation under water lubrication. The average COF in Figure 2a is the average value in Figure 1. The average COF is lower under water lubrication. The lubrication and cooling effect of water causes a lower COF.

In Figure 1b, under water dripping lubrication, only the bottom end of the metal ring is immersed in water. Heat is generated during the friction test. Less water is not enough to cool the metal ring well. Specifically, the temperature of the metal ring also gradually increases during the friction test. Furthermore, wear abrasive particles are gradually taken away by water. Wear particles are easily oxidized and deteriorated into hardened particles under humid and hot conditions, then adhere to the friction surface of the metal ring. Surface roughness of the metal ring increases, and the abrasive particles scatter in water. Consequently, the instability of the lubrication state formed, causing a large fluctuation of underwater COF.

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underwater COF. Composite C has the lowest COF under the help of graphite fillers. In Figure 2b, Composite B with mixed fillers has the lowest wear amount both under dry and water-lubricated conditions. It means that the mixed fillers of graphite and MoS$_2$ exhibit excellent comprehensive performance. Under dry sliding, abrasive particles can modify the wear surface and provide secondary lubrication, reducing the wear amount. Under water lubrication, dripping water gradually takes the abrasive particles away. Additionally, the hard oxidized abrasive particles adhering to the metal ring friction surface also aggravate the wear amount. Therefore, water-lubricated conditions are good to reduce the average COF but bad to improve wear resistance. Thus, the underwater wear and tear requires significant attention.

In Figure 3a, it is observed that shore hardness decreased after soaking for 24 h because water absorption causes hardness to reduce. The reduced hardness is a significant reason for increased underwater wear amount. Composite B with the lowest underwater wear amount has the highest shore hardness after soaking. In Figure 3b, Composite B with the highest hardness exhibits the lowest water absorption. Hence, higher hardness, lower water absorption, and lower underwater wear amount are exhibited. Thus, Composite B has the highest shore hardness, lowest water absorption, and lowest underwater wear amount. Finally, the mixed fillers improve underwater wear resistance.

### 2.2 Tribological Test Results under Different Mixed Filler Contents

As the results shown in Figures 1–3, Composite B with 2.5 wt % graphite and 2.5 wt % MoS$_2$ fillers exhibits the best comprehensive performance. Because graphite fillers help reduce COF, MoS$_2$ fillers help improve wear resistance, and the combination of the two has best comprehensive performance. Compared to dry sliding, the wear amount is larger, and the COF was lower under water lubrication, as shown in Figure 2. Therefore, excellent wear resistance is more important underwater. Therefore, to explore the optimal ratio of mixed fillers to improve the wear resistance, the graphite content is fixed at 2.5 wt %, and the content of MoS$_2$ is adjusted in the range of 0–10 wt %. Figure 4 shows the result of COF and wear amount under different mixed filler ratios. The COF and wear amount is first decreased and then increased with the increasing MoS$_2$ contents both under dry sliding and water lubrication. The fabric sample with the mixed filler of 2.5 wt % graphite and 5 wt % MoS$_2$ has the lowest underwater COF of 0.067 and the lowest underwater wear amount of 1.7 mg. Below, the fabric sample with 2.5 wt % graphite and 5 wt % MoS$_2$ is referred to Composite D in this study.

Figure 5 shows the shore hardness and water absorption under different mixed filler contents. In Figure 5a, b, Composite D has the highest shore hardness and the lowest water absorption under different mixed filler contents. As explained in Figure 3, high shore hardness causes little water absorption, improving underwater wear resistance. The suitable content of mixed fillers increases the hardness after curing, improves interface bonding force between layers, and makes water penetration between layers more difficult; as a result, water absorption is reduced. Finally, Composite D exhibits the lowest underwater wear amount.

Figure 6 shows the photographs of the metal ring after wear. In Figure 6a, it can be seen that there are slight wear marks and
Figure 7. 3D profile surface of fabric samples, (a1) not-wear, (a2) dry sliding, and (a3) water-lubricated of Composite B; (b1) not-wear, (b2) dry sliding, and (b3) water-lubricated of Composite C; (c1) not-wear, (c2) dry sliding, and (c3) water-lubricated of Composite D.

Figure 8. Microscope images under dry sliding and water lubrication: (a, b) composite A; (c, d) composite B; (e, f) composite C; (g, h) Composite D.
thin transfer film on the metal ring; hence, the wear amount is low under dry sliding. In Figure 6b, it can be observed that because of less water lubrication, the hot and wet metal ring move against the fabric composite. Lubricating water gradually becomes turbid because of the increasing abrasive particles under hot and humid tribological test conditions. The metal ring surface is oxidized under humid and hot conditions. Abrasive particles are taken out and bonds to the metal ring surface, and lubricating water is not conducive to form transfer film; hence, the wear amount is large under water lubrication.

2.3. Laster Microscope Observations of the Working Surface. In Figure 7a1−c1, Composites B, C, and D have the surface roughness (Ra) of 4.5, 3.8, and 4.3 μm before wear, respectively. Their Ra has little difference before wear because of the same processing technology. In Figure 7a2−c2, compared to the Ra before wear, the Ra of Composites B and D is reduced to 2.0 and 1.3 μm under dry sliding, respectively, but the Ra of Composite C is increased to 4.1 μm. This is mainly because of the severe wear of PTFE fibers of Composite C.

Under water lubrication in Figure 7a3−c3, the Ra of Composite B and D continues to decrease to 1.7 and 1.2 μm, but the Ra of Composite C continues to increase to 7.6 μm. Because an unreasonable graphite content breaks the resin matrix bonding, fails to improve interface adhesion between resin and fabrics, results in increased water absorption, and improves the underwater wear amount, consequently, the severely worn resin as well as the warped Nomex fibers increase Ra. Composite D has the lowest Ra after wear because a reasonable content of mixed fillers in Composite D improves interface bonding force between the resin matrix and the fiber fabric, reducing the porosity and permeability; after that, water absorption is reduced, and surface hardness is increased, thereby, the friction surface becomes flatter after wear, reducing Ra.

2.4. Microscope and SEM Images of the Wear Surface. Figure 8a, c, e, and g shows the microscope images under dry sliding, Figure 8b, d, f, and h shows the images under water lubrication. Under dry sliding, rolling deformation appears on the wear surface, the abrasive particles adhere to
the wear surface, and the surface becomes flatter. Also, under abrasive particle lubrication, the wear amount is low under dry sliding. Under water lubrication, water takes wear particles away gradually, and lubrication and surface-modified effect of abrasive particles are disappeared. Clear wear marks are observed on the surface.

In Figure 8d, h, the working surface of Composites B and D has high hardness, Nomex fibers are strongly combined with adhesive resin, PTFE fibers severely wear. The mixed graphite and MoS$_2$ fillers improve the bonding force of fibers to resin. In Figure 8b, Nomex fibers are exposed in the surface with PTFE fibers wearing deeply. In Figure 8f, Composite C has the lowest hardness and highest water absorption. After wear, PTFE fibers wear severely, caused by Nomex fibers exposed on the surface mostly. The PTFE content reduces much and the Nomex content increases greatly. The wear amount of Composite C is the highest underwater. Hence, the hardness working surface of Composites B and D reduces the underwater wear amount, but Composite C with low hardness improves the wear of PTFE fibers and resin. Finally, mixed fillers improve underwater tribological properties via improving the hardness and bonding force of the fabric sample.

Figure 9 shows the SEM images of Composite D with the mixed fillers and Composite C with the single graphite fillers. Figure 9a, b show the holes on working surface, holes appear at the fiber–resin interface caused by the overflow of air bubbles during the curing process. The holes in the fabric sample are the significant factor affecting water absorption. Water penetrates through the small holes, and Nomex fibers absorb the water infiltrated by the small holes, reducing its own strength. Resin and the fibers are more closely combined with the help of reasonable content of mixed fillers, which helps reduce the holes. As a result, water penetrates the inside of the fabric sample with more difficulty, preventing the Nomex fibers from losing strength after absorbing water. Hence, the shore hardness of Composite D is stable after soaking.

Figure 9c, d shows the surface of Composite D and Composite C under dry sliding. Abrasion marks caused by abrasive particles appear on the surface. Abrasion marks of Composite C are much more than Composite D under dry friction, illustrating that Composite D has better properties. Under water lubrication shown in Figure 9e, f, Composite D has excellent wear resistance with the help of mixed fillers. PTFE fibers are tightly bound in epoxy resin matrix with the help of mixed fillers. As shown in Figure 9g, epoxy resin of Composite C shows serious wear, Nomex fibers peel off, PTFE fibers wear severely, and fillers adhere to the friction surface or form abrasive particles under severe wear. Figure 9h shows a peeled Nomex fiber because of weakened adhesion between fabric and resin. Figure 9i shows graphite particles on the working surface after wear. The severe wear of Composite C is due to the deterioration of cured epoxy resin properties by the graphite filler.

3. CONCLUSIONS

This study uses graphite and MoS$_2$ fillers to enhance underwater tribological properties of hybrid PTFE/Nomex fabric/epoxy resin composites. The influence of single and mixed fillers as well as the content of mixed filler are studied; the result shows that combined fillers have better effect in improving tribological properties, and the mixed fillers with 2.5 wt % graphite and 5 wt % MoS$_2$ have the best underwater performance. Other conclusions are as follows:

1. Compared with the average COF, the maximum fluctuation amount of COF was 0.02 under dry sliding, but was 0.12 under water lubrication. The COF is stable under dry sliding but fluctuates under water lubrication because water contains abrasive particles, and some oxidized particles stick to the metal ring, which causes an unstable friction state between the metal ring and the fabric sample.

2. The fabric sample has a low COF and high wear amount underwater because the cooling and lubricating effect of water can reduce the COF. However, water also can take wear particles away that improve the wear amount.

3. The fabric sample with combined fillers has the best comprehensive tribological properties because the combined filler is helpful to improve interface bonding force and finally enhance tribological properties.

4. EXPERIMENTAL SECTION

4.1. Materials. The hybrid PTFE/Nomex fabrics were woven from PTFE fibers (density: 2.2 g/cm$^3$; elongation: 50%) and Nomex fibers (density: 1.36 g/cm$^3$; elongation: 32%), as shown in Figure 10a, b, and the fibers were produced by DuPont. PTFE and Nomex fibers were woven into hybrid PTFE/Nomex twill fabrics on the weaving machine (SXACT-C), as shown in Figure 10b. Figure 10c shows the weaving method of the hybrid PTFE/Nomex twill fabric. On one side, a PTFE fiber presses three Nomex fibers; on the other side, one Nomex fiber presses three PTFE fibers. As a result, one side of
the twill fabric was rich in PTFE fibers with 75%, and the other side was rich in Nomex fibers with 75%. The fillers were commercially available, as shown in Figure 10d, and the performance parameter of fillers is listed in Table 1. The resin was a thermosetting epoxy resin (epoxy value: 10d, and the performance parameter of fillers is listed in Table 1. The resin was a thermosetting epoxy resin (epoxy value: 10d, and the performance parameter of fillers is listed in Table 1.

### Table 1. Properties of Graphite and MoS$_2$

| materials | graphite | MoS$_2$ |
|-----------|----------|---------|
| grain size ($\mu$m) | 13       | 10      |
| purity (%)     | 99.9     | 99.9    |
| specific surface area (m$^2$/g) | 2.63     | 35.46   |
| density (g/m$^3$) | 1.04     | 4.8     |

≥0.85 wt %; volatile: ≤3 wt %; viscosity (25 °C): 2.5 Pa·s), as shown in Figure 10e. There were various types of epoxy resins, and different epoxy resins had various properties; as a result, the same fillers such as graphite and MoS$_2$ may have diametrically opposite effects on different types of epoxy resins. This work studied a certain type of epoxy, and its molecular formula was given as follows:

![Molecular formula of epoxy](image)

#### Figure 10. Schematic diagram of the ring-on-block tribometer.

The tribological test was performed using a ring-on-block tribometer, as shown in Figure 11. The wear area of the fabric sample always submerged in water.

#### Figure 11. Schematic diagram of the ring-on-block tribometer.

20Cr metal ring slide against the fabric sample block under the speed of 200 rpm at the load of 200 N for 1 h both under dry sliding and water lubrication. During the experiment, the same metal ring was used to test all fabric samples. Before each fabric sample was tested, the metal ring was polished with 1000 molybdenum sandpaper. The water lubrication method was to drip distilled water on the metal ring continuously to ensure the wear area of the fabric sample always submerged in water. The friction force was measured using the sensor, and the COF, the ratio of friction force to positive pressure (200 N), could be directly read from the computer running the friction measurement software. The hardness of fabric samples was measured using a shore hardness tester (measure range: 0–100 HA). All tests were performed under laboratory conditions (temperature, 25 °C; relative humidity, ~50%).

The weighing balance used in the study was a precision balance with an accuracy of 0.1 mg. To obtain the accurate weight value, the fabric sample was dried at 50 °C for 1 h before weight measurement. The wear amount was the weight difference before and after the friction test. Before the friction test, the fabric sample was weighed three times. After the friction test, the worn samples were also weighed three times to calculate the wear amount. The weight difference before and after soaking was water absorption. Its measurement method was to weigh 30 × 6 × 7 mm fabric samples three times first, soak them in water for 24 h, take them out, dry at 50 °C for 1 h, and then weigh three times again. The average weight difference of the three times between the first measurement and the second measurement was the water absorption of the fabric sample. The wear surface was analyzed using an optical microscope and a scanning electron microscope (SEM). The 3D profile was observed using a laser microscope.

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