



Effect of relative humidity and full immersion in water on friction, wear and debonding of unidirectional carbon fiber reinforced epoxy under reciprocating sliding



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ABSTRACT

Unidirectional carbon fiber reinforced epoxy was tested in ambient air at three different levels of relative humidity and under full immersion in demineralized water. Reciprocating sliding tests were performed at 23 °C against either stainless steel or alumina balls moving in parallel or anti-parallel direction to the fibers. We demonstrate in this work that humidity and water immersion affect significantly the fiber debonding. Under sliding against stainless steel or alumina at low relative humidity, fiber debonding is more pronounced than at high relative humidity and at water immersion. The wear depth increases with increasing relative humidity when sliding against stainless steel, whereas it remains practically constant against alumina. For all test conditions, the wear depth is larger when tested against stainless steel than against alumina. It was found that the thin moisture film formed at the surface of the stainless steel counter body leads to a higher corrosive risk than water immersion. More precisely, we demonstrate that high humidity leads to the production of oxide debris originated from the stainless steel ball which increases markedly the wear by abrasion. These debris lead to a high fluctuation of the coefficient of friction measured on carbon fiber reinforced epoxy composite sliding against stainless steel at 85% RH, whereas a steady state coefficient of friction is noticed against alumina.

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1. Introduction

Composite polymers are generally more resistant to aqueous and humid exposure than many metals. However, the mechanical strength of composite polymers usually degrades by water and humidity sorption [1,2]. Furthermore, debonding is one of the failure mechanisms in carbon fiber reinforced epoxy which can lead to untimely failure. In a detailed review on carbon fiber/epoxy interfaces, Hughes [3] explained that the weakest spot in debonding is usually the interface between the carbon fiber and the epoxy matrix. Such a debonding may originate from the composite fabrication process where differential thermal expansion stresses may be induced. More precisely, the resin contracts more than the fiber, and shrinkage stresses are induced on polymerization of the

resin [3]. Arnold [4] described swelling effects and residual stresses in different epoxy composites. He concluded that the most significant origin of residual stresses in such composites is the differential shrinkage on curing or cooling. Most polymeric matrix materials shrink to a certain extent on curing, whereas the reinforcements tend to retain a constant volume. The matrix will then have a tendency to shrink onto the reinforcing fibers. This results in a better adhesion, but may also lead to a compressive stress on the fibers, and a tensile stress in the matrix.

Epoxy matrixes absorb atmospheric moisture causing a resin softening, swelling, and a loss of the mechanical performance of composites [5]. Adamson [6] detailed the transport steps of moisture in epoxy matrixes below the glass transition temperature (T_g). He proposed a three stage process in which at first the absorbed moisture occupies the free volume of epoxy, then in a second stage water becomes bound to network sites, and finally a swelling may take place because water enters the densely cross-linked regions. Another study showed that water or humidity sorption leads to

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delamination and void growth in carbon fiber reinforced epoxy [7]. Also, expansion due to water sorption was found to cause surface cracking in carbon fiber reinforced epoxy laminates [8]. Generally, the lowering of T_g due to water or humidity uptake reduces the bonding at the interface between matrix and fibers [9,10]. Akay [11] investigated epoxy immersed in water for 21 days at 70 °C. He recorded a lowering by 17% of the interlaminar shear, 10% of the compressive stress, and 7% of the flexural strength in unidirectional carbon fiber/epoxy laminates associated with a gain of 1.5% in laminate mass due to moisture uptake. Many studies on the effect of moisture on the interlaminar fracture toughness of composites [12] revealed that one of the most significant factors of fiber debonding is the swelling of the matrix which causes compressive stresses at the interface, followed by micro-crack formation and fiber debonding. This is particularly common in epoxy resin composites where the equilibrium water sorption content is higher than in other matrix materials [4].

On the other hand, extensive research showed that the relative humidity (RH) has a strong effect on the tribological behavior of composites. Generally, a high RH lowers the coefficient of friction and the wear rate [13]. Additionally, polytetrafluoroethylene (PTFE) reinforced by carbon fibers, polyimide (PI) reinforced by carbon fibers (CF) and PTFE, PI reinforced by CF and MoS_2 , and polyetheretherketone (PEEK) reinforced by CF and PTFE composites maintain a lower coefficient of friction, and display a much higher wear resistance under sliding immersed in water against stainless steel than under dry sliding [14]. Komai et al. [15] found that water causes a reduction of the interfacial strength in several epoxy composites containing carbon and aramid fibers, especially under fatigue loadings. On the contrary, Walker and Zhi Hu [9] observed that the exposure to water increases the interfacial strength of short fiber interlaminar reinforcement layers in carbon fiber epoxy pre-impregnated composites. They also noticed that the bond strength to polyolefin reinforcement layers improved by exposure to water, possibly by a swelling mechanism [9]. In addition, it was found that water molecules improve the interfacial bond of epoxy reinforced with glass particles or fibers due to a combination of swelling stresses, and a water uptake that increases the matrix ductility due to plasticization [16].

Water can interact with epoxy resin in different ways. Apicella et al. [17] identified three possible modes of interaction: a) adsorption on hydrophilic centers on surfaces defining the void structure of the resin, b) condensation within the void structure, c) sorption and presumably bonding with the polymer network. Adamson [6] proposed that water first enters the void structure of epoxy resins and then migrates into the polymer network, but concluded that water is unable to disrupt the hydrogen bonding among polymer segments. Obviously, water affects mostly the resin and the interface on carbon fiber composites due to the good hydrolytic stability of carbon fibers. Similar results were found by Yamada and Tanaka [18] who studied the wear of various PTFE based composites against stainless steel under boundary lubrication with water. They assumed that the higher wear of the composites under water lubrication was due to the permeation of water molecules to the interface of the composites and the PTFE matrix, which results in the separation of fillers embedded in the PTFE matrix [18]. Lancaster [19] studied the wear behavior of various carbon fiber reinforced polymers sliding against metals in water, aqueous solutions, and organic fluids. He found that the wear of carbon fiber reinforced polymers as well as unfilled polymers under water lubrication is generally greater than under dry conditions. He concluded that the higher wear rate in water could not be attributed to a modification of the counter face by material transfer, because a transfer film on the counter face rubbed under water lubrication was not observed [19].

From the above discussion, it appears that the testing environment, and in particular the RH of ambient air can affect the mechanical and tribological performance of epoxy composite materials, and that these effects can be synergistic or antagonistic depending on the specific materials and properties studied. Plasticization of matrix materials by water can increase the resistance to crack formation, whereas in other cases, water uptake can enhance crack formation. Over the past three decades, the research in this area significantly increased aiming at a more reliable performance and lifetime assessment of composite polymers in different industrial applications. But, how failure processes are affected by environmental conditions under unidirectional or reciprocating sliding is still unclear.

In previous works [20,21], we examined the wear behavior of carbon fiber reinforced epoxy composite based on an in-depth analysis of the worn surfaces with reciprocating sliding in ambient air and with immersion in demineralized water. Immersion in water proved to be harmful to the wear resistance of the composite for sliding against stainless steel [21]. In the present study, environmental conditions of ambient air of different RH and a full immersion in water are considered and their effect on the friction and wear behavior of carbon fiber reinforced epoxy under reciprocating sliding against stainless steel or alumina balls is reported and discussed.

2. Experimental

A unidirectional carbon fiber reinforced epoxy was investigated. Its fabrication process is described in previous work [21]. Test samples of $5 \times 5 \text{ mm}^2$ were cut out of a mother plate of $20 \times 20 \text{ cm}^2$ using a diamond saw. The mother plate has a two layer structure consisting of an epoxy top layer of about $3 \mu\text{m}$ onto a fiber containing bulk material with epoxy as continuous phase [21]. Reciprocating sliding tests against either stainless steel or alumina balls with a diameter of 10 mm were performed in a test rig described earlier [20]. The sliding tests were carried out for 200,000 reciprocating sliding cycles in ambient air of 15, 50 or 85% RH, and immersed in demineralized water at 23 °C.

Reciprocating sliding tests were performed by putting the samples in an RH controlled tribotest chamber for about 15 min prior to the start of each test to stabilize the sorption onto the surface and subsurface of the test samples. The counter bodies were cleaned with acetone and subsequently with ethanol. Reciprocating sliding tests were performed at a normal load of 9 N and 7.3 N on using stainless steel and alumina ball respectively, to achieve the same maximum Hertzian contact pressure of around 100 MPa. A sliding frequency of 3 Hz and a peak-to-peak displacement amplitude of 600 μm were selected in order to achieve a gross slip sliding regime. Two different sliding directions, i.e. parallel and anti-parallel to the fiber orientation, were applied (Fig. 1). The repeatability was evaluated by performing a minimum of three replicate reciprocating sliding tests on each specimen for each set of testing conditions.

Focused ion beam (FIB) and scanning electron microscopy (SEM) (FEI NOVA NANOLAB 600) were used to prepare cross-sections through the worn areas in order to investigate the debonding and the outbreak of carbon fibers. White light interferometry (Wyko3300) was used to measure the maximum wear track depth.

The experimental sliding test conditions used can be grouped into three main divisions, namely one related to the sliding direction, another one related to the type of counter body ball, and finally one related to the environmental composition (see Fig. 2). The effect of each of these variables on the degradation of the carbon fiber reinforced epoxy was investigated in order to get a

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