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Erosion Resistance of CFRP Composite Materials with Different Fiber Weaves Exposed to Cryogenic Treatment

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ABSTRACT
Carbon fiber–reinforced polymers (CFRPs) are often exposed to solid particle erosion wear due to small particles moving freely in the atmosphere or in the environment and their impact on aircraft bodies. In this study, fiber weaves of CFRP composite materials of the following types were produced: unidirectional (U), plain weave multidirectional (P), and twill 2×2 weave multidirectional (T). Tests that involved three different CFRP composite materials were conducted at room temperature with two groups (pure and exposed to cryogenic liquid nitrogen treatment), at two different fiber directions (0° and 45°), and at four different impingement angles (30°, 45°, 60°, and 90°). Subsequently, erosion wear rate values were calculated and erosion rate–impingement angle graphs were produced for each fiber direction to study the effect of the different environments. Scanning electron microscopy images of surface deformation were compared between the different experimental conditions.

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Carbon fiber–reinforced polymer; cryogenic effect; erosion resistance; fiber weave; liquid nitrogen; solid particle erosion

Introduction

In many aerospace applications, especially in the aircraft industry, metal alloy materials are being replaced with composite materials. Among composite materials, carbon fiber–reinforced polymers (CFRPs) are widely used in the aircraft body because of their low density and high strength-to-weight ratio. Their high fatigue and creep resistance and good corrosion resistance are further important advantages. The light weight of composite materials, combined with the need for improved energy efficiency, contributes to the reduction in fuel consumption and CO_2 emissions by aircraft *(1)*.

Unwanted substances that are very small and light, such as particles, grains of sand, fuel ashes, etc., that are found in the atmospheric environment can cause solid particle erosion wear due to their impact on aircraft bodies when carried by the wind *(2)*. The mechanical properties of the materials used in aircraft bodies can therefore be negatively affected by this abrasive effect. Solid particle erosion wear is a type of wear caused by solid particles of different sizes that impact at different angles and velocities on the surface of a material and has become the most investigated type of wear in applications where energy conservation is of major importance *(3, 4)*. In solid particle erosion wear studies of polymer materials, it was determined that polymer materials and polymer matrix composites have less erosion resistance in dusty ambient service conditions compared to metals *(5–7)*. The use of CFRP materials instead of metal alloy materials that interact with the external environment means more detailed investigations into the conditions that develop solid particle erosion wear resistance are required. Academic studies have been carried out on this subject *(8–10)*, and there has been a widespread use of these materials; however, further in-depth studies are now expected.

Expansion of the parameters of CFRP composites that affect erosion wear resistance needs to be intensified. The characteristics of the matrix material used in the structure and the orientation of the fibers used as reinforcements represent the starting point of studies carried out to date. The basic components of the composites are the matrix and a reinforcement material. The reinforcement materials directly affect one or more operating properties of the composites, giving different mechanical and chemical properties *(9)*. Depending on the properties of the reinforcement material used, erosion resistance may increase or decrease *(11)*. It has been observed that the fiber orientation as well as the type of fiber in the composite material significantly affect the mechanical properties of the composite material *(12)*. Another study focused on how solid particle erosion wear changes according to fiber orientation *(13)*.

It is known from industrial examples that the temperature applied in heat treatment and the cooling rate have significant effects on the strength of the materials. Because of the necessity brought about by technological transformation, metal materials are being replaced by polymer-based composite materials and how the mechanical properties of these composite materials change with cryogenic treatment is being examined *(14, 15)*. CFRP materials, especially those

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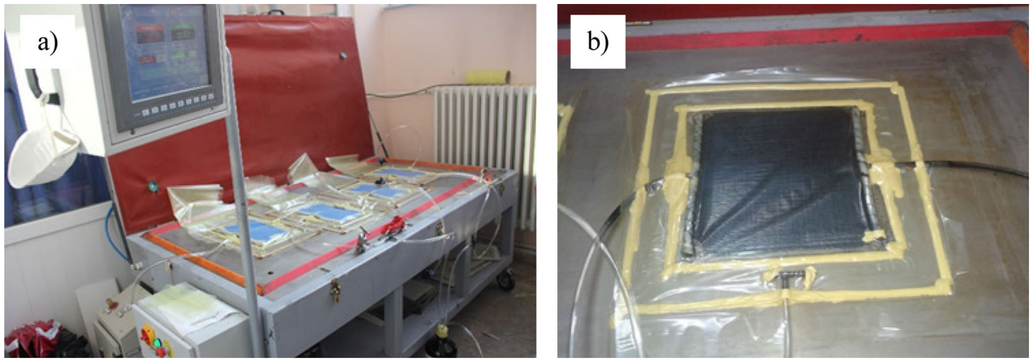

Figure 1. Vacuum-assisted resin infusion molding method: (a) production unit and b) production stage.

Table 1. Mechanical properties of the carbon fiber and epoxy matrix in the samples.

Mechanical properties	Specifications of the matrix	Specifications of the reinforcement (carbon fiber)
Density (g/cm^3)	1.13–1.17	1.76
Tensile stress (MPa)	60–75	3,950
Modulus of elasticity (GPa)	3.7–3.2	238
Elongation at break (%)	8–16	1.7

used extensively in the fuselage, have not been subject to any research on how cold environments affect the erosion resistance of these materials once the aircraft is airborne.

In this study, we aim to provide an additional dimension to the literature studies on this subject. Limited research on solid particle erosion wear under the influence of a cryogenic environment (approximately −196 °C) has been reported *(16, 17)*. Materials subjected to a cold environment—that is, cryogenically treated materials—have generally been materials that have less machinability, like hard titanium and layered CFRPs, where the surface of the tool and/or the material to be machined is not elevated to high temperatures *(16, 18–23)*. As a result, the fiber orientations of unidirectional (U), plain weave multidirectional (P), and twill 2 × 2 weave multidirectional (T) composite materials were produced as experimental specimens. The solid particle erosion wear behavior of three different CFRP configurations of two different types (pure and exposed to cryogenic effect), four different impingement angles (30°, 45°, 60°, and 90°), and two different fiber directions (0° and 45°; [0] and [45]) was investigated. The cryogenic effect is the behavior of materials at very low temperatures. Alumina erodent particles with an average impact velocity of 34 m/s and an average diameter of 400 µm were used to evaluate the erosion rates based on the weight loss in the test specimens, and the erosion ratio–impingement angle graphs were obtained for each fiber direction. The effect of a 30° impingement angle, fiber direction, and especially the cryogenic effect resulted in a significant difference in erosion resistance.

Experimental procedure and applied methods

Material production process

Specimens with different fiber orientations of plain-woven carbon fiber fabrics (0° and 0°/90° fiber orientation angles with 200 g/m^2 areal weight and fiber diameter of 7 µm) were used. A combination of hardener and resin is used in the production of plates in a ratio of 1:4, and details of the experimental unit and production stage were obtained using the vacuum-assisted resin infusion molding method, shown in Fig. 1. After the resin was impregnated by a vacuum, it was cured for 1 h at 70 °C and 2 h at 110 °C. The layers of the CFRP composite materials produced are approximately 1 mm thick and 1 mm was applied as the basic reference parameter in all materials as the distance between layers of the composite.

Table 1 provides the mechanical properties of the resin and fibers used in the samples. The epoxy matrix used in the composite materials is a brittle thermosetting resin, and the fibers have thermoplastic properties. As a result of combining these different properties, a revision occurred in the erosion wear and tensile strength, and how the pure and cryogenic environments directly affect these properties is reported *(24)*.

According to the orientation of the fibers used in the composite materials, the fatigue, bending, and wear resistance of the materials vary *(25, 26)*. The material strength is higher in directions perpendicular to the fiber orientation. Therefore, fibers can be placed at different directions in the matrix according to the loading conditions that the material will be exposed to. In this study, U, P, and T CFRP composite materials were used as test specimens (Fig. 2).

Cryogenic treatment process

Liquid nitrogen (LN_2) is preferred to achieve the cryogenic effect because it is easy to obtain, clean, environmentally safe, and widely available *(27)*. In industrial applications, the cryogenic cooling process used in machining processes can be done in four different ways: indirect cooling of the cutting tool, spraying the contact area of the tool and the workpiece with cryogenic liquid, cooling of the cutting tool before the process, and precooling the workpiece in cryogenic medium *(28)*. In this study, precooling was performed before the experimental samples were exposed to erosion wear. To prevent the rapid evaporation of liquid nitrogen, expanded polystyrene foam with a heat transfer coefficient of 0.039 W/(m.K) was used as the thermal insulation material and the test samples were kept in liquid nitrogen for 1 h, after which the temperature was observed and the results were recorded. Figure 3

Figure 2. Test samples according to fiber orientation: (a) unidirectional (U), (b) plain weave multidirectional (P), and (c) twill 2 × 2 weave multidirectional (T).

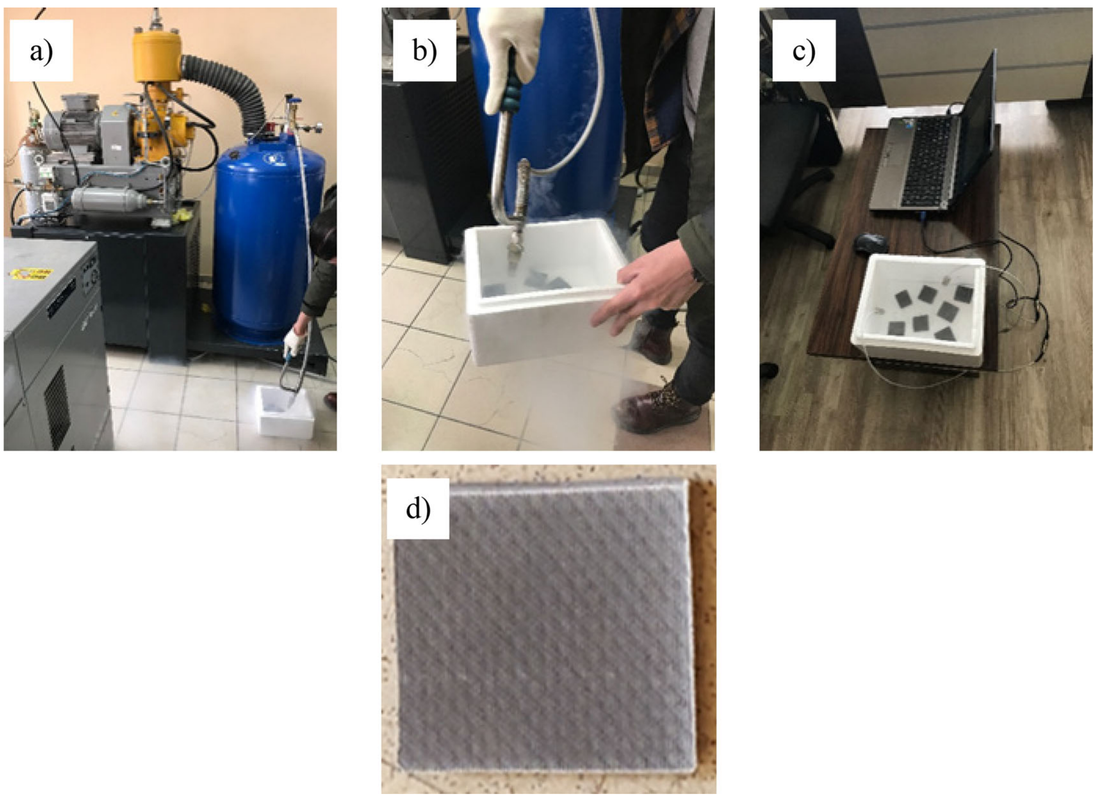

Figure 3. Cryogenic cooling process: (a) LN_2 unit, (b) LN_2 application, (c) temperature control, and (d) photograph of a sample after processing.

shows how all of these processes were carried out and photographs of samples subjected to cryogenic cooling.

Erosion testing process

Solid particle erosion (SPE) tests were performed in a test device that was specially produced according to ASTM G76-95 standard, shown schematically in Fig. 4.

In erosion wear tests, the erodent affected the sample surfaces at desired angles and speeds using dry and pressurized air. The pressure at the nozzle was controlled by means of a pressure regulating valve and a pressure gauge. An Al_2O_3 erodent with a rounded geometry and average diameter of 400 µm was used as the abrasive particles.

To determine the velocity of the abrasive particles, the double-disc method was used *(29)* and the abrasive particle velocity was set to 34 m/s. Using the impingement angle adjustment apparatus detailed in Fig. 4b, impingement angles of 30°, 45°, 60°, and 90° were applied. In addition, [0] and [45] fiber directions for each experimental sample were determined using the sample holder, and the results obtained were consistent with literature studies *(30, 31)*. The distribution of force components acting due to abrasive particle impact on fiber directions is shown in Fig. 5. Normal and tangential components that affect fiber direction and cause variability in solid particle erosion wear are visually explained in the schematic.

This study compared the variability in the CFRP composite materials in erosive particle impingement according to different fiber orientation ([0] and [45]).

Before and after the SPE tests, the test samples were weighed on a precision balance. The erosion rate (mg/kg) was determined by dividing the difference between the final and initial weights by the number of abrasive particles used in each experiment. To minimize the error rate in the

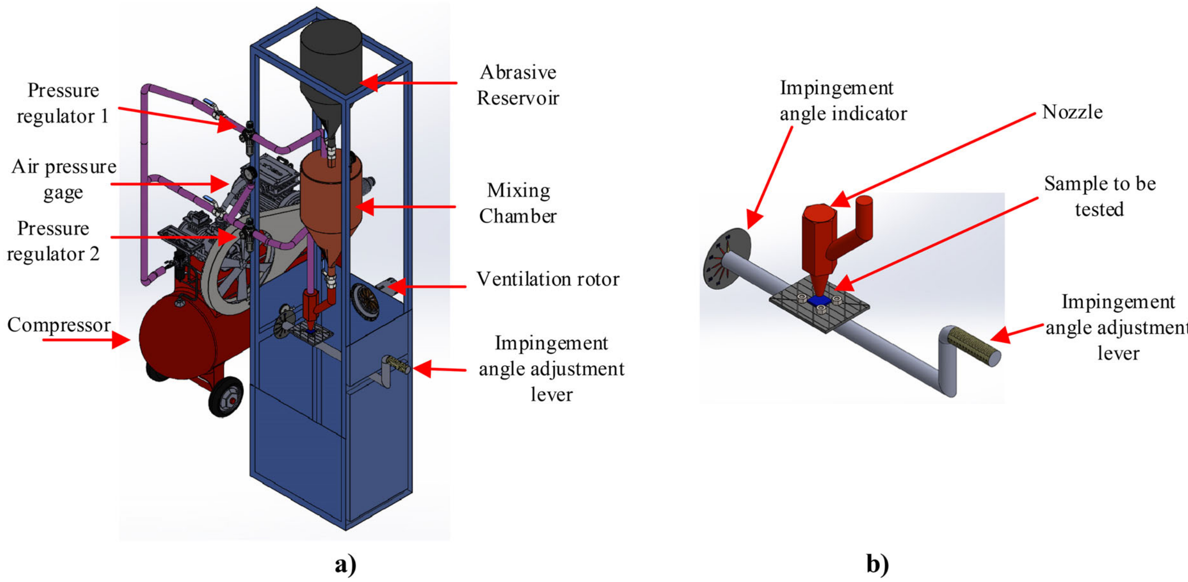

Figure 4. Schematic image of the SPE tester: (a) 3D drawing and (b) impingement angle adjustment lever.

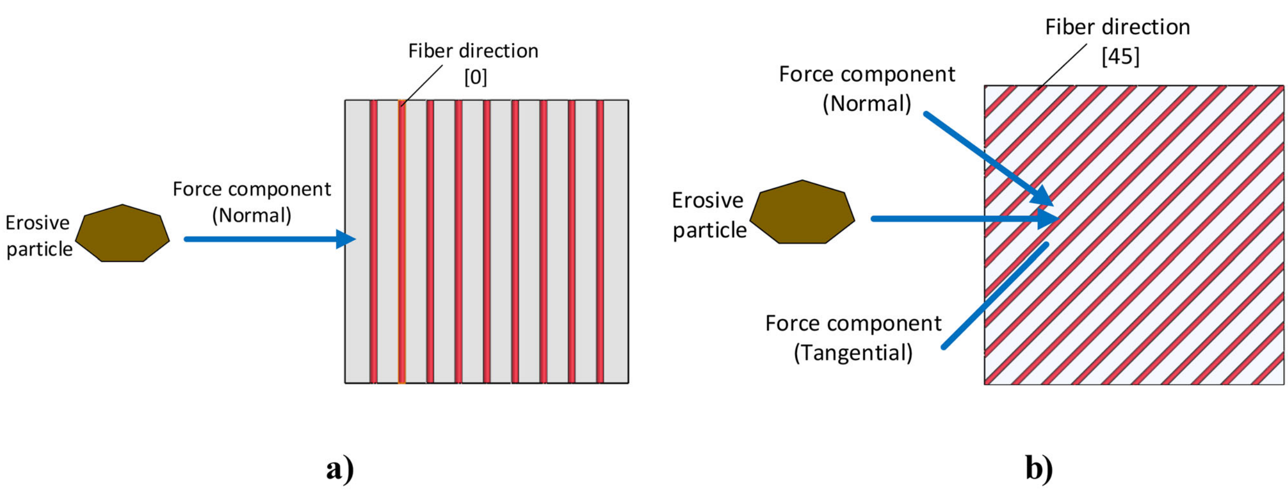

Figure 5. Force components of the abrasive particle impacting the surface according to fiber direction: (a) [0] fiber direction and (b) [45] fiber direction.

experiments, three experiments were carried out under the same conditions. Moreover, after the SPE tests, erosion scars were determined using ImageJ image processing *(32)* of photographs of the wear surfaces of the samples where the most wear had occurred (Fig. 6).

An optical laser measurement unit was used to define the wear depth of the erosion scar (Fig. 7). To ensure the accuracy of the measurement, the samples were placed on a granite measuring table and the depth of erosion wear was measured using an optical laser after position adjustment. This method provides an easy measurement to compare the results from other methods, and it was found that the results obtained by image processing were more compatible with the erosion rate values obtained due to weight loss.

Results and discussion

The main purpose for scientific examination of the cryogenic effect is determination of the wear and corrosion resistance of materials whose temperature is reduced below 0 °C. Based on this theory, the level of significance of the cryogenic process application industry has also increased. It has been determined that the working conditions in cold environments can be simulated by the effect of cryogens such as liquid nitrogen, and it is concluded that the erosion wear data of composite materials operating in these conditions (e.g., aerospace and aviation applications) will produce more meaningful results. Therefore, the materials in this study were examined under cryogenic conditions.

Unidirectional CFRP

CFRP test specimens with a unidirectional orientation were classified as unidirectional pure (UP) or unidirectional cryogenic (UC). These samples were subjected to SPE tests after separation into [0] and [45] configurations. The impingement angle and erosion rate change were examined after SPE wear tests of these samples (Fig. 8). Erosion wear increased significantly in samples that had been exposed to the cryogenic liquid effect for approximately 1 h. The erosion resistance of these samples decreased due to the cryogenic effect and there was an approximately 3.71 times increase in the erosion rate (at 30° impingement angle and [0] fiber direction) compared to samples that were not exposed to the cryogenic effect. The highest erosion wear occurred at an abrasive particle impingement angle of 30°. Fiber directions [0] and [45] were chosen to determine which fiber direction is more resistant to wear at different impingement angles of the abrasive particles. The resultant force consists of normal and tangential forces depending on the impingement angle of the abrasive particles. In the experiments, the samples with fiber direction [45] showed greater resistance to erosion wear *(30)*. A change in fiber direction from [0] to [45] decreased the erosion rate in the pure group by 4.8% and the group exposed to cryogenic treatment by 23.1%.

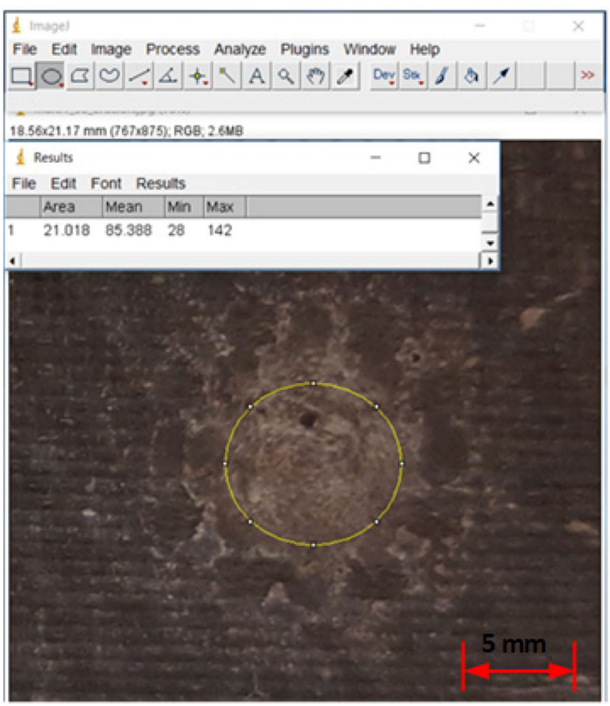

Figure 6. Determination of erosion scar by ImageJ image processing.

Plain weave multidirectional CFRP

Plain weave multidirectional CFRP test samples have good fabric stability but are less pliable. Plain weave multidirectional CFRPs were divided into two groups: plain weave multidirectional pure (PP) and cryogenic (PC). SPE experiments were conducted according to fiber orientation ([0] and [45]). The maximum erosion rate occurred at an abrasive particle impingement angle of 30° (Fig. 9). The erosion rate in the group exposed to cryogenic treatment was determined to be 4.2 times (at 30° impingement angle and [0] fiber direction) higher than the erosion rate of the pure group. When unidirectional CFRP test samples and plain weave multidirectional CFRP test samples were compared after exposure to LN_2, the plain weave multidirectional CFRP samples showed a higher erosion rate than the unidirectional test samples because of their high fiber density.

After the cryogenic effect, the amount of material removed from the sample surface decreased for the sample with fiber direction [45] because the force acting on the sample was divided into normal and tangential forces. Therefore, the angular impact effect of particles contributed

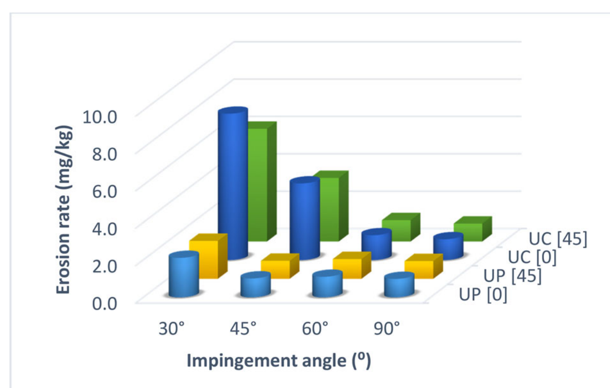

Figure 8. Impingement angle–erosion rate graph of the UP and UC test samples by fiber directions [0] and [45].

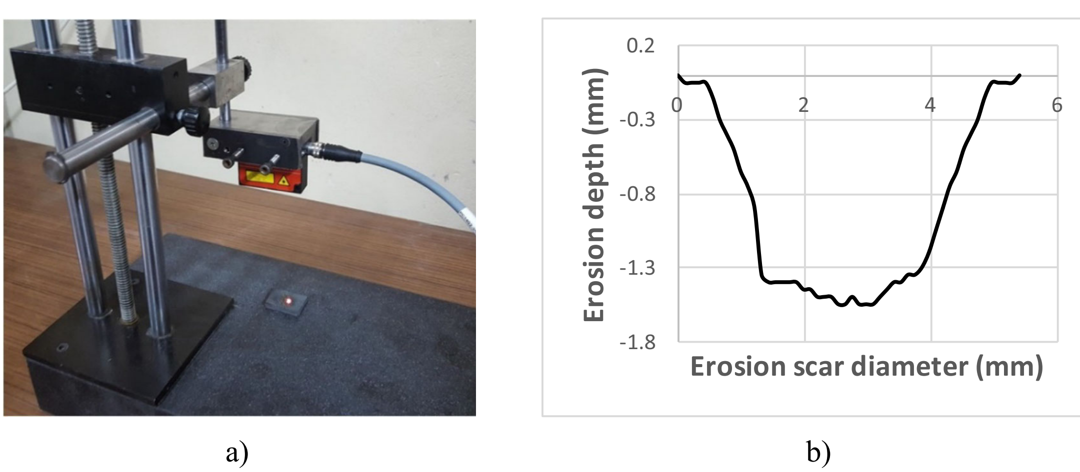

Figure 7. Optical laser measurement: (a) test unit and (b) graph of wear depth and erosion scar diameter.

positively to the erosion resistance in the change of fiber orientation.

Twill 2 × 2 weave multidirectional CFRP

Twill 2 × 2 weave multidirectional CFRPs are more flexible than plain weave multidirectional CFRPs and have better ductility while maintaining more fabric stability than four- or eight-harness satin weaves. Twill weave multidirectional CFRP test samples were also divided into two groups: twill pure (TP) and twill cryogenic (TC). SPE experiments in the fiber directions [0] and [45] were performed and graphs of the impingement angle and change in erosion rate are shown in Fig. 10. In the literature *(33, 34)*, the maximum erosion rate in ductile materials is obtained at a particle impingement angle of 30°, which was also observed in these test samples. Twill weave multidirectional CFRP test specimens exposed to cryogenic treatment had an erosion rate ~2.7 times (at 30° impingement angle and [0] fiber direction) higher than that of the pure group. It was concluded that the main factor in the difference in the rate of erosion in pure and cryogenic-treated samples is the orientation of the fibers in the fabric (unidirectional or multidirectional). In addition, the maximum SPE wear for the pure and cryogenic groups was obtained in the TC/CFRP samples. This can be explained by the fact that the matrix fiber combinations in the twill weave samples have a structure that is less resistant to erosion wear due to weak bonding forces.

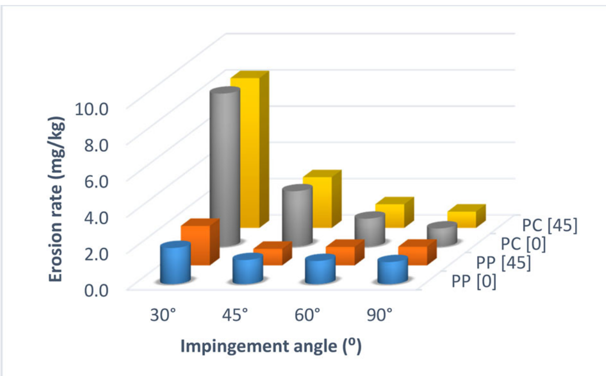

Figure 9. Impingement angle–erosion rate graph of the PP and PC test samples for fiber directions [0] and [45].

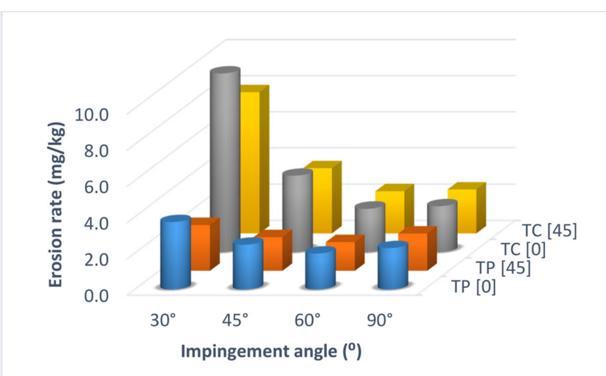

Figure 10. Impingement angle–erosion rate graph of the TP and TC test samples for fiber directions [0] and [45].

Steady-state erosion

After the SPE experiments on samples with different fiber orientations, graphs of the wear conditions were obtained and scanning electron microscopy (SEM) images of the unidirectional CFRP test samples (pure and cryogenic) were captured (Fig. 11). Because the maximum erosion rate was realized at an impingement angle of 30°, the SEM images of this sample were examined and were found to be compatible with those of ductile materials in the literature. It was observed that the bond between the matrix and the fibers was weakened by the cryogenic effect and the abrasive particles striking the surface were able to remove material from the surface more easily.

Figure 12 shows SEM images after erosion wear at an impingement angle of 30° of the plain weave multidirectional CFRP test specimens. Because these experimental samples have a regular and tight weave structure, increased resistance to erosion wear was observed. As seen in the SEM images, regions of fiber fracture were observed on the surface after erosion wear due to the tight weaving on the material surface. The groove densities were shallower compared to the other experimental sample types.

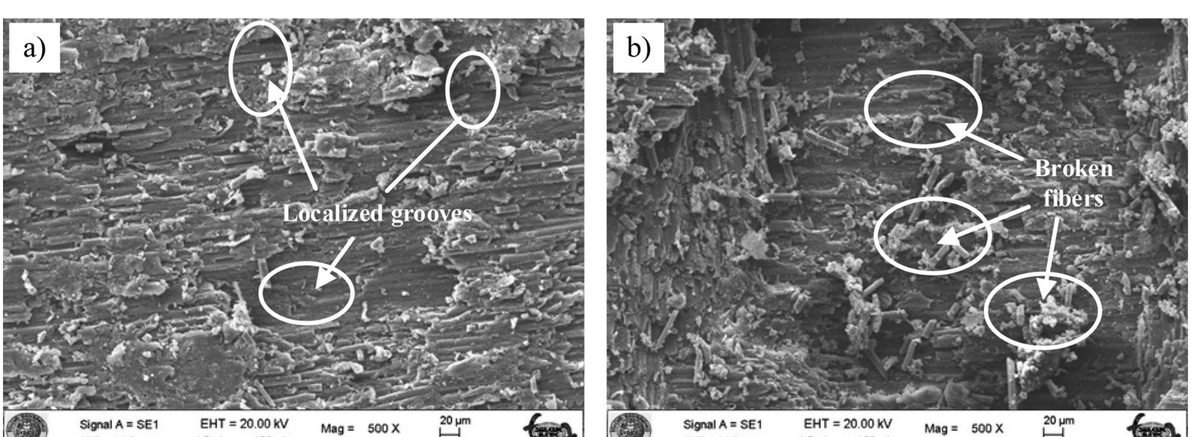

Figure 11. SEM images at 30° impingement angle and [0] fiber direction: (a) UP and (b) UC.

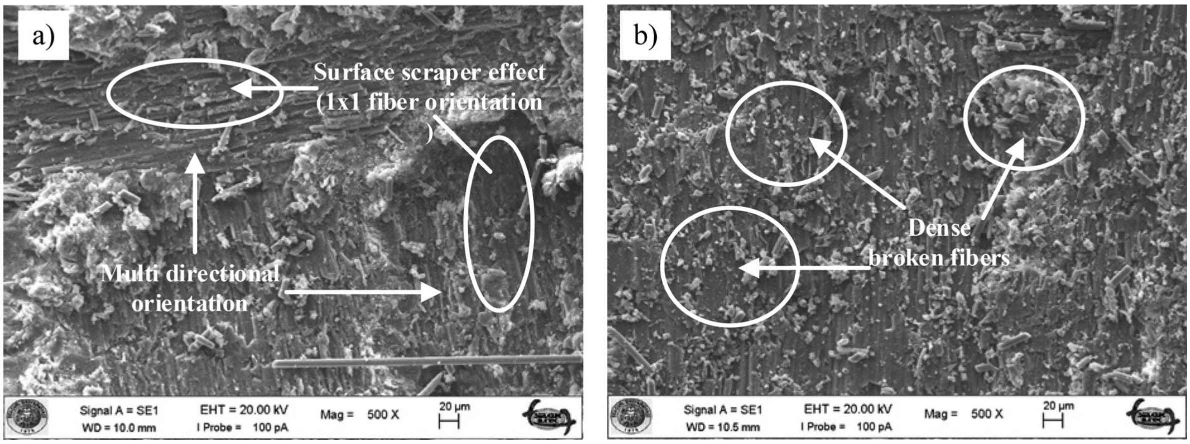

Figure 12. SEM images at 30° impingement angle and [0] fiber direction: (a) PP and (b) PC.

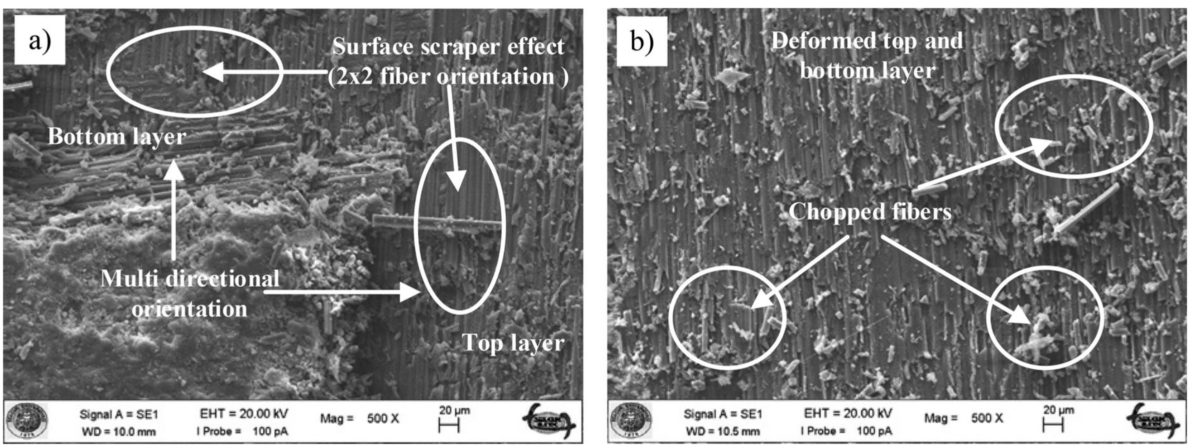

Figure 13. SEM images at 30° impingement angle and [0] fiber direction: (a) TP and (b) TC.

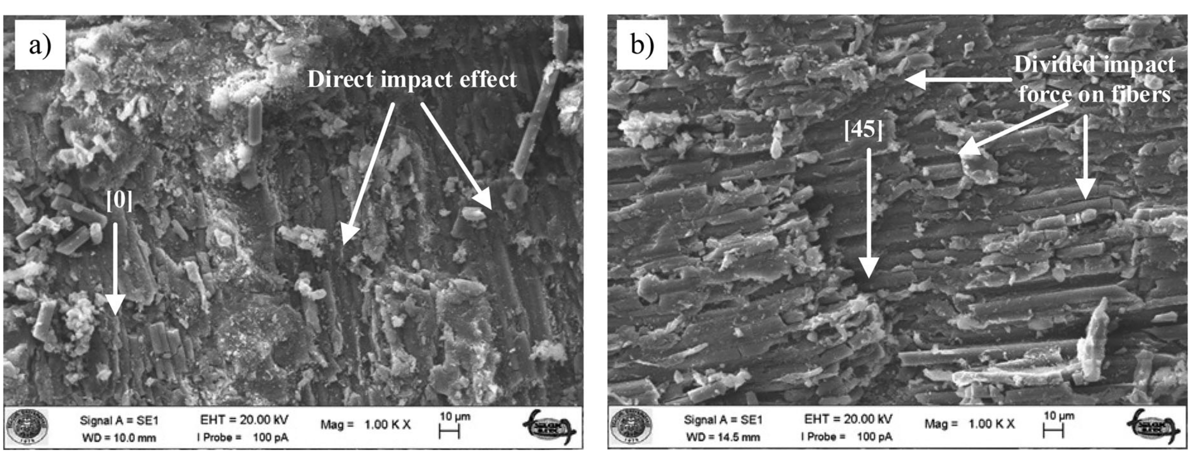

Figure 14. SEM images at 30° impingement angle: (a) PP [0] and (b) PC [45].

Figure 13 shows SEM images of the twill weave multidirectional CFRP test specimens with a 2×2 fiber configuration. As seen in the SEM images, the fiber weave of the twill weave samples is like a synchronized diagonal. Due to the loose weaving structure, abrasive particles that strike the surface cause more material removal, resulting in the highest erosion rate in this sample group.

SEM images of the [0] and [45] fiber directions at a 30° impingement angle were investigated (Fig. 14). Because the abrasive particles impact the fibers at an angle ([45]), the maximum impact force is not applied directly to the fibers; in other words, the force is divided into components, so less erosion wear occurred compared to the [0] sample.

The SEM images in Fig. 15, show deformations formed on the surface by abrasive particles impacting the surface at 30° and 90°. In the samples subjected to an angular effect (30°), the debonding and scraper effects on the surfaces were more dominant than on the samples that had been

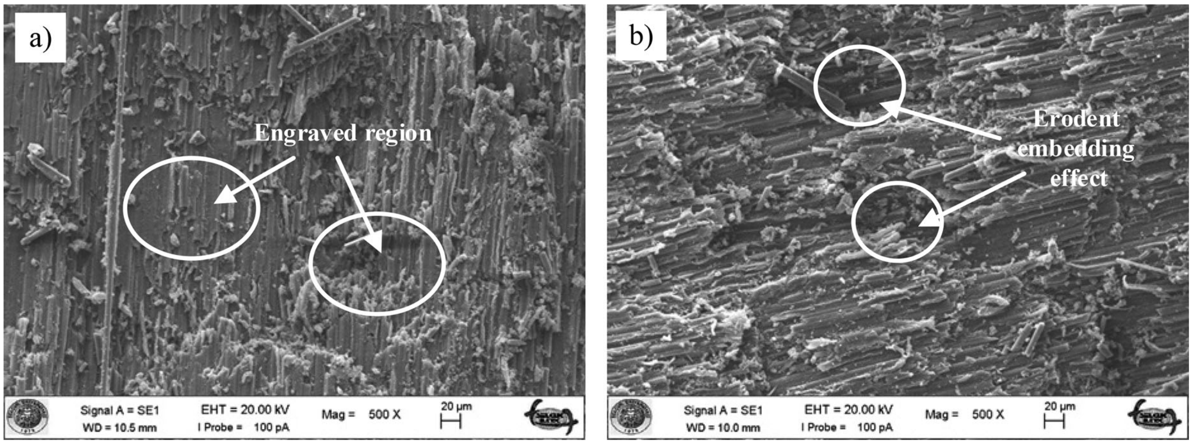

Figure 15. SEM images of T/CFRP test samples: (a) 30° and (b) 90°.

exposed to the particles impacting normal angle (90°). In addition to the normal angle effect of the particles, the fibers were more embedded in the surface.

The density of fiber–matrix adhesion of three different CFRP composite materials with variable fiber orientations and matrix densities is affected by the interface reaction that occurs due to the cryogenic effect. It was concluded that the main reason for this effect is the thermal expansion coefficient mismatch of the fiber and the matrix. This mismatch revealed the weak fiber–matrix interface due to increased thermal stresses. In this case, it has been shown that erosion resistance decreased in the cryogenic environment.

Conclusions

The following results were obtained from the experimental studies:

- In the experiments comparing pure and cryogenic effects, it was concluded that treatment with LN_2 clearly contributed to erosion. This effect at a maximum level was determined to be 370% in the unidirectional test samples, 420% in the plain weave multidirectional samples, and 260% in the twill 2×2 weave multidirectional samples. Therefore, UC, PC, and TC showed less erosion resistance than UP, PP, and TP.
- The direction of the fibers led to less erosion in [45] than [0] directions because the force was divided into components. This effect was seen in almost all test samples and showed decreasing erosion rates in the order TC > PC > UC.
- To obtain data for comparisons, erosion scars and depths were measured by an optical laser and image processing. The maximum erosion rate occurred at an impingement angle of 30° in all test sample groups. In addition, an increase in impingement angle tended to increase erosion resistance. This situation is consistent with the results of ductile materials reported in the literature *(33, 34)*.
- The test specimens produced with the combination of uni- and multidirectional and 1×1 to 2×2 were found to differ in erosion resistance. This demonstrates that results of experimental erosion studies depend on fiber orientation.
- Because the cold effect reduces intermolecular mobility, it causes a decrease in the elastic elongation property of the material. This facilitated the rupture of the material from the surface when the erosive particles impacted the sample surfaces.
- As a result of the differences in fiber orientation and the difference in weave structure, SEM images showed differences in groove depths and broken fibers.

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