Investigation into features of fracture toughness of a transparent E-glass fiber reinforced polyester composites at extreme temperatures

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In recent years, several investigators have made efforts to satisfy industrial and transportation demands with respect to high mechanical properties. Moreover, the transparent composites are needed for automotive and aircraft applications. Thus, developing more efficient and advanced transparent composite techniques has recently got more attention but seldom studied in harsh conditions. The main goal of the present study is to investigate the impact of exposing a transparent polyester composite to different temperatures ranging from high to very low (60, room temperature, 0, -30, -60, and -80 °C) and 50% humidity. The study of the effect on both fibers and matrix upon exposure to the different conditions and the composite represents an attempt towards climate resistance. In this research, fracture toughness of E-glass fiber reinforced unsaturated polyester composite at high and very low temperatures and humid environments were investigated. The investigation deals with the characterization of fracture strength properties according to the ASTM standards and mechanism of fracture by scanning electron microscopy. The transparent composite was fabricated from the unsaturated polyester matrix and E-glass fibers. Using of E-glass fiber for reinforcing with 15% volume percent for preparation of the transparent composite exhibits fracture toughness at very low temperatures. It was also noted that the technique of giving an excellent transparent polyester composite can be utilized confidently to architect and aircraft structures.

1. Introduction

One of the many challenges associated with aerospace engineering is material choice for the structures of the craft and other equipment. The combination of harsh environment, large forces at high cycles, and the need for lowest possible installed cost mean that the choice of materials is limited. Glass fiber reinforced polymers (GFRP) is one choice among many candidate materials for these applications. The most common polymers used for composite matrices are the epoxies and polyesters. For transparent composites that are required for different architect and aircraft structures it much challenging to fabricate the composite and maintaining both mechanical and optical properties. The task becomes tougher if the composite is expected to expose to extreme conditions such as high or very low temperatures. For the conventional polyester composites, few studies are available related to fatigue crack propagation in the woven laminates. These investigations had been conducted to study and develop fillers and tougheners that can enhance the fracture toughness of resin. However, the unsaturated polyester resins (UPR), which is a cost-effective and a chemical resist thermoset requires only curing at mild conditions [1, 2, 3]. To achieve a satisfactory toughness of these resins, more extensive studies are conducted and still ongoing. Numerous studies were conducted to improve the mechanical properties of UPR against harsh environments. Some studies have investigated the use of nanomaterials or natural fiber to reinforce polymers. Han et al. [4] reported the use of a UPR added to a 2 wt % nano-silica to form a nanocomposite. The formed nanocomposite exhibited a satisfactory fracture toughness and wear resistance under marine conditions. As the fracture toughness has decreased due to prolonged immersion in seawater, the nanoparticles of silica were able to reduce the fracture toughness loss to an acceptable level. Graphene oxides (GO) particles showed a good performance as a toughener of UPR [5]. This positive impact arises from the fact that its networking derivatives enhanced GO-matrix bonding and composite compatibility. Ali et al. [6] investigated the improvement of fracture toughness of UPR using bamboo fiber to manufacture woven layered composite. They found that the bamboo fibers reinforced UPR showed a satisfactory fracture property and the method of reinforcement was able to produce a high fracture and fatigue resistance composites. For the influence of temperature and moisture on...
the performance of composite materials, several studies have been conducted [7, 8, 9, 10]. Many experimental works have been set up to determine the extreme of interlaminar fracture that may result in composite failure at the time of practical use under harsh conditions [11]. Experimental evidence of these studies is strictly related to their studies' conditions and components in the composites. Most of these studies focus on following cracks in notched specimens loaded in tension–tension fatigue to calculate fracture parameter and examine fracture morphology. Owing to the big diversity of environmental conditions where the transparent UPR composites could be utilized and proceeding with the challenges to get an appropriate transparent composite of such mechanical and thermal defects, the main thrust for improving of more efficient technologies on new transparent UPR composite is viewed a vital issue in aircraft and buildings structure. Therefore, the main focus of the present investigation is to fabricate a new cost effective transparent UPR composite based on glass fiber reinforced the polyester matrix and examining it to withstand fracture loads, humidity and different extreme temperatures by novel technique; thus producing an environmentally benign high strength transparent composite. The present study aims to obtain a high-fidelity fracture toughness data of a transparent UPR composite under humid and different temperatures, with emphasis on very low temperature. Such low temperatures are encountered in article regions and at high altitudes.

2. Experimental work

2.1. Fabrication of composite material

A commercially available unsaturated polyester–styrene resin (polymer content = 65 wt %, styrene content = 35 wt %, untreated, Aropol) was utilized as a curable polymer matrix. The manufacture of the resin did not provide more information about the chemical ingredients. The molecular weight of the resin was measured by chromatography gave a 1700 g/mol of the as-received resin. The glass transition temperature (Tg) of the solidified resin was determined at 73 °C by Diffraction Scanning Calorimetry (DSC). At this temperature, the polymer matrix softens and turns into a ductile polymer. E-glass short fiber (low conductivity glass fiber) [12] was used for the reinforcement with a 15% volume.

The glass fiber reinforced polyester plates were fabricated using the process described elsewhere [13].

2.2. Test coupon fabrication

The specimens used for this study are the composite samples shaped according to ASTM Standard E399-09 [14] for plane-strain fracture toughness. The panels prepared in section 2.1 were used for this test after shaping. A Compact Tension (CT) specimen geometry (Figure 1) was used for this test, it is recommended for homogeneous structure and unidirectional fiber-reinforced structures.

2.3. Fracture toughness testing

Samples were prepared using ASTM E399 as a guide. To remove residual stress formed around the pre-crack tip, a sharp pre-crack was introduced to the sample by razor blade tapping. The notch was sharpened using a razor blade before testing. Samples were held at temperature for 1 h before testing. Samples were loaded using a constant displacement rate to failure using a 4448 N (1,000 lb) load cell. K was calculated using the original notch size and the K calibration coefficients listed in ASTM E399. A servo-hydraulic MTS 810 frame and a chamber of MTS 651 were used in this work. For this quasi-static test, the specimen was loaded such that the rate of increase of stress-intensity factor is between 0.55 and 2.75 MPa√m/min (30 and 150 ksi√in./min) during the initial elastic displacement. Figure 2 shows the frame under the environmental chamber during testing over a temperature range of +60° to -100 °C.

A nitrogen cooling chamber was used to cool the coupon during testing. A radiation thermometer measured a 1 °C temperature rise/drop in surface temperature during the test. The temperature in the test room with the testing machine is maintained at 21 °C.

All fracture tests were conducted at a displacement rate of 1 mm/min. The crack length (a) to width (w) ratio of a/w = 0.2 was used, where a is the pre-crack length, w is the distance from the center of loading pin to the edge of the CT specimen). At least 3 CT specimens were tested at each temperature Figure 3 shows the CT specimen before and after failure. To obtain fractography of the failed specimens, the samples were first completely broken apart by hand, if they were not already completely broken from fatigue testing and studied by optical microscope. The samples were then studied with a scanning electron microscope (SEM) after gold-sputter coating.

The temperature effect on the mechanical properties of composites derives partly from the internal stresses introduced by the differential thermal coefficients of composite components. Such internal stresses change magnitude with temperature change, in some cases producing matrix cracking at very low temperatures. The cyclic stress intensity (∆K) was calculated according to the following equation [15]:

![Figure 1. Fracture toughness specimen.](image1)

![Figure 2. Fracture toughness specimen clamped on MTS servo-hydraulic multipurpose testing machine.](image2)
Figure 3. CT specimen before and after failure.

Figure 4. Load versus displacement curves for the polyester composite at $R = 0.1$ and different temperatures a)60 °C, b) RT, c)0 °C, d)-30 °C, e)-60 °C, f)-80 °C, g)-100 °C.
\[ \Delta K = \frac{\Delta P \cdot B}{W \cdot \sqrt{w}} \sqrt{f' \left( \frac{w}{w} \right)} \]  
(1)

\[ f' \left( \frac{w}{w} \right) = \left( \frac{2 + \frac{w}{w}}{1 - \frac{w}{w}} \right)^{7/4} \left[ 0.886 + 4.64 \left( \frac{w}{w} \right)^{2} - 13.32 \left( \frac{w}{w} \right) + 14.72 \left( \frac{w}{w} \right)^{-1} - 5.6 \left( \frac{w}{w} \right)^{3} \right] \]  
(2)

where:
- \( \Delta P \) = load range, [lbs], \( B \) = thickness, [in], \( W \) = width [in.], \( f \left( \frac{w}{w} \right) \) = geometric correction factor for compact tension specimen.

The energy of interlaminar fracture toughness \( E_{IFT} \) was determined from [16, 17].

\[ E_{IFT} = \frac{9S0a^2}{2b(3w^2 + 2W)} \]  
(3)

where:
- \( S \): Fracture load applied by the apparatus for crack propagation.
- \( \Delta \): local displacement.
- \( a \): a crack length
- \( l \): is a half breadth of fracture toughness specimen
- \( b \): Apparatus beamwidth.

3. Results and discussion

3.1. Effect of temperature and humidity on load-displacement behavior

The results of the DSC study indicate that the curing reaction starts at room temperature and gets completed almost around 50 °C. This indicates that the unsaturated polyester resins have a good induction period under this condition and therefore it can be expected that at room temperature the unsaturated polyester resins would have a very long induction period which is needed for better damage resistance. The sample is heated at 20 K/min while the sample is cooled slowly (0.1 K/min) goes through its \( T_g \) at 73 °C. If it is then heated at 20 K/min, it does not transform until 73 °C. This means that the sample must absorb more energy to reach the enthalpy of the rubbery state. This results in an endotherm, superimposed on the glass transition step. The heating and the cooling rates must, therefore, be stated.

Fracture behavior of the fiberglass polyester composite has been studied in terms of fracture toughness at different temperatures. The impact of exposure to different temperatures is shown in Figure 4. Figure 4 reveals that the unsaturated polyester composite in this study displays a non-linear behavior of load versus displacement at room temperature and higher temperatures (60 °C). For temperatures of 0 °C and below the loading, the response is linear. For temperatures well below 0 °C, that is -30° to -100 °C, an initial linear response develops into a non-linear load-displacement response at about 0.5 mm displacement. This change in behavior is possibly due to significant micro-cracking in the matrix and at the fiber-matrix interface. However, the load-carrying capacity is not compromised as the peak load increases with lower temperatures which are previously observed by Perrin et al. [18]. The increase in peak load achieved is due to the higher clamping pressure of the matrix on the reinforcing glass fibers as the temperature decreases. The increased clamping pressure is due to the higher thermal expansion coefficient of the polyester matrix as compared to the glass fibers, and the higher compressive pressure promotes greater load transfer to the fibers. Furthermore, at lower temperatures, the mobility of the polymer chains is low, which enhances the loading capacity at lower displacements. The peak load increased by 80% from the room temperature value of 1250 N–2250 N at -100 °C. From Figure 4 it should be noted that as the temperature decreases not only the peak load increases but also the displacement at peak load increases, at -100 °C the displacement approaches 2 mm, which is the displacement at +60 °C when the matrix is much more compliant begin closer to the glass transition temperature of 73 °C and the strength has been degraded [19, 20, 21]. Thus, the elastic energy in the composite is significantly higher at -100 °C than at room temperature.

Figure 5 shows that exposed specimens of the unsaturated polyester composite to different temperatures illustrate an increase in fracture toughness over time along with the propagation of crack displacement. At room temperature, the fracture toughness, \( K_c \), approaches a maximum of 5.1 ksi in\textsuperscript{0.5} that corresponds to 0.94 mm (0.037 inches). Fracture toughness increases as temperature decrease over the cryogenic range of 0 through -80 °C. According to previous works conducted at very low temperatures [18, 22, 23], it was found that the mechanism of failure of short fiber reinforced composite follows Mode I interlaminar fracture due to the ductile-brittle transition of the phases incorporated in the transparent composite. The fracture toughness index obtained in the present study jumped to 7.3 ksi in\textsuperscript{0.5} with a 1.3 mm (0.052-inch) as temperature decreases to -60 °C and a maximum value of 10 ksi in\textsuperscript{0.5} and 1.65 mm(0.065-inch) displacement as the temperature has been reduced to -80 °C. Thus, the \( K_c \) increasing rate depends on the environment temperature and exposure time, which reveals a larger displacement. The declining value in \( K_c \) of the samples was more in comparison to that of exposure to high temperature +60 °C acids. Melting or debonding and the interface between the resin (polymer matrix) and glass fiber is possibly the main cause of that decline [24].

3.2. Mechanism of fracture under humidity and different temperatures

The purpose of this evaluation was to study the fracture surfaces of the composites after fracture toughness testing at various temperatures. Samples were tested at -100 °C, -80 °C, -60 °C, 0 °C, ambient temperature, and +60 °C. Some of these samples are shown in Figure 6.

The sharp increase of fracture toughness index as temperature decreases may attribute to the increase in multiple microcracking and fiber bridging for the post-curing of the polymer matrix during reinforcement. As temperature increases to 60 °C, an obvious decrease in fracture toughness is observed. Figure 5 shows that fracture toughness at 60 °C is 3.5 ksi in\textsuperscript{0.5} at 18.4 mm (0.766 inches) compared to 8.6 ksi in\textsuperscript{0.5} at -80 °C recorded at 1.2 mm (0.048 inches) at the same time of testing. This indicates an over 50% increase in fracture toughness. Based on Eq. (3), the \( E_{IFT} \) for the transparent reinforced polyester composite were evaluated at different temperatures. It was calculated that \( E_{IFT} \) for the tested specimens are 3943, 4252, 3814, 3654, 4765, and 963 J/m\textsuperscript{2} at -100 °C, -80 °C, -60 °C, -30 °C, room temperature and +60 °C respectively. It can be observed that temperature decreasing will influence a high impact on the mechanical property. Basically, for the fabricated transparent composite the interlaminar fracture energy \( E_{IFT} \) increased gradually with decreasing temperature up to room temperature. Owing to the humid environment of testing, raising the temperature promoted the absorption rate of molecular water into the composite from the humid environment at different low temperatures. However, because the glass fiber is hydrophobic to water the latter did not affect the energy of fracture of the composite and did not interfere between the polymer matrix and the fiber [17, 25, 26]. Yet, the energy of fracture for the samples tested at room temperature and -80 °C revealed that the fabricated transparent composite showed superior cold environment resistance. The difference between \( E_{IFT} \) values is very little and my attribute to the tiny cracks developed at -80 °C as fractography showed later. Marom [27] showed that interlaminar fracture energy decreased by 25–30% as the temperature increased from room to 50 °C.

Figures 7 and 8 show the surfaces of each sample at different temperatures and magnifications. These figures show the fracture surfaces of each sample with the fatigue origin notch to the right of each image. Figure 7 shows fracture surface areas near fibers from sample tested at -100 °C; crack arrest (arrow) is shown. The fracture surface of the general area (top) and area away from fibers (bottom) from the sample is shown and smoothness is observed in the bottom image. The fracture
Figure 5. Fracture toughness vs. displacement of polyester composite materials comparing atmospheric to different temperatures: a) 60 °C, b) RT, c) 0 °C, d) -30 °C, e) -60 °C, f) -80 °C, g) -100 °C.

Figure 6. Samples after fracture toughness-testing; from left to right: +60 °C, ambient temperature, -30 °C, -60 °C, and -80 °C.
surface of the general area from the sample tested at -80 °C is shown in Figure 7-b. Fracture surface areas away from fibers from the sample are shown in this figure. Smoothness in top image and crack arrests in bottom image (arrows) are observed. The direction of crack growth is indicated by arrows. The fracture surface of the general area from the sample tested at -60 °C is shown in Figure 7-c. Fracture surface areas away from fibers from sample tested are shown and directions of crack growth are pointed by arrows. The fracture surface of the general area (top) and area away from fibers (bottom) from the sample tested at -30 °C is shown in Figure 7-d. The direction of crack growth is pointed out by crack arrest (arrows). Fracture surface area (top) and fracture origin (bottom) near fibers from sample tested at -30 °C.

Figure 7. a) Fracture at -100 °C b) Fracture at -80 °C c) Fracture at -60 °C d) Fracture at -30 °C.

The fracture surface of the general area (top) and area away from fibers (bottom) from sample tested at 0 °C; the directions of crack growth (arrows) are pointed in Figure 8-a. Fracture surface area near fibers from sample tested and directions of crack growth (arrows) are shown. Figure 8-b shows the fracture surface of the general area (top) and area away from fibers (bottom) from the sample tested at ambient temperature; directions of crack growth (arrows) are shown. Fracture origin (arrow) is observed in this figure. The fracture surface of the general area (top) and area away from fibers (bottom) from the sample tested at +60 °C; note directions of crack growth (arrows). The bottom image shows fracture surface areas near fibers from sample tested and directions of crack growth and crack arrests (arrows). It can be observed that the glass
fiber prompted the fracture resistance at very low temperature and the failure initiates at the interlaminar zone and propagate along the edge of the zone. Similar behavior was reported by Zhang et al. [28] and Vu and Cuong [29].

4. Conclusions

The present work has investigated the influence of a range of environmental temperatures on fracture toughness properties of transparent E-glass fiber reinforced polyester composite materials to manufacture a relatively high transparent, fracture toughness resist and climate change resist polyester composite. According to the present study, it was concluded that the mechanisms of toughening were altered by operation at different temperatures. The manufacturing of a multiphase polyester composite changes the microstructure of the composite network and strength of bonding of the network that leads to modifying the fracture behavior. The enforcing of polyester resin with a volume percent of 15% and curing procedure was responsible for operation below the threshold value of the fracture toughness, \( K_{IC} \), and a satisfactory fracture resistance was obtained at low temperatures. The failure of the composite at high temperature was caused by the phase added to the polymer network, the fiber, that is responsible for initiating the irreversible deformation of the composite. Thus, another important characteristic of the toughening mechanism was the influence of exposure to extreme temperature on yield stress and strain of the polymer matrix-glass fiber structure. The fractography shows that the most notable difference among the samples was the smoothness of the fracture surfaces away from the fibers at lower temperatures. As the testing temperature increased, more fracture features, such as crack arrests, were observed. This may explain why the fatigue marks observed in Figure 8 were more prominent in the lower temperature samples. These samples did not dissipate as much energy locally, so the energy traveled further throughout the composite. Fractures near the fibers appeared to emanate in several directions, also aiding in the dissipation of energy.

Figure 8. Fracture surface areas near fibers from sample tested at a) 0 °C; b) ambient temperature and c) +60 °C.
Declarations

Author contribution statement

Saad R. Ahmed: Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.
Sanjeev Khanna: Conceived and designed the experiments; Wrote the paper.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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