Evaluation of the mechanical characteristics of hygrothermally aged 2-D basalt-aramid/epoxy hybrid interply composites

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Abstract

Polymer composites used in outdoor applications are exposed to environmental factors such as temperature and moisture which may affect the mechanical performance of the composites. In this study, the influence of moisture absorption on the mechanical properties of basalt-aramid/epoxy hybrid interply composites were evaluated. Two different types hybrid interply composites were taken for the investigation namely (30₁₀/0₃/30₁₀) and (45₁₀/0₃/45₁₀). Composites were prepared using compression molding process and cut specimens were subjected to three different ageing environments for 180 days. Selected ageing conditions are, (i) ambient temperature ageing (ii) Sub-zero temperature ageing (-10°C) and (iii) Humid temperature ageing (40°C and 60% Relative humidity). Mechanical tests of the aged composites were carried out to analyse the behaviour of the composites. Moisture uptake of the specimens follow Fick’s law of diffusion with saturation absorption of 5.44%, 3.12% and 1.80% for ambient, sub-zero and humid specimens respectively. Results revealed that (30₁₀/0₃/30₁₀) aged composites possess higher mechanical properties compared to (45₁₀/0₃/45₁₀) aged composites. Highest reduction in properties were observed in ambient aged specimens followed by humid and sub-zero specimens. Scanning electron microscopy (SEM) was employed to observe the damage modes of the fractured specimens. Matrix deterioration, micro cracks and fibre fracture were the major types of failures observed in aged laminates.

Keywords: Basalt-aramid/epoxy composites, ageing conditions, moisture gain, mechanical characterisation, scanning electron microscopy

1. Introduction

Hybrid polymer composites are nowadays extensively used in many engineering applications such as aerospace, automobile, electronic, sports and goods industries. The most promising properties of polymer composites include high strength to weight ratio, low density, corrosion resistance, high endurance limit, improved stiffness etc.[1]. Recently, researchers are showing more interest towards natural/mineral fibre reinforced composites due to rising environmental issues from the general public[2]. Basalt fibres are natural materials extruded from the volcanic rocks after melting at high temperatures. They have properties which are similar to glass fibres and in addition they have good
thermal properties[3]. Basalt fibres in its original form are brittle in nature thus generally hybridised with synthetic ductile fibres such as aramid/Kevlar which makes it suitable for impact applications[4]. Aramid fibres are a family of fibres which have excellent heat-resistant and impact properties which are commonly used in military and ballistic applications[5]. These hybrid composites are exposed to different environmental conditions in outdoor applications. One such scenario is exposure to change in environmental conditions i.e. wet or humid environment at different temperatures[6]. Polymer composites are sensitive to temperature and moisture, under such situations, they engross moisture and experience dilatational expansion. The process of moisture absorption and associated induced stresses due to swelling may result in decline in damage tolerance and durability of the structure[7]. The reliability of the structure and service life performance of polymer composites are intensely reliant on the bond strength of the fibre/polymer interface[8]. Moisture absorption into polymeric composites occurs mainly by diffusion. Transport through voids/micro cracks and capillarity are the other mechanisms of moisture ingestion[9]. The major consequences of moisture absorption are matrix plasticization and swelling. Plasticization results in softening of the matrix and it decreases the glass transition temperature (Tg), whereas swelling is associated to the differential strain produced by the swelling force induced by the moisture while stretching polymeric chains[10]. The presence of moisture in composites can lead to significant variations in the mechanical, chemical and thermophysical behaviour of polymer resin by plasticization and hydrolysis[11]. The extent of moisture absorption of polymer resin is considerably different than that of the fibre. This mismatch in moisture uptake results in volumetric expansion between the matrix and the fibres, and lead to the development of localized stress and strain in composites[12].

Vasudevan et al.[13] explored the influence of surface Kevlar layer orientation on the quasi-static (tensile and flexural) characteristics of Glass-Kevlar/epoxy interply composites. Authors concluded that composites with 30° oriented surface Kevlar layers exhibit higher tensile and flexural strength whereas 45° oriented surface Kevlar layers possess higher percentage elongation. Oguz et al.[14] investigated the effect of hydrothermal seawater ageing on the mechanical characteristics of glass/aramid composites by immersing at 25°C and 70°C for 1000h. Results revealed that reduction in flexural and impact properties was higher at high temperature. Authors also found that flexural strength reduction of hybrid laminates were between the strengths of two plain composites. Ray et al.[15] studied the effect of temperature during humid ageing glass-carbon/epoxy composites. Investigations revealed that elevated temperature increases the moisture diffusion and reduces the ILSS by reducing the interfacial properties. Kumar and Kumar[4] studied the influence of sea water immersion on the performance of carbon and glass fibre hybrid composites. Specimens were exposed to sea water environment at room temperature for 90 days. Results showed that mechanical properties of alternate sequenced hybrid composite was the highest in both dry and sea water aged condition. Several research articles are available on the mechanical behaviour of aramid-basalt/epoxy composites but effect of hygrothermal ageing on the mechanical characteristics of the composites for different orientations of the surface aramid fibre for impact applications has not been reported. As per the past studies, fabricating the composites with aramid as surface sheets is effective for impact applications as it controls the delamination as compared to inner aramid layers[16]. The load bearing inner basalt layers are protected from impact damage by the outer aramid layers. Hence, this work investigates the effect of different ageing conditions on the on the mechanical performance of composites for two different orientations of surface aramid fabrics namely (30/-60) and (45/-45).

2. Methodology

2.1. Materials and composite fabrication

Composites were fabricated using 2-D plain aramid fabrics of 480 gsm and basalt fabrics of 400 gsm incorporated in an epoxy matrix (CT/E 556) and hardener (CT/H 951). Materials were obtained from M/s Composite Tomorrow, India. Hybrid composites were fabricated as shown in figure 1 for two different orientations of surface aramid fabric namely (30/45/30/45) and (45/45/45/45), where, number of layers are denoted by the subscripts and type of material is denoted by the superscripts. For
example, \((30_1^0/0_3^0/30_1^A)\) composites were fabricated by sandwiching three layers of basalt between two layers of surface aramid fabrics. Open mold hand layup followed by compression process was used to fabricate the laminates. Curing was done under room temperature for 24 h. Thickness of the laminates were maintained at 2.8 mm by keeping the spacer blocks during the compression process.

![Figure 1. Schematic representation of interply hybrid composite.](image)

### 2.2. Ageing methods

Initial weights of the prepared composite specimens were measured using a weighing balance of least count 0.001 g. Three different ageing conditions were selected to understand the moisture absorption process and their effect on the mechanical properties of the composites. The following ageing treatment were carried out on the composite specimens. (i) Ageing of specimens at ambient temperature in distilled water. (ii) Ageing of specimens at sub-zero temperature in deep freezer (-10°C and distilled water immersion). (iii) Ageing of specimens at elevated temperature in an environmental chamber (40°C and 60% relative humidity). All the test specimens were subjected to three different accelerated ageing environments for a period of 6 months (180 days). The amount of moisture absorbed by the specimens in terms of weight percentage were measured periodically as per the ASTM D5229[17]. The moisture diffusion in percentage \((M)\) of the specimens at different time intervals can be calculated using the equation (1);

\[
M(t) \, (\%) = \frac{m_t - m_0}{m_0} \quad \text{----------------- (1)}
\]

Where, \(M(t)\) is the % of weight gain by the sample, \(m_0\) is the initial weight of the sample and \(m_t\) is the weight of the sample after time \(t\).

### 2.3. Mechanical Characterization

Mechanical tests were performed after 180 days of ageing to understand the influence of moisture absorption on the mechanical properties of the composites. Four major tests were carried out on the aged specimens viz. tensile, flexural, ILSS and impact tests as per the ASTM standards. Tensile test was performed as per ASTM standard D3039[18] on BiSS make Universal Testing Machine(UTM) of 50 kN load capacity. As per this test, specimens should have dimensions of 250 mm × 25 mm × 2.8 mm (Length × Width × thickness). Flexural tests were conducted on UNITEK-9450 universal testing machine of 50 kN load capacity as per ASTM D7264[19]. For flexural test, length of the specimen is taken as 1.2 times the span length and a standard width of 13 mm. ILSS and Charpy impact tests were carried out on Zwick Roell universal testing machine as per the standards ASTM D2344[20] and ISO 179-1[21] respectively. ILSS required a specimen dimensions of 6t (length)× 15 (width) with a span length of 4t (where t-thickness of the specimen). Specimens were tested at a cross head speed of 2 mm/min for tensile test and 1mm/min for flexural and ILSS. Amount of load applied and the
displacements of the specimens during testing were acquired from the computer connected to the machine. Five specimens from each category were considered for the test and average value was reported.

3. Results and discussion

3.1. Moisture absorption

Figure 2 shows the percentage of moisture gain with respect to time of ageing for three different ageing conditions. Results revealed that the rate of moisture absorption of the specimens is time dependent. All the samples absorbed moisture rapidly at the initial stage and then attained saturation level at the completion of 180 days. Both (30^1_A/0^B/30^1_A) and (45^1_A/0^B/45^1_A) composites showed similar tendency for moisture absorption and followed Fick’s law during initial period of ageing. The moisture absorption rate was highest for ambient aged specimens and lowest for humid aged specimens. Ambient temperature aged specimens absorbed 5.44% of moisture, sub-zero temperature aged specimens absorbed 3.12% and humid temperature aged specimens absorbed 1.80% of moisture after 180 days of ageing.

![Figure 2. Moisture gain versus square root of time](image_url)

3.2. Tensile strength

Figure 3 shows the average tensile strength of aged (30^1_A/0^B/30^1_A) and (45^1_A/0^B/45^1_A) basalt-aramid/epoxy hybrid composites. (30^1_A/0^B/30^1_A) aged composites showed higher strength and stiffness compared to (45^1_A/0^B/45^1_A) aged composites for all the three aging conditions. Maximum amount of deterioration of mechanical properties was seen in the ambient specimens followed by humid and sub-zero specimens. Tensile strengths of (30^1_A/0^B/30^1_A) specimens are found to be 105.1 MPa, 131.35 MPa and 114.26 MPa for ambient, sub-zero and humid specimens respectively. A slight change in surface fibre orientation i.e. (45^1_A/0^B/45^1_A) resulted in decrease in strengths by 3.32%, 14.5% and 7.2% respectively for ambient, sub-zero and humid specimens compared to (30^1_A/0^B/30^1_A) specimens. When the fibres are aligned in the direction of loading, fibres bear the maximum load and as the orientation of the fibres increases, shear stresses will be developed between the laminae which results in extensive delamination. This leads to matrix dominated failures where maximum load will be taken by the polymer instead of fibres which is the primary reason for the reduced loading capacity of (45^1_A/0^B/45^1_A) composites compared to (30^1_A/0^B/30^1_A) composites.
Figure 3. Average tensile strength of aged specimens

3.3. Scanning Electron Microscopy (SEM)

Figure 4 shows the SEM images of fractured tensile test specimens of \((30_1^A/0_3^B/30_1^A)\) and \((45_1^A/0_3^B/45_1^A)\) composites for different ageing conditions.

Higher amount of material deterioration was observed in case of ambient and the humid temperature aged composites due to increased rate of moisture absorption which led to deterioration of matrix dominated properties. For sub-zero specimens, absorbed moistures occupied the voids present in the composites and frozen due to low temperature conditioning. This led to higher rate of de-bonding at the interface and resulted in increased failure strain compared to ambient and humid specimens.
3.4. Flexural strength and Interlaminar shear strength (ILSS)

The three-point bending test results are shown in figure 5(a). The highest flexural strength was observed in (30/0/0/30) composites followed by (45/0/0/45) composites. Ambient specimens absorbed maximum amount of moisture and resulted in maximum degradation of strengths. Maximum flexural strength was observed in sub-zero specimens which is 222.0 MPa followed by 198.41 MPa in humid and 177.18 MPa in ambient aged (30/0/0/30) specimens. A small shift in aramid fabric orientation i.e., (45/0/0/45) resulted in decrease in flexural strengths of 8.52%, 6.5% and 5.87% respectively in sub-zero, ambient and humid specimens respectively.

![Figure 5](image)

*Figure 5. (a) Average flexural strength and (b) Average ILSS of aged laminates*

Similar variations were seen in ILSS test specimens as shown in figure 5(b), with (30/0/0/30) specimens showing highest ILSS which is 12.25 MPa followed by humid and ambient specimens which are 11.89 MPa and 10.26 MPa respectively. (45/0/0/45) aged composites showed 4.60%, 8.72% and 9.1% reduction in ILSS compared to (30/0/0/30) composites. The reduction in mechanical strengths is attributed to combined effect of humidity and temperature which resulted in swelling and degradation of epoxy matrix. The decrease in flexural strengths of aged specimens is due to weakening of interface bond between epoxy and the fibres leading to early failure of composites.

3.5. Impact strength

The ability of a material to resist the failure due to sudden impact is called impact strength. Charpy impact is one among the many low velocity impacts which is used to compare the impact strength of materials. Comparison between the impact strengths of (30/0/0/30) and (45/0/0/45) specimens for three different ageing conditions are shown in figure 6. Impact strength of (30/0/0/30) specimens subjected to sub-zero ageing are found to be highest i.e. 107.6 kJ/m² followed by humid and ambient specimens which are 95.4 kJ/m² and 86.9 kJ/m² respectively. (45/0/0/45) ambient specimens showed least impact strength which is 81.89 kJ/m² compared to all the other conditions. (30/0/0/30) laminates found to distribute the impact damage over larger area from the impacted zone and controlled the delamination which consequently increased the damage resistance compared to (45/0/0/45) composites. Ageing of composites resulted in swelling and reduced the interfacial bonding which led to reduced deformation at the impact region during the impact test.
4. Conclusion
In this study, effect of ageing on the mechanical response of the (30<sub>A</sub>/0<sub>B</sub>/30<sub>A</sub>) and (45<sub>A</sub>/0<sub>B</sub>/45<sub>A</sub>) oriented basalt-aramid/epoxy hybrid composites was investigated. Following conclusions can be drawn from the results obtained.

- The moisture uptake depends on the ageing environment. Ambient specimens absorbed highest i.e. 5.44% of moisture followed by sub-zero and humid which are 3.12% and 1.8% respectively.
- Degradation of material properties were in the order of Ambient > Humid > Sub-zero.
- The reduction in tensile strengths of (45<sub>A</sub>/0<sub>B</sub>/45<sub>A</sub>) specimens are in the order of 3.5%, 14.5% and 7.2% respectively for ambient, sub-zero and humid conditions compared to (30<sub>A</sub>/0<sub>B</sub>/30<sub>A</sub>) aged specimens.
- Maximum degradation in bending strength and ILSS was observed in ambient specimens of (45<sub>A</sub>/0<sub>B</sub>/45<sub>A</sub>) composites followed by humid and sub-zero aged specimens.
- An 5.2%, 8.5% and 4.41% reduction in impact strength was observed in ambient, sub-zero and humid aged specimens of (45<sub>A</sub>/0<sub>B</sub>/45<sub>A</sub>) composites compared to (30<sub>A</sub>/0<sub>B</sub>/30<sub>A</sub>) composites.

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