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Thermal shock effect on Mechanical and Physical properties of pre-moisture treated GRE composite

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Abstract: Many practical situations may be encountered under which a GFRP (Glass fibre reinforced polymer) composite, during its service life, is exposed to the severities of sudden temperature fluctuations. Moisture absorption of GRE (Glass fibre reinforced epoxy) composites followed by various gradients of temperature fluctuations may cause thermo-mechanical degradation. It is on this context, the hand layed GRE composite samples are exposed to up-thermal shock (-40°C to +50°C) and down-thermal shock (+50°C to -40°C) for various time interval after several periods of moisture (hydrothermal/hygrothermal) conditioning. The thermally shocked GRE specimens are put to 3-point bend test to divulge inter laminar shear strength (ILSS). Least ILSS values are recorded for the samples with maximum period of moisture treatments under with both up-thermal and down-thermal shock conditions. Lower glass transition temperature (T_g) values, as revealed through the low temperature DSC test, are exhibited at maximum durations of both up-thermal and down-thermal shock for the samples with higher periods of hygrothermal/hydrothermal treatments. SEM fractographs of representative GRE specimens after optimum period of moisture treatments and thermal shock show the various modes of failures.

Key Words: GRE composite, thermal shock, ILSS, glass transition temperature (T_g)

1. Introduction

GFRP (Glass Fibre reinforced polymer) composites have established their performance for various engineering applications [1-3]. Now-a-days, aerospace structures require polymeric composites for exhibiting excellent specific strength and high corrosion resistance [4, 5]. Such materials during their use in concerned environment are exposed to more or less temperature fluctuation combined with/without moist environment [6, 7]. Thus, durability of FRP composites subject to their application in such stringent environment is a great challenge with respect to lower damage tolerance [8]. Moisture is a prime deteriorating agent for long term sustainable performance of GFRP composites. Moisture induced degradation like plasticization [9] and swelling [10] can reduce the performance of polymer matrix as well as decrease the fibre/matrix interfacial bond strength. In addition, temperature variation can change the moisture absorption rate, which in turn can result local swelling in some
location of the composite body by non-equilibrium moisture absorption/desorption kinetics [11]. Thermal shock due to sudden change in thermal gradient can cause unequal thermal expansion of fibre and matrix [12]. This may result in strain misfit [13] between the components of the FRP composite system and generation of residual stress [14]. A particular example may be found in the aviation industry where an aircraft descends to the ground level from a height, the temperature suddenly changing from the cryogenic level to the atmospheric temperature. It is only desirable to evaluate the composite under such severe ambient conditions to predict its performance and utility. In addition to it, if the composite has prior exposure to moisture coupled with certain temperature above ambience, the ingressed moisture plays a vital role in influencing the performance of the composite under the exposure to thermal shock conditions.

Catastrophic failure of polymer composites may be resulted due to creation of thermal stresses around the defects across the fibre/matrix interface [15]. The environment pertaining to instant variation of temperature is a questionable issue, where temperature of moisture environment affects the relative rates of moisture absorption/desorption process. Post-thermal shock to pre-moisture treated composite may enhance the increasing/decreasing rate of desorption of moisture (which absorbed during moisture conditioning), which may responsible for the fluctuation of inter laminar adhesion strength as well as glass transition temperature ($T_g$) [16, 17]. This may cause diminution of ILSS as well as glass transition temperature ($T_g$) in some cases [18, 19]. Frozen moisture during exposure of the composite samples to sub-ambient temperature may increase the mechanical locking between fibre and polymer matrix. This may cause enhancement of mechanical properties in some cases [20]. The choice of polymer and fibre is motivated by the literature [21, 22].

The present investigation is aimed at monitoring the mechanical and physical properties of pre-moisture treated E-glass fibre/epoxy composite exposed to thermal shock environment (both up-thermal shock and down-thermal shock) for various time periods. The property of pre-moisture treated glass fibre reinforced epoxy (GRE) composite is thoroughly investigated and documented [17]. In the present work, the mechanism of fluctuation of mechanical properties are interpreted with respect to moisture absorption/desorption kinetics during transition of sub-ambient to elevated temperature and vice-versa of thermal shock exposure.

2. Materials and methods

Composite material comprising matrix phase as epoxy resin (LAPOX L-12) and reinforcing agent as E-glass fibre was fabricated by hand laying method. The properties of the constituting components were reported in our previous paper [9]. Diamine (LAPOX K-6) was added to epoxy resin in the proportion of 1:10 by weight for curing purpose. The composite was cured for 48 hours at room temperature. Then, short beam shear (SBS) specimens were prepared adopting ASTM D 2344-84 (1984) reference standard for 3-point bending test.

Two batches of such samples were exposed to moisture treatment (95% of R.H. with 65°C as hygrothermal conditioning for 3 months and immersion of samples in distilled water as hydrothermal conditioning for 4 months). The details of the mechanical and physical properties of GRE composites subject to above mentioned treatments were rigorously investigated in our previous paper [17]. In the present investigation, the moisture treated GRE composite specimens were exposed to thermal shock exposure. For up-thermal shock, different batches of pre-moisture treated SBS specimens of GRE composite were first kept in -40°C in a cryogenic chamber for various time of exposure (1, 2, 3, 4 and 5 hours) and then suddenly transferred to +50°C in electric oven for 2 hours of fixed duration. Similarly, for down-thermal shock treatment, different batches of pre-moisture treated SBS specimens of GRE composite were first kept in +50°C in electric oven for various time periods (1, 2, 3, 4 and 5 hours) and then instantly exposed to -40°C in cryogenic chamber for 2 hours of fixed time. The thermally shock treated samples were put to 3-point bend test in INSTRON-1195 to determine load at peak. ILSS (Inter laminar shear strength) thermally shocked composite samples were also documented by the formula [4]:

$$\text{ILSS} = 0.75 \frac{p_s}{bt}$$

(1)
where, $p_b$ as breaking load (load at rupture in kN), $b$ as width of the specimen and $t$ as thickness of the specimen.

Low temperature differential scanning calorimetry (DSC) test was performed for the representative samples with extreme periods of moisture conditioning and thermal shock treatment. DSC test was carried out by ADSC module of Mettler-Toledo-821 interfaced with STAR software for thermal analysis purpose. Temperature scanning was considered in the range 30-150°C at 10°C/min scan rate. Glass transition temperature ($T_g$) for the test specimens are determined by indicating the first change in DSC thermogram. The fracture surface of the GRE composite samples after 3-point bend test are also imaged through scanning electron microscope (SEM: ZEISS EVO 60) to reveal the various failure mechanism for the sample subject to maximum duration of moisture conditioning and both types of thermal shock treatments of optimum periods.

3. Results and Discussion

3.1 Inter laminar shear strength (ILSS)

Results pertaining to ILSS for all pre-moisture treated (hygrothermal and hydrothermal conditioning) samples exhibit a zig-zag trend for shorter durations of both up-thermal and down-thermal shock followed by a general lowering trend as the duration of thermal shock increases (Figure 1 and Figure 2). Least ILSS values are exhibited for the samples exposed to maximum period of both hygrothermal/hydrothermal moisture conditioning with maximum durations of up-thermal and down-thermal shocks.

![Figure 1](image1.png)

The more or less zig-zag trend of variation in ILSS may be attributed to complex deformation behaviour due to local inhomogeneity owing to local swelling of some locations of matrix and/or fibre/matrix interfacial region. These phenomena are more prominent for lower periods of moisture exposures when the time of exposure is not sufficient to show for average stimuli by the composite body to the above mentioned cause. The samples, however, when exposed to longer periods develop compressive residual stresses resulting in reduction of mechanical properties.

The reason for lowering of ILSS with time of up-thermal shock is many fold i.e., it is the combined effect of many factors. The moisture desorption associated with the thermal shock may form fine whiskers or inter-connected pores when the composite after an initial cryogenic exposure is subjected to +50°C under up-thermal shock conditions [11]. These outlets as a result of moisture desorption can generate residual stresses in the matrix or at the interface, which finally bring in a decreasing trend in ILSS values. The elevated temperature (+50°C) further may generate thermal stresses due to mismatch of thermal expansion coefficients of matrix and fibre. These phenomena may result in decrease in interfacial bond strength giving rise to strain mismatch along the interface. Thus, the ILSS decreases indicating the deterioration of the mechanical property of the composite.

Figure 2 exhibit the variation of ILSS of composite samples exposed to down-thermal shock after pre exposure to hygrothermal/hydrothermal conditioning. It is interesting to see that for lower periods of
exposure to down-thermal shock, the ILSS initially increases. This may be due to matrix hardening [23, 24], the duration at -40°C being almost equal to that at +50°C. However, with increase of down-thermal shock duration the material is exposed to +50°C for a longer time (for all durations of down-thermal shock exposure to -40°C is for 2 hours only). This may result in desorption of moisture causing formation of fine whiskers or inter-connected pores. This is why the ILSS decreases with longer durations of exposure to down-thermal shock. Similar interpretations can be made in the case of hydrothermally treated samples also. As reported out by Loken et. al. [24] and Salin & Sefaris [25], these damage phenomena in composites exposed to cryogenic conditioning consist of pot-holing, delamination and micro-cracking. All such failures are associated with the thermo-mechanical degradation [26] process due to thermal shock exposures.

Figure 2. Variation of ILSS for GRE composite after down-thermal shock treatment
(a) pre-hygrothermally treated (b) pre-hydrothermally treated

3.2 Glass transition temperature ($T_g$)

The DSC thermograms interpreting the $T_g$ values of representative selected samples are shown in Figure 3 and Figure 4. All the $T_g$ values of representative samples (Figure 5) are compared with that of the value of as-cured sample (107.04°C) [17]. The small variations in $T_g$ values for the cases observed is due to minute change of free volume due to hygrothermal/hydrothermal conditioning followed by up and down-thermal shock. Glass transition temperature ($T_g$) is a function of cross-linking density of polymer, affecting free volume. $T_g$ got depressed for all moisture treated samples exposed to higher durations of both types of thermal shock treatments. This result may be observed due to creation of swelling stress during +50°C exposure of final stage of up-thermal shock and breaking of the brittle polymer due to action of excess cryogenic stress during -40°C of final stage of down-thermal shock treatment [4, 16].

Figure 3. DSC thermograms for hygrothermally conditioned samples subject to optimum duration of thermal shock treatments
Figure 4. DSC thermograms for hydrothermally conditioned samples subject to optimum duration of thermal shock treatments

Figure 5. Variation of $T_g$ for GRE composite after minimum and maximum duration of both up and down-thermal shock treatment
(a) pre-hygrothermally treated (b) pre-hydrothermally treated

Though cryogenic compressive stress is responsible for increase of $T_g$ of the polymer [15], the excess amount of same can lower the $T_g$ value up to very small extent by volumetric increase of frozen moisture. The frozen moisture may sometimes tend to get accommodated in the polymer matrix by creating voids or sometimes by breaking up of the epoxy chain structure.

3.3 SEM fractographs
SEM fractographs of pre-moisture conditioned (with maximum periods) composite specimens with concurrent post-thermal shock (minimum and maximum duration of thermal shock treatments) reveal that the mode of failure may be fibre breakage, debonding between fibre and matrix, fibre pull-out, crazing of matrix (cusp formation), matrix cracking etc. (Figure 6, Figure 7). The causes of failure are observed due to anyone of the mentioned modes or any combinations.
4. Conclusions
Mechanical properties of GRE composite sample show initial fluctuating trend due to non-equilibrium kinetics of moisture absorption/desorption process. ILSS of pre-moisture treated GRE composite samples decrease with increasing duration of up-thermal shock. Modulus values for all moisture treated GRE samples are lowered at higher exposing periods of both up-thermal and down-thermal shock treatments, despite of exhibiting initial non-equilibrium increasing trend. GRE composite samples exposed to maximum period of moisture treatment (both hygrothermal and hydrothermal), exhibited early glass transition phenomenon for higher durations of up-thermal and down-thermal shock treatments. The failure modes of thermally shocked GRE composite with pre-moisture conditioning are mostly due to debonding process between fibre and matrix as a consequence of fibre pull-out owing to strain misfit phenomenon.

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