Thermal Shock Behavior of Hydrothermally Conditioned

E-Glass Fiber/Epoxy Composites

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Abstract

The glass fiber reinforced polymer (GFRP) composites suffer from significant moisture absorption properties, moisture due to both the ambience and direct immersion in ordinary water during the course of their practical applications. The quantity of moisture absorbed and its effect on the composite component in any engineering application is also severely influenced by the temperature fluctuations. Hence the present work aims at evaluating the glass fiber/epoxy composite under hydrothermal exposure coupled with up and down-thermal shocks for various lengths of time. Inter laminar shear strength (ILSS) values of shocked and non-shocked composite specimens were determined using INSTRON-1195 testing system. Low temperature differential scanning calorimetry was used to monitor the glass transition temperature (T_g). A longer hydrothermal exposure showed an increase in the ILSS value after an initial low value. The ILSS seemed to vary with up and down-thermal shock differently for hydrothermally treated sample. Despite little variation in T_g value, the depression in the same was observed under up-thermal shock compared to down-thermal shock conditions. The fractographs of shocked and

non- shocked specimen, as obtained from scanning electron microscope (SEM), divulged the chief mode of failure viz. fiber fragmentation and delamination.

Key Words: Glass fiber/epoxy composite, Inter Laminar Shear Strength, Thermal Shock, Glass transition temperature

1. Introduction

Composite materials offer a number of potential advantages in various applications such as aerospace, ship building, automotive and civilian sectors due to their superior properties over many conventional materials like aluminium and steel. Glass fiber reinforced polymer matrix (GFRP) composites exhibit some improved properties like low density, higher specific strength and stiffness, superior corrosion resistance, improved fatigue properties and design flexibility.[1] Fiber reinforced plastics (FRP) plates, symmetrically as well as asymmetrically laminated, are widely employed in a variety of industrial applications.[2] Additional information is needed to obtain the durability related properties of composite materials with applications in various hostile environments.[3] In recent years, the glass fiber reinforced polymers, especially, glass/epoxy composites are widely applied in several civil engineering applications ranging from seismic retrofit of columns and strengthening of walls, slabs to new building frames and bridges.[4]

FRP composites can perform their usefulness in a variety of environments out of which humidity and temperature are considered as more abusive concerning the long term stability of the material. Temperature variation and absorption of moisture can cause irreversible hydrothermal deterioration of the material.[5] It is possible that the presence of moisture in the composite, whether by itself or in combination with other chemicals, can initiate undesirable

structural changes within the fiber reinforcement and the matrix or the interface, and this can potentially reduce the durability of GFRP.[6] The effects of such type of environmental exposure like moisture coupled with elevated temperature on fiber-reinforced polymer composites are of significant concerns for such applications, where the service life can span several decades and little or no maintenance is expected.

Epoxy resin as a potential polymeric material, is used as a matrix in most of the GFRP composites.[7] This resin is very much prone to moisture absorption due to the presence of –OH groups which attract the polar water molecules by hydrogen bonding.[8] Moisture absorption is a matrix dominating property in fibrous composites. The amount of moisture absorbed at the interface also depends on the nature of the glass surface i.e., the lower the functionality of the silane, the higher is the cross-linking density at the interface and lesser will be the amount of moisture absorbed. Moisture penetration at the interface contributes free volume of mesoscopic origin in addition to the one existing in the bulk of the matrix phase.[9]

Higher temperature accelerates diffusion rates of moisture and, generally, hydrothermal ageing process.[10] Epoxy resin absorbs water from the ambience with the surface layer reaching equilibrium with the surrounding environment very quickly followed by diffusion of water into all the material. The water absorbed is not usually in the form of liquid form but consists of molecules or group of molecules linked by hydrogen bonds to the polymer. In addition, liquid water can be absorbed by capillary action along any crack which may be present or along the fiber-matrix interface. The deviation from Fick's law for the diffusion process in glass fiber becomes more pronounced at elevated temperatures and for material immersed in liquids i.e., hydrothermal condition.[11]

Moisture transport in polymers is due to molecular sized holes within the polymeric matrix and to the matrix water affinity. Two states of water may be possible in water-absorbed epoxy system. The unbound free water; filling the voids, does not cause swelling. On the other hand, hydrogen bonded water is responsible for the swelling process in the polymer. The amount of volume changes due to the moisture induced swelling is significantly less than the volume of moisture absorbed. This reveals that a large portion of the absorbed water resides in the voids.[12, 13] The water absorption behavior is affected by two competitive processes: (1) hydrolysis, which accelerates to annihilate water to the diffusion process, (2) Hydrophilisation, which is responsible for the enhanced rate of water uptake causing swelling and plasticization of the composite body.[14]

E-Glass fiber/epoxy composite suffers from degradation of matrix dominating property such as inter laminar shear strength, in-plane shear strength, compressive modulus and fracture strength due to absorption of moisture.[9] Plasticization[15] and swelling[16] of the matrix is referred to as physical degradation of the composite material, where as, chemical degradation includes matrix hydrolysis and fiber degradation.[4, 17] The presence of moisture and the stresses associated with moisture-induced expansion may lower the damage tolerance and structural durability.[15] These effects are generally reversible when the material is removed from moist environment, but high temperature exposure can produce irreversible effects which can cause chemical degradation of the matrix and attack on the fiber-matrix interface which lead to the debonding of the same,[4,19] by promoting the increase in internal voids and chain expansion, thereby affecting the glass transition temperature.[13, 18]

The failure of the bond between the fiber and resin occurs under the influence of temperature gradient as polymers have higher thermal expansion coefficients compared to metals and ceramics. The differential thermal expansion is the main cause of thermal shock in composite materials. The variation of thermal expansion between fiber and polymeric matrix may be responsible for the creation of stresses at the interface.[19] The concentration of thermal stresses around the defects may result in the catastrophic nature of failure of the composites. The instant variation of temperature is a great issue for GFRP composites in such environment.[20] In addition, a change in temperature is certain to bring in changes in relative rates of moisture absorption and relaxation processes in the matrix body.[21] It is therefore imperative to investigate the durability of these materials with hydrothermal environment and concurrent thermal shocks.

Glass transition temperature of polymer matrix composite plays an important role when the durability and long term life are considered. The fibrous composite needs to be put to service at a temperature below T_g where it is both glassy and stiff and can hold the fibers tightly with a well defined appropriate fiber-matrix interface.[4] Hence the agents which lower the T_g of the matrix need to be critically investigated during their service life. Durability data regarding the effects of hydrothermal ageing combined with thermal shock are not abundant and needs to be addressed. It is in this context that the present work aims at evaluating the response of hydrothermal aged glass fiber/epoxy laminated composite to up and down-thermal shocks.

2. **Experimental Details**

The layer based composites were fabricated using the conventional hand lay-up method. The liquid epoxy resin (Araldite LY556) is mixed with a curing agent/hardener (diamine HY951) and applied with a brush and roller taking care to work it into E-type woven glass fiber. After 48

hours of curing, at room temperature, short beam shear test samples (length 50 mm and breadth 12 mm) are cut out from the fabricated composite laminate as per the ASTM D 2344-84(1984) standards. Any moisture that would have entered into the composite body during ambient curing is driven out by heating the specimens in an electric oven at 55^0 C to 60^0 C. The complete removal of moisture is ensured by alternate heating and weighing till a fixed weight is obtained. The laminated composite samples were then immersed in beakers containing distilled water separately for 2, 4, 6 and 8 days. The beakers are kept in an electric oven which was maintained at 65^0C early before the exposure. The percentage of moisture gain was calculated for each duration of exposure.

The percentage of moisture absorption, $M(t)$, can be determined from the weight gain of the specimen, as a function of time, as given by the formula:[22]

$$M(t) = w_t - w_0 / w_0 \qquad (1)$$

where, w_0 and w_t are the weights of the dry and wet specimens at a time t respectively.

An electric oven and a cryogenic chamber (deep freezer) are maintained at $+50^0$C and -40^0C respectively. The hydrothermally treated samples are exposed to up-thermal (-40^0C for 5, 10, 15 and 20 minutes in the cryogenic chamber, then to $+50^0$C for 30 minutes in the electric oven) shock and down-thermal ($+50^0$C for 5, 10, 15 and 20 minutes in the electric oven, then to -40^0C for 30 minutes in the cryogenic chamber) shock, in groups, separately. The shock treated samples, after hydrothermal exposure, were subjected to 3-point bend test using INSTRON-1195 test system to tabulate inter laminar shear strength (ILSS) data. The crosshead velocity was maintained to be 1 mm/min.

Inter laminar shear strength was calculated by the formula: [23]

$$ILSS = 0.75 \times 1000 \left(p_b / bt \right) N/mm^2 \text{ or (MPa)} \tag{2}$$

where, p_b = breaking load (load at rapture), b = width of the specimen, t = thickness of the specimen.

The glass transition temperature (T_g) was measured for the shocked as well as unshocked samples by adopting differential scanning calorimetry (DSC) techniques. The T_g measurements were performed on a Mettler-Toledo 821 with intra cooler using STAR software in DSC module. The experiments were performed in the temperature range of 30^0C-150^0C. Scanning electron microscope (SEM) fractographs are investigated to study the mode of failure in each case. To facilitate a comparison, the characterization of as-cured sample, after hydrothermal exposure, is carried out separately.

3. Results and Discussion

3.1.   Moisture Gain after Hydrothermal conditioning

Data pertaining to moisture absorption, as a function of time, when exposed to hydrothermal conditioning is illustrated in Figure 1. Here the moisture intake increases with time of exposure. The weight percent moisture uptake plots show a continuing uptake trend rather than a plateau even after continued immersion in distilled water at elevated temperature. The driving forces behind the ingress of moisture which involve absorption, diffusion and permeation are the

concentration difference of moisture between the two phases. Thus it is evident that the initial absorption of moisture into the composite bodies in hydrothermal conditioning investigated is concentration dependent and obeys Fick's 2nd law, the absorption of moisture being a direct function of the time of exposure.[24] The experimental data exhibit that, at higher duration of hydrothermal conditioning period, the rate of moisture absorption is much enhanced, notwithstanding the fact that the initial concentration dependent rate of moisture ingression would have decreased subsequently with the concentration gradients decreasing with time progressively with the uptake of additional moisture. In this case, the moisture absorption is anomalous; it can't be explained by Fick's law and is thus Non-Fickian.[25] The continuing trend in moisture uptake is indicative of supplementary mechanism of moisture intake with lengthening of exposing time, establishing the fact that longer exposures only helped in greater degradation of the composite which assists further moisture uptake delaying the saturation level of moisture uptake by the composite body.

3.2.   ILSS of hydrothermally conditioned samples without thermal shock treatment

The illustration, as shown in Figure 2, pertains to the data of ILSS of hydrothermally conditioned glass fiber/epoxy laminated composite as a function of exposure time. The ILSS of as-cured sample was found to be 29.75 MPa.

The reduction in ILSS for initial duration of hydrothermal ageing (up to 2 days) may be related to the weakening effects of higher thermal and moisture induced swelling stresses at the interface and/or in the matrix resin.[15] It may also be hypothesized that initial period of ageing could result

in either breakdown of chemical bonds or secondary forces of attraction at the interface. The further increase in ILSS for consequent duration may be attributed to the partial regeneration of bond strength across the fiber/matrix interface.[9]

3.3.   ILSS of Up-Thermally shocked composites after hydrothermal conditioning

The data relevant to ILSS of hydrothermally exposed sample, after up-thermal shock treatment, are shown in Figure 3.

 The initial increment in ILSS for hydrothermally treated sample for shorter duration of shock, for 2 days, may be attributed to the variation in rate of desorption of moisture (absorbed during the hydrothermal treatment) due to thermal contraction for -40^0C and expansion for $+50^0$C. The rate of moisture desorption is very less in sub ambient region where as the rate is very high in thermal region.[14] Hence maximum of the moisture content is desorbed due to larger thermal gradient for shorter duration of shock. As a result, the sample relaxed due to accommodation of the residual stress generated due to post curing strengthening effect.[19]

   The initial increase in ILSS for 4 and 6 days for hydrothermally treated sample is less as compared 2 days, notwithstanding the fact that the rate of desorption remains same. The amount of desorption is quite low compared to the amount of moisture absorbed during ageing treatment. Hence the stress relaxation is not so much prominent due to lower post curing strengthening as is the case in 2-days of hydrothermally aged sample. The ILSS for the same case (4 and 6 days hydrothermally treated sample) is almost a constant value from shorter to longer duration of shock with some fluctuation within some range of values, which may be attributed to the continuing of stress relaxation up to the longer duration of shock.

For the longer duration of shock, the 6 days hydrothermally treated sample shows a decrease in ILSS. The residual stress generated due to differential thermal contraction and expansion may not be able to accommodate the misfit of strain between the two components of the composite. This misfit of strain may be due to additional interfacial cracking, which may be responsible for the interfacial degradation.[19]

The decrement in ILSS for 8 days hydrothermally treated sample from shorter to longer duration may be analyzed due to the absence of stress relaxation for this situation. Here the amount of moisture desorption during final phase of thermal shock period is very less as compared to higher amount of moisture absorbed during hydrothermal dipping as illustrated in Figure 1. Hence the residual stress generated due to differential thermal contraction (during -40^{0}C) and expansion (during +50^{0}C) is much more than that due to post curing effect for shorter to longer duration of shock. Hence the relaxation of interface between glass fiber and epoxy resin would have been hampered due to non accommodation of large amount of residual stress.

3.4   ILSS of Down-Thermally shocked composites after hydrothermal conditioning

Figure 4 represents the trend relating the variation in ILSS of hydrothermally conditioned composite sample after down-thermal shock.

The increase in ILSS for 2 days of hydrothermally treated sample for shorter to longer duration of down thermal shock may be attributed to the cryogenic hardening of the matrix phase at low temperatures.[26] In the final phase of the shock (-40^{0}C), the residing moisture inside the

specimen, after moisture desorption, gets frozen, which might be responsible for providing mechanical keying between fiber and polymer interface.[1]

The initial slight increase in ILSS in 4 and 6 days of hydrothermally treated sample for shorter duration of shock may be attributed to the formation of strong double hydrogen bond in the epoxy matrix or at the interface.[9] Moisture desorption during initial phase of down-thermal shock may be the driving force for this phenomenon. A fluctuation in ILSS within some range of values for these samples is observed from shorter to longer duration of shock. The sudden change in thermal gradient from sub-ambient to elevated region may have affected the stress relaxation leading to either deterioration or enhancement of mechanical properties.

8 days of hydrothermally treated sample showed the response towards thermal shock as that of 6 days, except for prominent increase in ILSS during the shock period $+50^0C$ (5 to 10 minutes) to -40^0C (30 minutes). This may be attributed to the polymerization of the remaining unopened epoxide molecules by additional ring opening polymerization mechanism during adsorption of moisture by the application of down-thermal shock .[9] The moisture adsorbed during the initial phase of down-thermal shock may have induced the additional ring opening polymerization. This phenomenon is responsible for increase in the amount of cross-linking at the interphase region which helps in the completion of the left curing process.

3.5 Variation in T_g with Up and Down-Thermal Shocks

The variation in T_g as a consequence of exposure to hydrothermal environment followed by up-thermal shock and down-thermal shock including no shock condition are presented in Figure

5. The T_g variation is considered only for the maximum duration of hydrothermal ageing and shock treatment.

The T_g (both onset and midpoint) for 8 days of hydrothermal exposure for up-thermal shock as well as down-thermal shock conditions and no shock condition do not show any considerable variations. This may be due to a similar impact on samples due to moisture absorption and the subsequent thermal shocks. T_g is lowered when the availability of free volume is more. It is an acceptable theory that, at and below the glass transition temperature, $1/40^{th}$ of the total volume of the material is free volume.[4] The invariance in T_g for the cases presented above may, therefore, be due to small or no change in free volume resulting from moisture absorption followed by thermal shock conditions. The invariance in T_g may also have been caused due to plasticization of the matrix, which tends to lower T_g and a simultaneous formation of hydrogen double bonds which tends to elevate T_g as a consequence of moisture absorption.[27]

In a small scale analysis, the T_g value of up-thermally shocked sample is less than the sample exposed to no-shock and down-thermal shock. This may be due to the creation of more free volume in the epoxy matrix during the final phase of up-thermal shock due to epoxy chain swelling and expanding. It is in this context that, up-thermal shock for the composite specimen proved to be more detrimental than down-thermal shock treatment.

3.6 SEM Micrographs

The failure modes have been analyzed and established with the aid of SEM micrographs as presented in Figure 6 through Figure 8.

The micrographs clearly indicate that the mode of failure can be any combination of the following; viz fiber fragmentation, fiber breakage and fiber/matrix de-adhesion.

From Figure 6, it is evident that the detachment of glass fiber from the epoxy matrix is clearly visible. The plasticization with concurrent swelling during maximum duration of hydrothermal exposure may have been responsible for this mode of failure. The fiber fragmentation is clearly visible in Figure 7. The fiber breakage, as illustrated in Figure 8, may have happened due to the generation of cryogenic compressive stresses during the maximum duration of down-thermal shock.

4. Conclusions

The following conclusions can be drawn from this work:

(i)   For shorter exposing time of hydrothermal ageing, moisture ingression into the composite is diffusion driven, i.e., obeys Fick's 2nd law. For the higher conditioning time, the anomalous nature of moisture absorption is observed.

(ii)   As compared to the 'as-cured' sample, ILSS value of samples with hydrothermal exposure decreased initially while showing a considerable increase later on.

(iii)   The hydrothermally treated sample for 8 days after up-thermal shock shows a gradual decrease in ILSS from shorter to longer duration of up-thermal shock.

(iv)   Despite large fluctuation in ILSS values for hydrothermally treated sample after down-thermal shock, increase in the same is observed for 8 days aged sample at some duration

of shock {+50^{0}C (5 to 10 minutes) to -40^{0}C (30 minutes)} due to secondary polymerization mechanism.

(v)   Apart from a slight variation in T_g value for 8 days of exposure to hydrothermal condition after up and down-thermal shocks, the least value is observed for the same aged material after up-thermal shock. Up-thermal shock for the composite specimen proved to be more detrimental than that of down-thermal shock treatment.

(vi)   The principal mode of failures for shocked and no-shocked composite specimens, as obtained from SEM micrographs, are fiber fragmentation, fiber breakage and fiber/matrix de-adhesion.

Acknowledgements

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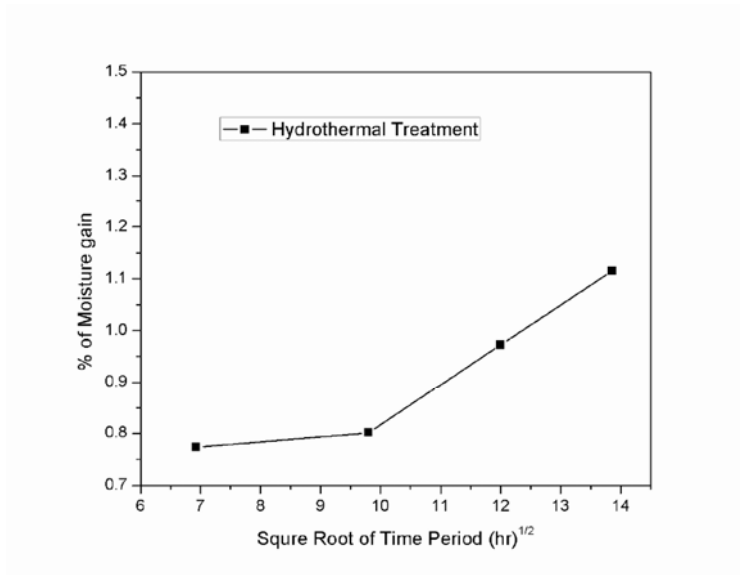
FIGURE CAPTIONS

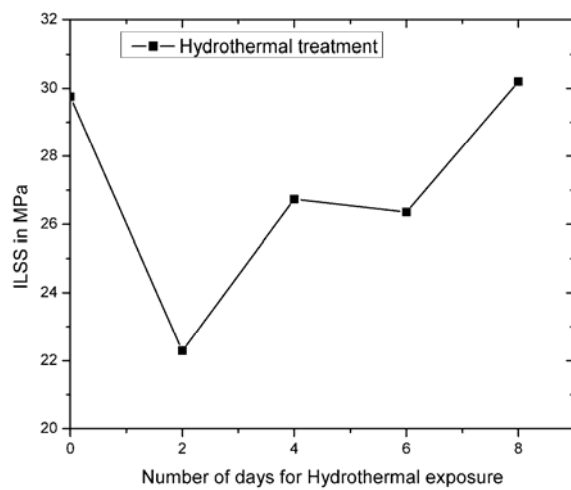Figure 1. Percent of moisture gain for hydrothermal conditioning

Figure 2. Variation of ILSS of hydrothermally treated sample with time (days) without thermal shock
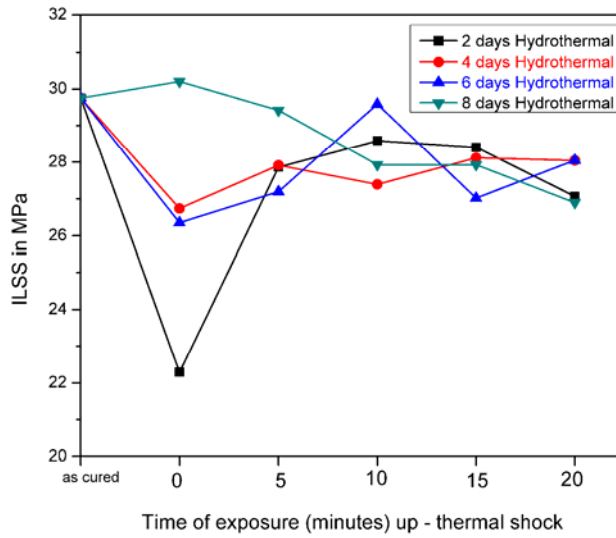
Figure 3. Variation of ILSS of hydrothermally treated sample with the time of exposure of up thermal shock
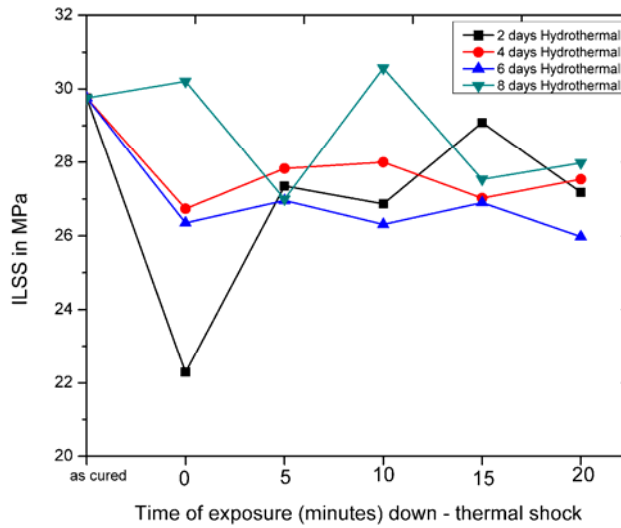
Figure 4. Variation of ILSS of hydrothermally treated sample with the time of exposure of down thermal shock
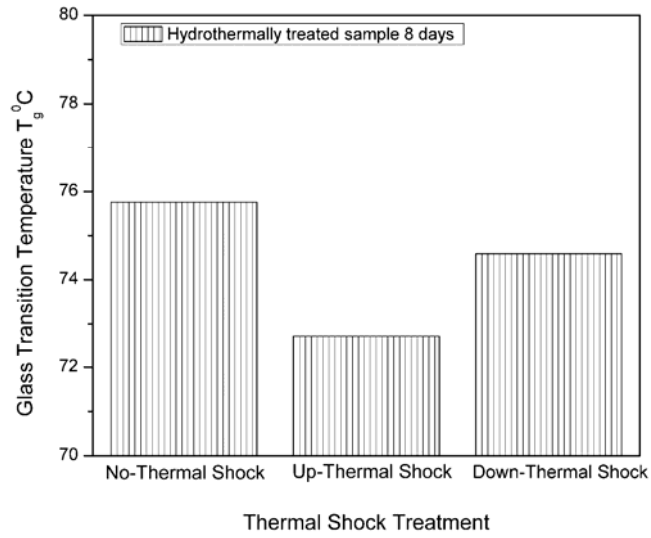
Figure 5. Variation of Glass transition temperature of hydrothermally treated sample with up and down thermal shock treatment
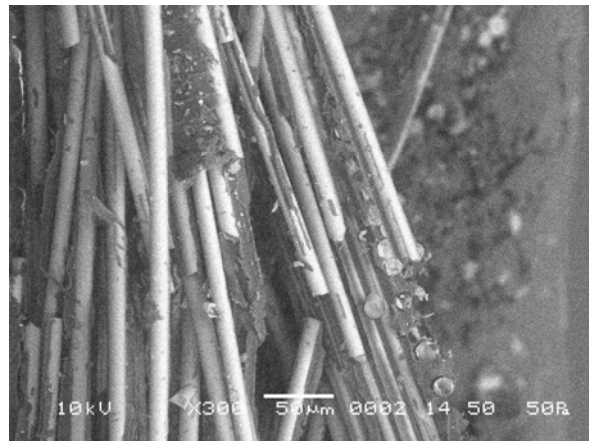
Figure 6. Scanning electron micrograph of 8 days hydrothermally treated sample without thermal shock

Figure 7. Scanning electron micrograph of 8 days hydrothermally treated sample after up thermal shock for maximum duration

Figure 8. Scanning electron micrograph of 8 days hydrothermally treated sample after down thermal shock for maximum duration

Figures

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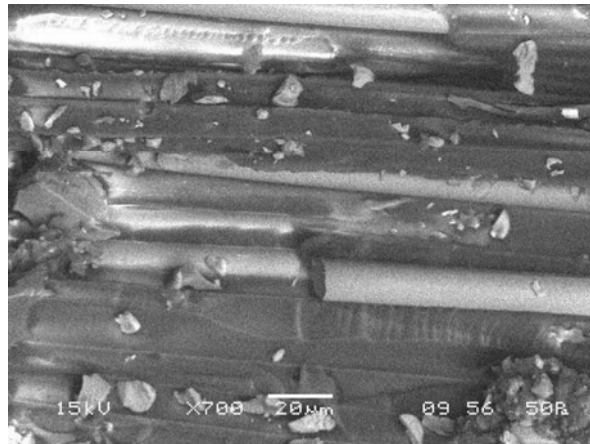
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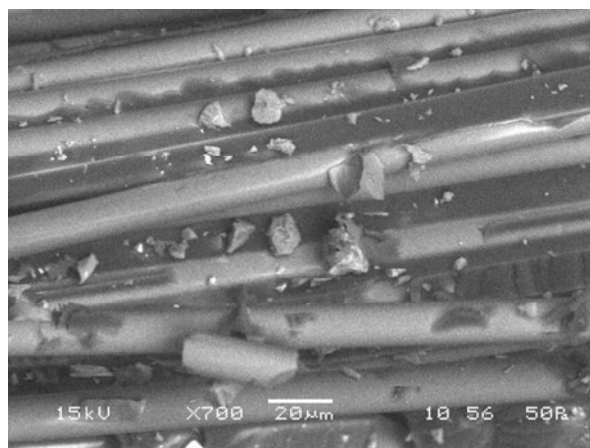

Figure 8. Scanning electron micrograph of 8 days hydrothermally treated sample after down thermal shock for maximum duration