

# Freezing and thermal spikes effects on interlaminar shear strength values of hygrothermally conditioned glass fibre/epoxy composites

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Today increasing numbers of structural components are either being designed with polymeric composites or their metallic counterparts are being replaced by composites. The hygroscopic nature of polymeric systems, however, necessitates a complete understanding of the interaction between structural integrity and a moist environment. Research reports published in the past 20 years have already established the fact that there is a certain amount of degradation of mechanical properties, especially shear, flexural, compression and  $\pm 45^\circ$  axial tensile strengths, when the polymeric composites are exposed to thermal and moist environments [1-3]. It has been reported [4, 5] that the interlaminar shear strength (ILSS) value of glass/epoxy and carbon/epoxy composites decreases with the absorption of moisture. There is an initial increase in the ILSS value of about 10% with 0.1 wt % absorbed moisture for carbon/epoxy composites [1, 4]. Research developments on the hygrothermal response of laminated polymeric composites have shown that absorbed moisture reduces the glass transition temperature of polymeric composites and also causes plasticization, resulting in the reduction of the composite strength properties dominated by matrix characteristics [6, 7].

In this investigation the absorbed moisture of glass/epoxy composites is further treated at  $-4^\circ\text{C}$ , so that this moisture gets frozen. Unfortunately, no work has been carried out to study the response under such conditions in connection with hygrothermal degradation of these new materials. However, such an assessment is necessary on the performance of flight-critical airframe structures. The result shows that there is a further deleterious effect on the ILSS values of such hygrothermally conditioned composites. An effort is also made to study the variation of loading speeds on the response of delamination failure of materials in that condition. Here, also a distinct strain-rate sensitivity of the composites with the presence of frozen moisture was observed. The same effect for the plain hygrothermally conditioned glass/epoxy composites has already been observed and reported [8].

The work was carried out with woven cloth glass fibre and epoxy resin (Ciba LY-556/HY-951), and the weight fraction of fibre in the composites was 0.60. The composites made of these were exposed to moist/thermal environments at  $70^\circ\text{C}$  and 95% rela-

tive humidity (RH). The different batches of specimens were removed from the humidity chamber after different exposure times. Some of the specimens at each stage were exposed to  $-4^\circ\text{C}$  for 24 h. Then three-point bend tests at each stage for both types of conditioned (plain moist and frozen moist) specimens were carried out with an Instron machine. The effect of the loading speed on the ILSS values of frozen moist specimens were observed at 1 and  $10\text{ mm min}^{-1}$  crosshead speeds. The high-temperature excursion was carried out at  $250^\circ\text{C}$  for 10 min (thermal spike), for two series of moist glass/epoxy composites specimens: one hygrothermally conditioned at  $90^\circ\text{C}$  and 95% RH and the other immersed in a hot water bath controlled at  $90^\circ\text{C}$ . Then the variation of ILSS values due to the effects of the thermal spike were investigated.

The ILSS values of both plain moist and frozen moist specimens were recorded for the same level of moisture content and plotted against the square root of the exposure time (Fig. 1). The figure shows that except for the initial period of exposure, the shear strength of specimens with frozen moisture was lower than that of plain moist specimens with the same exposure time. When the moisture inside the materials becomes frozen, this may lead to a further increase of swelling stresses, due to the volume expansion of moisture during freezing, so this treatment turns out to be more deleterious to ILSS values for the same amount of moisture. The initial exception may be due to the strain-free state of the

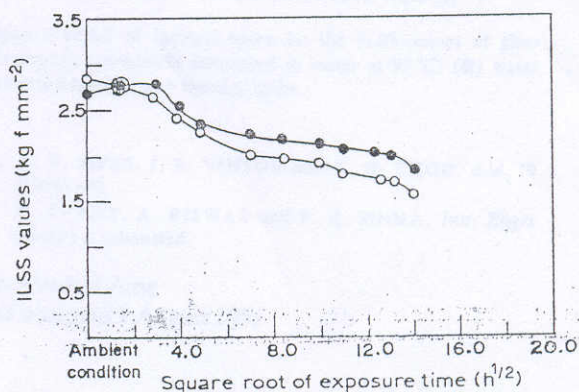


Figure 1 Comparison of the ILSS values of glass fibre/epoxy composites with the presence of (●) plain moisture and (○) frozen moisture. Hygrothermal conditions  $70^\circ\text{C}$ , 95% RH.

composites, as the swelling stress that is developed during freezing can release the residual strains induced by differential thermal contraction during the cooling of the composite from its curing temperature.

Fig. 2 shows the effect of loading speeds on the ILSS values of frozen moist composites, plotted against the square root of the conditioning time. The overall nature of the curve shows that the ILSS values for lower loading speed ( $1 \text{ mm min}^{-1}$ ) is lower than that for the higher loading speed ( $10 \text{ mm min}^{-1}$ ) for the same exposure time. The more detrimental effect for the lower loading speed may be due to the greater time available for the absorbed moisture to diffuse through debonded gaps into the stress-concentrated areas, consequently causing further deterioration of the fibre-matrix interface and/or matrix itself during testing.

Figs 3 and 4 were drawn to investigate the effect of thermal spikes on the ILSS values of composite specimens hygrothermally conditioned at  $90^\circ\text{C}$  and 95% RH, and conditioned by immersion in hot water at  $90^\circ\text{C}$ , respectively. These figures show that the overall effect of thermal spikes is desorption of absorbed moisture, and thereby an increase in the ILSS values compared with those of conditioned specimens without thermal spikes. However, some deviations at the very first periods are observed for both cases (Figs 3 and 4). This may be due to the presence of a higher amount of residual stresses in the composites. For the specimens without thermal spikes hygroscopic (swelling) stresses which are being developed during conditioning counteract the curing stresses, resulting in a lower amount of resultant residual stresses, as these stresses are opposite in nature. This can explain the higher ILSS values of plain conditioned specimens at the initial periods of exposure compared with that of conditioned specimens with thermal spikes.

The present investigation can lead to the following concluding statements. First, when the absorbed moisture becomes frozen, the detrimental effect on ILSS values is greater than that of composites with only moisture. Secondly, the higher loading speeds result in less deleterious effect on ILSS values of glass/epoxy composites with frozen moisture. Finally, a thermal spike at  $250^\circ\text{C}$  of hygrothermally conditioned composites may result in only desorption of absorbed moisture and thus result in less deterioration of the ILSS values.

## References

1. C. M. K. JOSHI, *Composites* 14 (1983) 196.
2. C. H. SHEN and G. S. SPRINGER, *J. Compos. Mater.* 11 (1977) 2.
3. O. ISHAI, A. GARG and H. G. NELSON, *Composites* 17 (1986) 23.
4. B. C. RAY, A. BISWAS and P. K. SINHA, in Proceedings of the Science and Technology of Composites, Adhesives and Sealants Conference, India, 1989, p. 321.
5. *Idem*, *J. Met. Mater. Processes* submitted.
6. E. L. McKAGUE *et al.*, *J. Compos. Mater.* 9 (1975) 2.

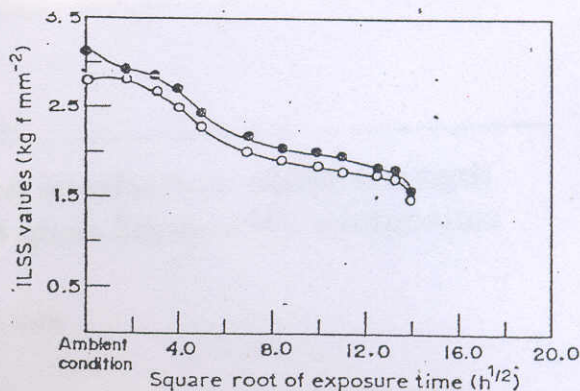


Figure 2 Variation of the ILSS values of glass fibre/epoxy composites with the change of crosshead speed: (O)  $1 \text{ mm min}^{-1}$  and (●)  $10 \text{ mm min}^{-1}$ . Hygrothermal conditions  $70^\circ\text{C}$ , 95% RH.

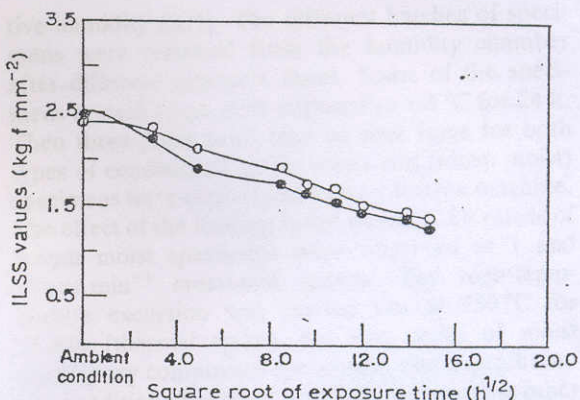


Figure 3 Effect of thermal spike on the ILSS values of hygrothermally conditioned glass fibre/epoxy composites: (●) plain conditioned and (O) with thermal spike. Hygrothermal conditions  $90^\circ\text{C}$ , 95% RH.

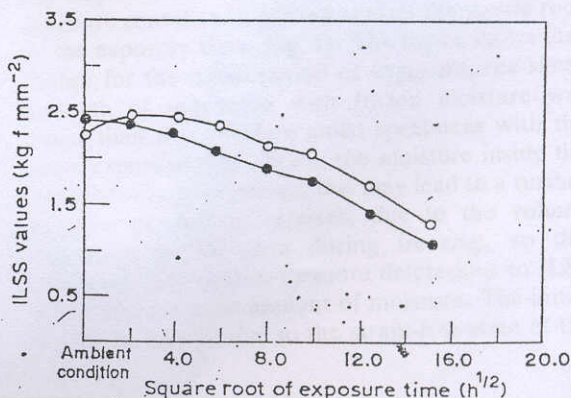


Figure 4 Effect of thermal spike on the ILSS values of glass fibre/epoxy composites immersed in water at  $90^\circ\text{C}$ : (●) water immersed and (O) with thermal spike.

7. R. B. PIPES, J. R. VINSON and T. W. CHOU, *ibid.* 10 (1976) 129.
8. B. C. RAY, A. BISWAS and P. K. SINHA, *Inst. Engrs (India) J.* submitted.

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