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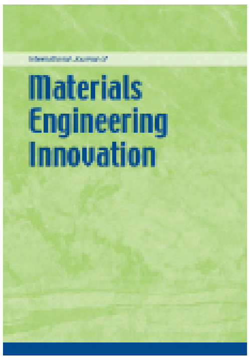

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Effect of elevated temperature on indentation response of glass/epoxy laminates hybridised with milled fibres

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Abstract: This work investigates the effect of elevated temperature on quasi-static indentation response of glass/epoxy laminates. The glass/epoxy laminates with cross-ply $[0/90]_{3S}$ configuration were fabricated by hand lay-up technique and subjected to quasi-static indentation test at elevated temperature of 60°C and 90°C. Furthermore, the glass/epoxy laminates were hybridised with milled glass fibres to enhance the indentation resistance. The contact stiffness, peak load, and permanent dent induced during indentation were evaluated and correlated with unmodified glass/epoxy laminates. Furthermore, the residual load resistance was assessed by conducting three point bending test. Results show that the rise in elevated temperature drastically decreased the peak force, and contact stiffness by an average of 50% and 70%, respectively. Whereas at 30°C, the glass/epoxy laminates modified with milled fibres show greater potential in peak load and contact stiffness by an average of 40% and 50%. Filler debonding/pullout toughening mechanisms attributes to the improvement in peak force, and contact stiffness. It was also found that as the permanent dent grows larger, the residual load bearing capability decreases.

Keywords: indentation response; milled glass fibres; elevated temperature; contact stiffness; residual load.

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1 Introduction

Engineering constructions made of laminated composites have superior properties such as high stiffness, excellent corrosion and fatigue resistance, and light weight. These laminated structures are designed to achieve great strength without facing a significant weight penalty. Composites' tailoring capability ensures that assemblies are created in near-net-shaped components, resulting in faster installation of structures with enhanced durability in a variety of environments (Hamza et al., 2021; Kang et al., 2010; Kumar et al., 2019, 2020; Shruthi et al., 2020). In contrast, these composite structures are subject to accidental impact damage while in operation, particularly in tropical high temperature environments whenever a tool is dropped on the wing (or) debris collides with the fuselage (Kumar et al., 2020; Shruthi et al., 2020; Caminero et al., 2016). Also because aircraft are exposed to temperature ranges between 70°C to 125°C, the risk of low velocity impact damage is likely to occur at elevated temperatures (Caminero et al., 2016; Suvarna et al., 2014; Kumar et al., 2015; Kakakasery et al., 2015).

Furthermore, composites used in aircrafts, ships, and offshore structures are susceptible to transverse loading conditions such as indentation damage during in-service maintenance/repair operations due to interaction/contact with assemblies such as pillars, fixtures, other hard objects/debris, etc. This impact/indentation can happen at the same contact location (or) at an eccentric place in the structures (Simeoli et al., 2014; Bull et al., 2015). Low velocity impact (or) indentation damage events, in general, result in failure modes such as matrix deformation, matrix micro-cracking, interfacial debonding, lamina splitting, delamination, fibre breaking, and fibre pullout (Tong and Isaac, 2008). It was reported that the damage induced in low velocity impact and the quasi-static indentation (QSI) are nearly identical. The contact response between the sample and indenter and the occurrence of sequential damage during loading can be examined in QSI (Flores-Johnson and Li, 2011; Xiao et al., 2007). As a result, it is important to analyse the impact/indentation behaviour as a function of temperature and also evaluate the residual load bearing capacity of composite laminates.

Delamination is typically caused by mode II interlaminar shear stresses caused by local bending during transverse loading. Out of plane properties are frequently described in terms of interlaminar fracture toughness, which is related to polymer matrix fracture toughness and fibre-matrix adhesion (Zhu et al., 2012; Sela and Ishai, 1989). The use of micro/nano-sized fillers can improve the fracture toughness of the polymer matrix. Crack pinning, crack deflection, debonding/pullout, fracture/bridging of filler, and shear banding are all toughening mechanisms provided by these fillers (Zhu et al., 2012; Sela and Ishai, 1989; Kawaguchi and Pearson, 2003; Singh et al., 2002; Lingyu et al., 2009). Because of their nanoscale diameter, greater aspect ratio, high specific surface area, axial stiffness, and strength, nanofillers such as carbon nanotubers (CNTs) have considerable potential to improve the fracture toughness of composites. However, the inclusion of these nanofillers has a negative impact on the mechanical properties of the composites.

It has been observed that additional surface functionalisation processes are needed to achieve uniform dispersion and distribution of these nanofillers in matrix. Agglomeration is further promoted by the filtering of CNTs via fibres and the build-up of these fillers across the micro-channels of the fibres (Tugrul et al., 2008; Himel and Nandagopal, 2018). The flexural strength/modulus, fracture toughness, and critical stress intensity factor of short glass fibres were found to increase with increasing filler content (Hanumantharaya et al., 2019; Pande and Sharma, 1984; Yesgat and Kitey, 2016). Cholake et al. (2015) evaluated the fracture toughness of an epoxy matrix enhanced with milled carbon fibre fillers. They discovered that introducing milled carbon fibre fillers into the epoxy matrix resulted in uniform dispersion, and the critical stress intensity factor (K_{IC}) for filler modified epoxy samples increased by 250% when compared to baseline samples.

Reis et al. (2014) studied the impact performance of glass/epoxy composite laminates and the effect of fire exposure. The maximum load, displacement, and elastic recovery were found to be substantially dependent on impact energy. Furthermore, it was reported that peak load and elastic recovery reduced after fire exposure, while maximum deformation increased. Im et al. (2001) studied the effect of temperature (30°C–120°C) on impact damages such as matrix cracking and interfacial delamination in CFRP composite laminates. It was found that there was a linear relationship between impact energy and delamination area as temperature increased. However, neither impact energy nor impact temperature appears to have a clear relationship with post-impact residual strength.

Suresh Kumar et al. (2017) studied the influence of hybridisation on the impact and post-impact characteristics of composite laminates. QSI tests on quasi-isotropic glass/epoxy, glass/basalt/epoxy (G/B/G, B/G/B), and glass/carbon/epoxy (G/C/G, C/G/C) laminates with acoustic emission monitoring were used to simulate low velocity impact behaviour. They concluded that adding basalt fibre and carbon fibre to glass fibre improved indentation damage resistance. Djele and Karakuzu (2021) investigated the effects of temperature, quasi-static crosshead speed and impact energy on the behaviours of quasi-static punch shear and low velocity impact punch shear. S2 glass/epoxy and carbon-Kevlar hybrid composite laminates were subjected to punch shear using a flat nose impactor. An increase in contact force was observed with increasing impact energy and the effect of temperature had a significant effect in damage mode.

The effect of temperature on QSI and low velocity impact behaviour of laminates were experimentally investigated by Karakuzu et al. (2021). Carbon/Kevlar hybrid composites and S2 glass/epoxy composites were subjected to different impact energy levels and crosshead speeds. They found that the energy required to perforate the samples at a specific crosshead speed was at the same level compared to low velocity impact test. Also, it was reported that the energy level had a significant effect on impact damage whereas the effect of crosshead speed and temperature on impact damage was insignificant. Further, Jefferson et al. (2016) studied the effects of post-cure temperature and fibre reinforcements on the multiple QSI response of repaired glass/epoxy laminates. The inclusion of reinforced fibres in the polymer matrix hindered crack propagation under out-of-plane loading, according to the results. However, the indentation response of the repaired laminates was affected by the type of reinforcing material used and the post-cure temperatures.

Several studies have been conducted on the effect of environmental temperature on the impact loading behaviour of composite laminates. In contrast, only a few studies have been conducted on QSI loading at elevated temperatures. The present study focuses on the influence of elevated temperature on the QSI behaviour and residual performance of glass/epoxy laminates modified with milled glass fibres. The previous study found that incorporating milled glass fibres into glass/epoxy composites by 5% weight of epoxy increased flexural strength, mode I and mode II fracture toughness (Saravanakumar et al., 2022; Kannivel et al., 2020; Kumar et al., 2021). The glass/epoxy samples were subjected to a QSI test at various depths of 3 mm to 6 mm, respectively. The test parameters of peak force, absorbed energy, residual dent, and damage area were examined, and the findings were compared to the baseline samples. The residual load bearing capability of the indented samples was also determined using a three point bending test.

2 Experimental procedure

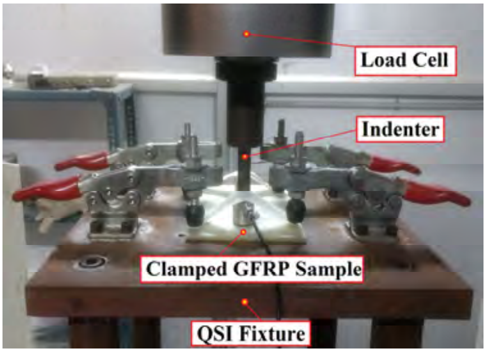
2.1 Materials and fabrication of composite laminates

Unidirectional 220 GSM glass fabrics (12 layers) and LY556 epoxy resin with HY951 hardener were used as raw materials in a weight-to-weight ratio of 1:1 to fabricate the laminates. Hand lay-up technique was used to fabricate the glass/epoxy laminates, which had a cross-ply stacking sequence of $[0°/90°]_{3S}$ configuration. Milled glass fibre fillers were added to the epoxy resin (by 5% weight of epoxy) using sonication and mechanical stirring to ensure uniform distribution of the filler in the resin. Later, the liquid was degassed to remove any trapped air bubbles (Yesgat and Kitey, 2016; Cholake et al., 2015). The hardener was added to the mixture at a weight-to-weight ratio of 1:10 and mixed to begin the curing process. To impregnate the fibres, the epoxy matrix mixture was evenly dispersed using a brush and roller. Similarly, the baseline laminates without milled glass fibre fillers were created as fabricate above. All laminates were allowed to cure for 24 hours at room temperature. The samples were cut from a 500 mm × 500 mm manufactured laminate using an abrasive water-jet cutting machine. The fabricated laminates have a nominal thickness of 4 mm (± 0.25 mm).

2.2 QSI test

The QSI was performed at a cross-head speed of 1 mm/min on the Tinius Olsen 100 kN universal testing machine (UTM). The test was performed using an indentation fixture in accordance with the ASTM D6264-98(04) standard (Suresh Kumar et al., 2017). As illustrated in Figure 1, the glass/epoxy samples with dimensions of 150 mm × 100 mm were rested on the fixture and clamped on both sides. Later, an indentation test was performed directly on the samples' centres. The specimen was indented with a hemispherical end steel tup having a diameter of 12.7 mm. The UTM's load-displacement data was recorded using a digital data acquisition system. In each case, four specimens were tested, and the average results were used. The QSI tests were performed at specified indentation depths of 3 mm, 4 mm, 5 mm and 6 mm. Temperatures of 30°C, 60°C and 90°C were used for the QSI loading. The indentation behaviour and test parameters such as indentation force, residual dent depth, energy absorbed, contact stiffness, and damage area size were examined for varied indentation depths.

Figure 1 Glass/epoxy samples in QSI test fixture (see online version for colours)



2.3 Three-point bending test

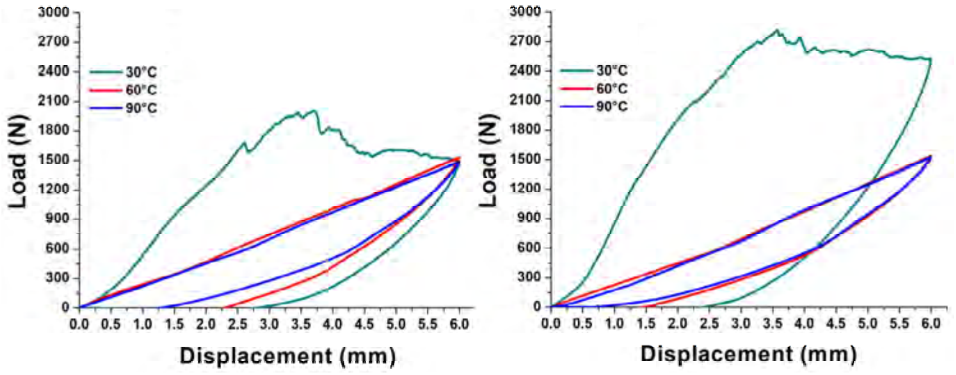
The flexural test was carried out at room temperature on the post-indented glass/epoxy samples using a displacement control regime. To perform the flexural test, the post-indented samples were reduced to 150 mm × 50 mm using a diamond saw. During cutting, care was taken not to damage the indented zone (Saravanakumar et al., 2018). The tests were performed at room temperature using a 100 kN Tinius Olsen UTM with a constant cross-head speed of 1 mm/min. The span length was kept constant at 100 mm, and four repetitions were performed for each sample category. The test measured the residual flexural load, and the results were correlated with the non-indented samples.

3 Results and discussion

3.1 QSI loading

The typical load-displacement behaviour of glass/epoxy composite laminates subjected to indentation loading at different temperatures is shown in Figure 2. Continuous damage progression occurs as the depth of the indentation increases, with a quick change in slope and abrupt load drop due to delamination and fibre breaking, respectively. The material's contact stiffness is indicated by the initial slope of the load-displacement profile. Figure 2 reveals significant variations in the load-displacement profile. There are significant changes in loading behaviour between baseline and modified samples indented at room temperature. Both baseline and modified samples indented at elevated temperatures, on the other hand, show no substantial changes in loading profiles.

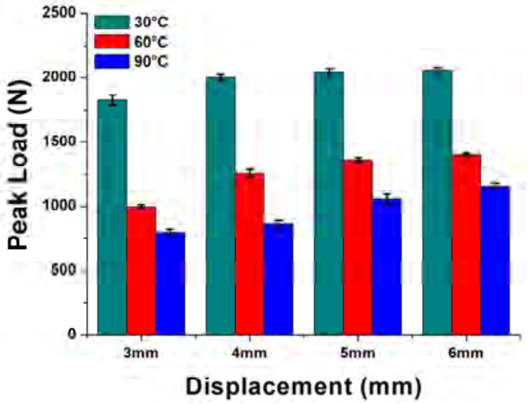
Figure 2 Load-displacement response of glass/epoxy samples at different temperatures, (a) baseline (b) modified (see online version for colours)



(a) (b)

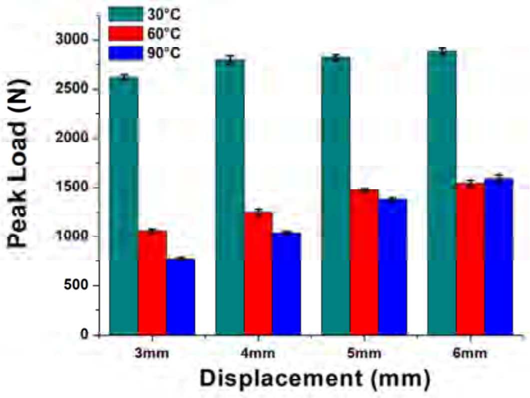
The peak load exhibited by baseline samples is shown in Figure 3. It can be seen that when the indentation displacement increases, the peak load increases. The load resistance offered by the laminates decreased with increasing elevated temperature. At 60°C, the reduction in peak load was only about 36%. Whereas at 90°C, a substantial reduction in peak load of about 50% was observed in both the cases of glass/epoxy samples (shown in Figures 3 and 4). This result indicates that the indentation load resistance is significantly affected by the elevated temperature. The peak load of samples decreased as the temperature increased from 30°C to 90°C due to polymer softening and interfacial dilatation at the fibre/polymer interface. As the temperature increases, the maximum change in mobility of the polymer chains occurs, allowing the material to bear no load without deformation (Kumar et al., 2017; Puneet and Srinivas, 2018).

Figure 3 Peak load of baseline glass/epoxy samples at different temperatures (see online version for colours)



The peak load of modified glass/epoxy samples is depicted in Figure 4. The glass/epoxy laminates hybridised with milled fibres exhibit a considerable increase in peak load in all of the cases. The inclusion of milled fibres resulted in a nearly 40% increase in peak load at 30°C. This increase in peak force was attributed to the toughening mechanisms of filler debonding/pull-out (Saravanakumar et al., 2022; Kannivel et al., 2020). At 90°C, however, the increase in peak load was only 20%, indicating that load carrying capability diminishes with increasing temperature. This was linked to polymer degradation and interfacial bonding weakening at the fibre/matrix interface area. The rate of degradation increased with temperature, decreasing the stress transfer efficiency at the interfacial area (Kumar et al., 2017).

Figure 4 Peak load of modified glass/epoxy samples at different temperatures (see online version for colours)



As discussed earlier, the samples were indented for predefined displacement of 3 mm, 4 mm, 5 mm and 6 mm, respectively. The residual dent experienced by baseline and modified glass/epoxy samples are shown in Figures 5 and 6, respectively. When the indentation displacement increases, the residual dent also increases in all cases. With increasing elevated temperature, the permanent dent induced on the samples decreases. In baseline samples indented at 60°C, the residual dent decreased by an average 25%. Whereas at 90°C, about 45% decrease in residual dent was observed. This result evidence that the increasing elevated temperature causes the suppression of delamination progression and therefore resulting in minor residual dent and damage area (shown in Figures 9 and 10).

Figure 5 Residual dent of baseline glass/epoxy samples (see online version for colours)

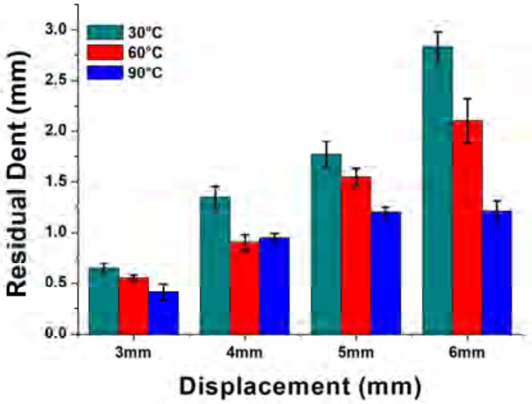
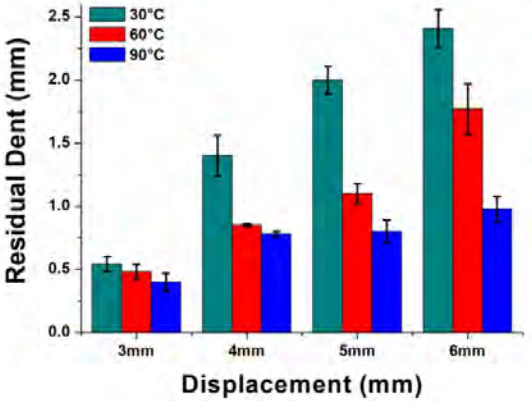


Figure 6 Residual dent of modified glass/epoxy samples (see online version for colours)



In all cases of filler modified laminates show 15% decrease in permanent dent (shown in Figure 6). The permanent dent induced on the samples decreases with increasing elevated temperature. The residual dent in modified samples indented at 60°C was reduced by roughly 15%. In comparison to samples indented at 30°C, a 45% reduction in residual dent was seen at 90°C. This was attributed to the polymer softening and interfacial dilation at the fibre/polymer interface. As the temperature increase, the matrix becomes plastic in nature which affects the load transfers between the fibre/matrix. Hence, the samples exhibited reduced residual dent at elevated temperatures (Kumar et al., 2017; Puneet and Srinivas, 2018).

The contact stiffness of baseline and modified glass/epoxy samples are shown in Figure 7. At 30°C, the glass/epoxy laminates hybridised with milled fibres exhibited 50% improvement in contact stiffness. The mechanisms of filler debonding/pullout toughening contribute to the increase in peak force and contact stiffness (Saravanakumar et al., 2022; Kannivel et al., 2020). Both the baseline and modified glass/epoxy samples show that the contact stiffness decreased with increasing elevated temperature. The reduction in contact stiffness due to increase in temperature was about 70%. Peak load and stiffness was found to be significantly reduced at proximity of glass transition temperature (Karakuzu et al., 2021). At elevated temperatures, the mobility of the molecules in the polymer chains increases significantly, increasing local deformation and, consequently, reduced peak load and stiffness of the material.

Figure 7 Contact stiffness of glass/epoxy samples (see online version for colours)

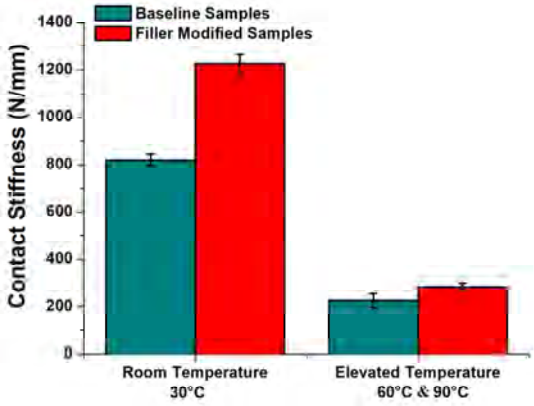


Figure 8 Relationship between the peak load and residual dent (see online version for colours)

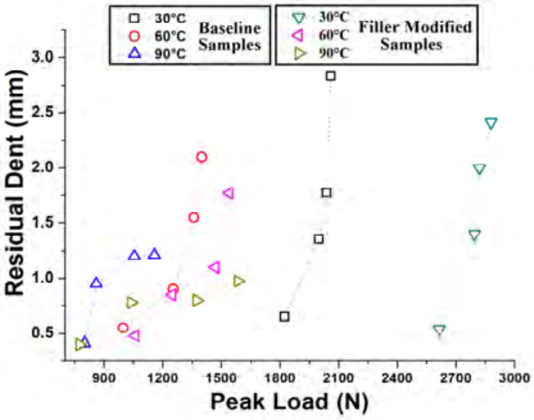


Figure 9 Damage area of baseline glass/epoxy samples (see online version for colours)

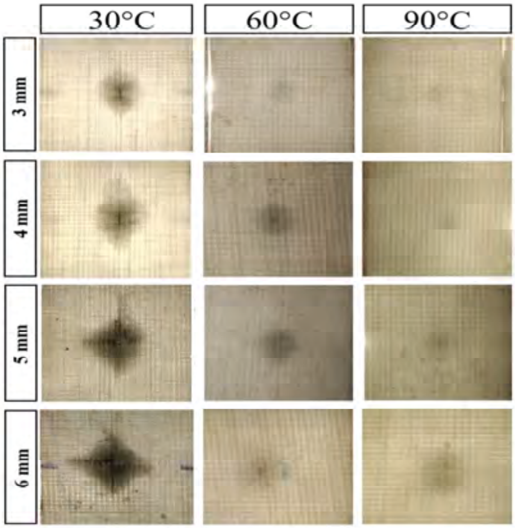
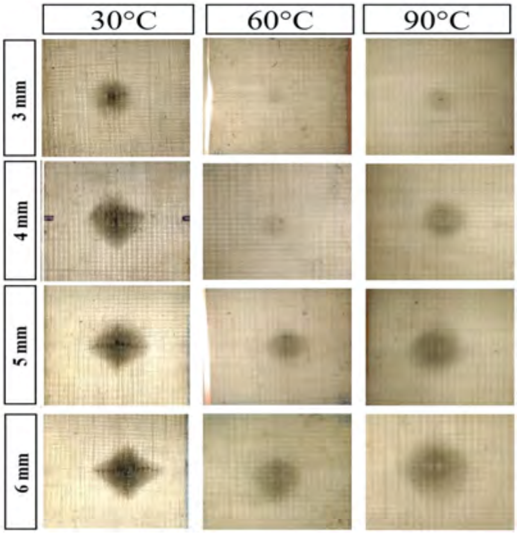


Figure 10 Damage area of modified glass/epoxy samples (see online version for colours)

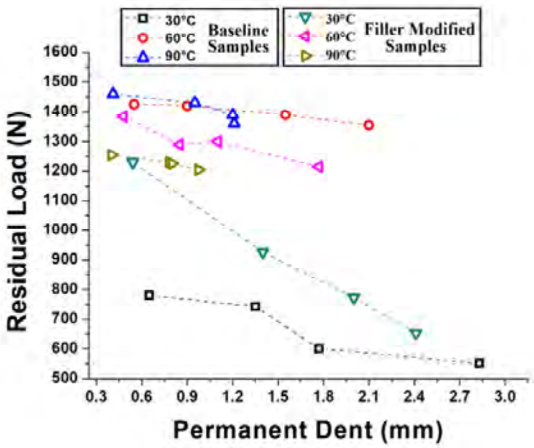


The relationship between the peak load and residual dent of glass/epoxy samples are shown in Figure 8. In all cases, the samples modified with milled fibres have greater peak force with minor residual dent than the baseline samples. Typically, the permanent dent increases with peak load. The permanent dent and damage area induced on the samples at 30°C, 60°C and 90°C are shown in Figure 9. As the temperature increases, the overall load transfer efficiency degrades, resulting in lower peak load and lesser residual dent. It is evident that the damage area expands with increasing depth of indentation. However, when the temperature increases, the size of the permanent dent and damage area diminished. Polymer softening and glass fibre/polymer interfacial thermal stress are responsible for the reduced peak load and stiffness at elevated temperatures. Subsequently, the modified samples indented at 30°C show less damage area than baseline samples as shown in Figure 10. On the other hand, the filler modified samples indented at elevated temperature show slight increase in damage area than the baseline samples. This variation in damage size was attributed to improved energy absorption and peak load exhibited by the modified samples.

3.2 *Post-indented performance*

The post-intended performance of baseline and filler modified glass/epoxy samples were evaluated by conducting flexural loading (Kannivel et al., 2020; Kumar et al., 2021). The residual load bearing capacity of glass/epoxy samples were evaluated by performing three point bending test. Figure 11 depicts the relationship between the permanent dent and residual load bearing capacity of glass/epoxy samples. This plot relates the load bearing capacity of indented baseline and modified samples. The permanent dent is the residual dent created on the sample after indentation. It can be concluded that when the permanent dent increases, the residual load bearing capacity decreases. At 30°C, both samples showed a significant loss in load bearing capacity. However, the reduction in residual load was minimal at elevated temperature.

Figure 11 Relationship between the permanent dent and residual load (see online version for colours)



The residual load carrying capacity of baseline and modified samples are shown in Figures 12 and 13, respectively. The residual load bearing capability of samples indented at 30°C decreased substantially with increasing indentation depth. In contrast, this reduction was insignificant in samples indented at elevated temperature. At 30°C, the glass/epoxy samples hybridised with milled fibres exhibited almost 40% improved residual load than the baseline samples. However at elevated temperature, the modified glass/epoxy samples showed a minor decrease in residual load. This observation was attributed to the flexibility of polymer matrix during elevated temperature.

Figure 12 Effect of temperature on residual load of baseline sample (see online version for colours)

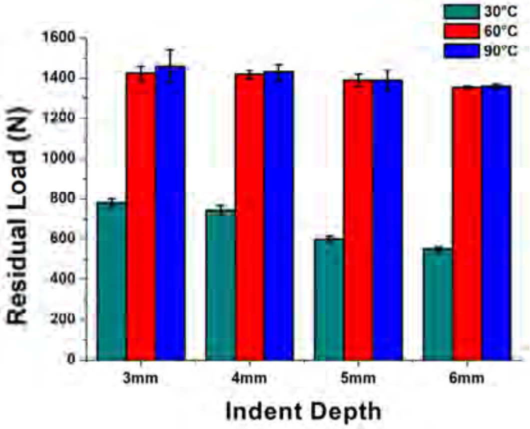
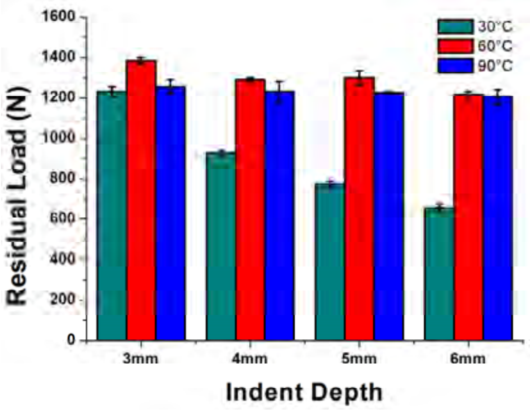


Figure 13 Effect of temperature on residual load of modified sample (see online version for colours)



At extremely high temperatures (90°C), substantial polymer softening occurs in the near glass transition temperature zone, reducing interfacial adhesion and causing interfacial debonding. Increase in temperature causes poor adhesion between fibre and matrix interfaces, resulting in poor load transfer and formation of additional cracks (Kannivel et al., 2020; Kumar et al., 2021). The reduction in residual load was attributed to the premature progression of indentation damage. The samples indented at 30°C possess more delamination damage, consequently resulting in poor residual load compared to samples indented at 60°C and 90°C which was evidenced in Figures 9 and 10.

4 Conclusions

In this research work, the effect of elevated temperature on QSI response of glass/epoxy laminates was investigated. Hand lay-up technique was used to fabricate the glass/epoxy laminates with a cross-ply $[0/90]_{3S}$ configuration, which were then subjected to QSI tests at 60°C and 90°C. Also, to improve indentation resistance, the glass/epoxy laminates were hybridised with milled glass fibres. The contact stiffness, peak load, and permanent dent induced during indentation of unmodified glass/epoxy laminates were evaluated and correlated. Furthermore, a three-point bending test was performed on the samples to determine the residual load resistance. It was concluded that the peak force and contact stiffness were both reduced by an average of 50% and 70%, respectively, as a result of increase in elevated temperature. At 30°C, however, the laminates modified with milled fibres show a 40%and 50% increase in peak load and contact stiffness, respectively. It can be concluded that when the permanent dent grows larger, the residual load bearing capability decreases.

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