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Thermo-mechanical debonding map for repairing composite-metal adhesive joints

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ABSTRACT

Adhesive bonding is one of the dominant joining technologies in Design for Assembly to obtain innovative engineering products with complex geometries. These permanently bonded joints could be disassembled for repair purposes using the thermo-mechanical debonding technique to salvage the high-cost composite components without any damage. In this work, the temperature dependent storage modulus, loss modulus and tensile properties of the bulk modified epoxy film adhesive were measured through instrumented tests. Then, the glass fibre reinforced composite and Ti-6Al-4V alloy sheets were bonded with the modified epoxy film adhesive and the floating roller peel performance was studied as a function of temperature to understand the load-displacement and failure mechanisms for generating a debonding map. The debonding region was identified in the temperature range between $1.17T_g$ and $1.33T_g$. The knock-down in storage modulus and initial peel load exhibited a similar trend with respect to the temperature.

Keywords: Debonding; Hybrid joints; Floating roller peel; Composite repair

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1 Introduction

Hybrid light-weight structures such as fiber-metal laminates are made up of long fiber reinforced polymers and thin sheets of metals which have a synergetic effect of both composites and metals such as high strength, lightweight, good fatigue life and better impact damage tolerance [1]. However, these joints need to be repaired in various cases (such as improper bonding during production, foreign object damages in service, etc.) and reassembled again to serve its purpose [2]. Debonding technologies are gaining more attention in the after-market services domain which would help in separating the adhesively bonded adherends and thereby benefitting the economy and environment [3–5]. They can be classified broadly into two categories, namely conventional and smart techniques. Mechanical separation, thermal degradation, thermo-mechanical, electrical and chemical methods are few examples of conventional techniques whereas the use of debondable-filler modified adhesives and Di-Alder adhesives are considered as smart techniques. The latter one comprises of the adhesives that modified with foaming agents, magnetic particles, carbon nanotubes, electrolytes, expandable graphite flakes or functional scrims to aid clean and easily separable joints [6–11]. The selection of the appropriate debonding method depends on the nature of the adhesive assembly including the volume, adherend materials, operating conditions, and joint accessibility.

Paste adhesives can be easily functionalized with the debonding fillers but it would knock-down the mechanical performance of the joints [12,13]. Constraints in debonding filler selection and modification of structural film adhesives make thermo-mechanical debonding one of the feasible methods for disassembling the adhesively bonded joints.

Thermo-mechanical debonding is a hybrid process of applying both mechanical and thermal loads to disassemble the adhesive joint. It requires the knowledge of temperature dependent mechanical behaviour of the joints and the corresponding failure modes. An epoxy adhesive

designed for use at ambient temperature (20°C) may be strong and brittle at lower temperatures but would have low ultimate tensile strength, exhibiting ductility at higher temperatures [14]. The Mode-1 fracture toughness is insensitive to the temperature range below the glass transition temperature (T_g) and decreases significantly above T_g [15]. This is due to the increased mobility of the polymer chains in the adhesive at high temperatures. The single lap joint's (SLJ) shear strength depends on the bulk adhesive strength. At higher temperatures, the internal joint stresses are redistributed as: (a) the edge peak stresses are lower due to adhesive yielding and (b) due to ease of shear deformation of larger quantity of adhesive in the bond line [16]. SLJs made up of carbon fiber reinforced polymeric composite (CFRP) and Ti-6Al-4V adherends exhibited poor joint strength at elevated temperatures, especially at the T_g of epoxy film adhesive. The SLJ's strength was 25 MPa at room temperature and decreased to 8 MPa at T_g , as the failure mode progressed from adherend failure to adhesive failure [17]. The key process parameters in thermo-mechanical debonding technique such as debonding force and temperature should be optimized for the identified practical constraints and failure modes.

Among the various peel tests, floating roller peel is a suitable test method for assessing the adhesion strength of the hybrid adhesive joints having mismatch in strength and stiffness [18–22]. The peel force (F) is commonly denoted as the force required to separate per unit width (w) of the bonded adherends. It is the sum of (a) force required to debond the adhesive joint (Jw), where J is the adhesive fracture energy rate, (b) frictional force between the roller and thin metallic adherend, (c) force required to perform the plastic work on thin metal adherend and (d) the alignment weight (D) [23]. The test can be extended to identify the associated debonding process parameters.

In this study, the thermo-mechanical properties of the bulk modified epoxy film adhesive were studied using dynamic mechanical analyzer and tensile testing. Eventually, a debonding map is developed based on the floating roller peel (FRP) strength and failure modes of the tested

joints at different temperatures across the glass transition temperature of adhesive (T_g). This would salvage the high-value composite adherends for repair and remanufacturing purposes.

2 Materials and manufacturing

Bulk modified epoxy film adhesive in a particular geometry were fabricated for conducting dynamic mechanical analysis (DMA) and tensile tests, whereas composite adherends were manufactured for fabricating the hybrid bonded joints. The modified epoxy film adhesive procured from 3M® was thawed until it reached the room temperature (23°C). The exact chemical composition of the adhesive is unknown and kept confidential due to the proprietary reasons. It is used in aerospace applications requiring high toughness and service temperature up to 121°C. The adhesive layers were vacuum degassed for every two layers and placed inside a rectangular mould. After vacuum-bagging, the entire set-up was cured in an autoclave, using a curing cycle with dwell temperature of 121°C for 60 minutes, under a pressure of 310 kPa. The cured adhesive plate was cut into sample size of 35 mm x 10 mm x 3 mm. Similarly, the dog-bone type IV tensile test specimens were fabricated according to ASTM D6318-14 [24].

An 8 harness satin weave, E-glass /M21 epoxy prepeg was used to fabricate the composite adherends as stipulated by Hexcel. Mechanical properties of the composite adherend can be referred from the technical data sheet [25]. The 0.4 mm thin metallic adherend made up of grade 5 Ti-6Al-4V alloy has Young's modulus of 117 GPa and tensile strength of 1000 MPa. The surface preparation of the metallic adherend as follows: (a) manually dry-grit blasted with white alumina powder of 120 μ m grit size, (b) washed with tap water to remove the sand particles and observed no water break-down on the surface to confirm the wettability, (c) dried at 60°C for 30 minutes in a convective oven, (d) degreased with acetone soaked lint-free cloth, (e) sol-gel [26] treated with a mixture of zirconium n-propoxide, acetic acid and propyl alcohol (AC-130-2), (f) and primed with EW5005 [27] and cured as recommended by the supplier. The

GFRP composite adherend surface was dry-grit blasted, washed with tap water and finally degreased with acetone.

The sample preparation is same as explained in our previous study [21]. The surface treated GFRP composite and Ti-6Al-4V alloy adherends were bonded by a layer of modified epoxy film adhesive and cut according to the ASTM D3167 standard [28]. The test specimen dimensions can be referred from Figure 1.

3 Experimental methods

The glass transition temperature plays an important role in the thermo-mechanical debonding process as the modified epoxy film adhesive is expected to have adverse mechanical properties when compared to the temperature range within the service limit. Similarly, the tensile stress-strain behaviour at different temperature would indicate the change in the tensile strength and ductility of the adhesive. However, the debonding force is dependent on the temperature, joint design and loading parameters. So the DMA and tensile tests were conducted using bulk adhesive test specimens whereas the floating roller peel test was performed on the adhesively bonded hybrid joints. The test setup and parameters are described in the following subsections.

3.1 DMA testing of adhesive

All the cured modified epoxy film adhesive samples were tested on a DMA Q800 supplied by TA Instruments, Singapore as per ASTM standard [29]. The sample was well positioned in a single cantilever clamp and tightened to 1.13 Nm using a torque wrench and heated to a maximum temperature of $1.5T_g$ at a rate of $2^\circ\text{C}/\text{min}$. The testing oscillation amplitude was set at $20\text{ }\mu\text{m}$ with a frequency of 1 Hz.

3.2 Uniaxial Tensile testing of adhesive and floating roller peel testing of the hybrid joints

The bulk modified epoxy film adhesive tensile test and FRP test on hybrid joints were conducted in a controlled thermal chamber using a Shimadzu universal testing machine (UTM) supplied by Shimadzu Asia Pacific Pte Ltd, Singapore. The tensile test was conducted under displacement control at cross-head speed of 1mm/min whereas the Ti-6Al-4V adherend of hybrid joint was peeled at a displacement rate of 135 mm/min as per ASTM standard D3167. The FRP peel performance of the hybrid adhesive joint was tested at six different temperatures: $0.21T_g$, $0.75T_g$, T_g , $1.08T_g$, $1.25T_g$ and $1.33T_g$, where T_g is the glass transition temperature of the modified epoxy film adhesive measured from the DMA tests. The FRP behaviour of the joints at $0.21T_g$, $0.75T_g$ and T_g was presented in our previous study on the rebonded joints [21].

4 Test results and discussion

Dynamic modulus and T_g of modified epoxy film adhesive were identified from the DMA test results. The tensile properties of bulk modified epoxy film adhesive and the floating roller peel behaviour of hybrid joints for different temperatures are reported with the corresponding failure modes.

4.1 Dynamic modulus and glass transition temperature of the modified epoxy film adhesive

Figure 2 illustrates the temperature-sweeping dynamic mechanical behaviour of the modified epoxy film adhesive where three different regions were identified: (a) Glassy region where a slight decrease in the storage modulus was observed until $0.75T_g$. As the temperature was increased from $0.21T_g$ to $0.75T_g$, the free volume space would increase at the molecular level and a few chain atoms of the plasticizers or different polymeric composition in the modified

epoxy film adhesive were expected to move around, (b) Glass transition region near T_g where a significant reduction in storage modulus was observed in the temperature range between $0.75T_g$ and T_g . The ratio of the viscous portion to the solid portion can be measured by the $\tan\delta$ of the DMA curve, that reached the maximum peak value of 0.95 near the T_g . Further increase in free space would lead to free movement of the chain of atoms and a co-ordinated molecular motion of the epoxy adhesive begins. The crank-shaft model explains different motions such as stretching, bending, rotation, co-ordinated movements and slippage, (c) Rubbery plateau region after T_g , where the storage modulus response curve is flattened. Here the length of plateau depends on the molecular weight of the polymer [30]. All the testing temperatures were normalised to the T_g of the modified epoxy film adhesive that was measured as 120°C using DMA.

4.2 Tensile behaviour of the bulk modified epoxy film adhesive

The engineering stress versus strain response of bulk modified epoxy film adhesive at $0.21T_g$ and $0.75T_g$ are as plotted in Figure 3. After reaching the maximum tensile stress of 41.4 MPa at $0.21T_g$, the adhesive exhibited higher yielding due to its ductility and failed at a tensile strain of 0.11 mm/mm. At $0.75T_g$, the adhesive was subjected to a maximum tensile stress of 13.2 MPa and showed highly non-linear behaviour because of an increase in the mobility of polymeric chains. No failure was observed until a tensile strain of 0.23 mm/mm is reached. The test was stopped at this stage due to the limitations of thermal chamber height. On molecular basis, the chain of atoms attained a sufficient thermal energy at higher temperature to scale up the co-ordinated motion in the increased free volume. Further reduction in Young's modulus (3.84 times) and increase in failure strain (> 2 times) reveal that the adhesive becomes more flexible and ductile at $0.75T_g$. The measured tensile test data is summarized in table 1.

4.3 Temperature dependent floating roller peel strength of hybrid joints

Figure 4 shows the FRP force and displacement response of hybrid joints at three different temperatures below T_g : $0.21T_g$, $0.75T_g$, and T_g , where the peel force attained a peak value of 359 N/25 mm, 317 N/25 mm and 253 N/25 mm, respectively. Further loading of joints at $0.21T_g$ and $0.75T_g$ showed a short plateau region before reaching another peak load due to a transition in failure mode at about 20 ± 2 mm, then a gradual failure of the joints was observed. The average FRP force of hybrid joints was increased by 141.6% and 330.1% with an increase in the temperature from $0.21T_g$ to $0.75T_g$ and T_g correspondingly [21].

The FRP load versus displacement response is plotted for joints tested above the adhesive glass temperature at $1.08T_g$, $1.25T_g$, and $1.33T_g$ in Figure 5. The peel force was increased linearly up to a displacement of about 1 mm and reached a peak value of 78.36 N/25 mm, 73 N/25 mm and 60.65 N/25 mm at $1.08T_g$, $1.25T_g$, and $1.33T_g$, respectively. The average FRP force of the joints was decreased by 85.5%, 94% and 94.4% respectively as the temperature was increased from T_g to $1.08T_g$, $1.25T_g$, and $1.33T_g$.

4.4 Thermo-mechanical debonding map for composite repairs

The debonding map was obtained using the thermo-mechanical peel performance of the material systems used in the hybrid joint assembly. Figure 6 shows the four regions (A, B, C, and D) of debonding map based on average peel force and temperature.

Region A: Adhesive joints tested below T_g , predominantly shows an interlaminar failure of the composite adherends (Figure 7). An increasing trend in average peel force with temperature is due to the transition of failure mode from inter-laminar failure of composite (ILFC) to cohesive failure (CF). The flexible adherend thickness, composite fiber architecture and peel angle

greatly affect the joint performance and integrity of the composite adherends. Severe damage to composite adherends and higher debonding force makes Region A unsuitable for debonding.

Region B: When tested close to T_g , the bonded joints exhibited cohesive failure (Figure 8) and yielded the highest peel force of 183.5 N per 25 mm width. After this glass transition region, the average peel force starts to decrease drastically and the failure mode remains the same as cohesive. The service temperature of the GFRP adherend is same as the T_g of the modified epoxy film adhesive. This narrow region (from T_g to $1.17T_g$) could not be used for practical debonding due to the uncertainties involved in the heating process and transient heat transfer of varying thickness of flexible adherends. Although no damage to the composite adherend is guaranteed, it is difficult to debond manually. In practical cases, the bonded components may have a bond length of a few hundred millimeters that require higher work on metallic adherend deformation and debonding. As the human muscle force is limited, region B is only suitable for robotic-assisted automatic metal-work removal process in which debonding force and joint temperature can be maintained at desired levels.

Region C: Above $1.17T_g$, the joint loses most of its peel strength. For example, the average FRP force at $1.25T_g$ is 94% lower than T_g and 100% cohesive failure was observed along the bond length. Region C, that ranges from $1.17T_g$ to $1.33T_g$ is the most suitable region for manual debonding and salvaging the composites (Figure 9).

Region D: Above $1.33T_g$, the adhesive has much lower FRP strength due to viscous flow, but there is a possibility of thermal damage to the underlying composite adherends. It is to be noted that the glass transition temperature of the GFRP is 1.62 times the T_g of the modified epoxy film adhesive. Heating the hybrid joints above or $1.33T_g$ might lead to thermal damage of epoxy matrix and affect the mechanical properties of the composites, which will be unfit to be re-

introduced in service. Hence, region D is also unacceptable for the thermo-mechanical debonding process.

Figure 10 shows the crack path selection of bonded hybrid joints tested at different temperatures. As the temperature increased from $0.21T_g$ to $0.75T_g$, the path of crack kinking into the composite adherend was changed to adhesive- metal adherend interface. The combined loading of mode I and mode II along with the influence of T-stresses at the crack tip could explain the observed crack path selection. As the peeling angle was not strictly constrained, the hybrid joints at $0.21T_g$ undergone varying combined loading along its bond length, revealed by multiple failure modes and local residual curvature of the metallic adherend [21]. The mode mixity can be avoided by properly selecting the metallic adherend thickness and roller diameter [31]. Further increasing the temperature to $1.33T_g$, the crack propagated in the adhesive bond line due to reduced strength and stiffness of the epoxy adhesive.

de Freitas and Sinke [19] conducted FRP tests using aluminium-aluminium (Al-Al) bonded joints and CFRP-CFRP joints at room temperature and 80°C . As the temperature was increased from room temperature to 80°C , the following observations were made: there was no change in the average FRP force and failure mode in Al-Al, whereas it increased by 3.78 times in case of CFRP-CFRP joints and the failure mode changed from composite adherend failure to cohesive failure. A similar trend in average FRP force and failure mode shift were observed in the current study.

The initial FRP peak load from the plateau (in between 2 mm to 8 mm) of the hybrid joints measured as a function of temperature was normalised with the maximum value i.e., from $0.21T_g$. Similarly, the storage modulus values were normalised from the value measured at $0.21T_g$. Variation of normalised adhesive joint strength and normalised storage modulus were plotted as a function of normalised temperature in Figure 11. It is interesting to observe that

the trend lines for both follow a similar pattern. The decrease in modulus and peel strength is gradual until $0.75T_g$, moderately high between $0.75T_g$ and T_g , very steep or rapid reduction from T_g to $1.08T_g$ the reduction and almost constant beyond that till $1.33T_g$. Hence, as a rule of thumb, the DMA response obtained for storage modulus can guide the appropriate selection of adhesive debonding temperature, though it shall not describe the failure pattern and mechanism of the hybrid joints.

5 Conclusion

In conclusion, a debonding map as a function of temperature and average floating roller peel force was developed and classified into four regions based on the measured force, failure modes, practical constraints and potential damages to the GFRP adherend. The optimum debonding temperature of hybrid adhesive joints was identified between T_g to $1.17T_g$ for the machine-assisted debonding process whereas between $1.17T_g$ to $1.33T_g$ for the manual-thermo-mechanical debonding process. The predominant failure mode of hybrid adhesive joints changes from composite adherend failure into combined failure of adhesive/cohesive, as the temperature was increased to T_g and above. The variation in the initial peak peel load and storage modulus shows a similar trendline and the DMA test inferences can be used as a thumb rule for selecting the appropriate debonding temperature for thermal assisted debonding of hybrid adhesive joints.

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Table 1. Tensile properties of the bulk modified epoxy film adhesive.

Tensile properties	Temperature (°C)	
	0.21T _g	0.75T _g
Tensile strength (MPa)	41.4	13.18
Young's modulus (GPa)	1.42	0.37
Yield stress (MPa) @ 0.2% offset	36.27	7.84
Failure strain (%)	11	>23
Failure stress (MPa)	39.7	-

Figure 1. Geometric details of the floating roller peel test specimen (not to scale). The nominal adhesive thickness is 0.2 mm.

Figure 2. DMA response of the bulk modified epoxy film adhesive.

Figure 3. Engineering tensile stress versus strain response of the bulk modified epoxy film adhesive at $0.21T_g$ and $0.75T_g$.

Figure 4. FRP behaviour of hybrid joints at $0.21T_g$, $0.75T_g$ and T_g [21].

Figure 5. FRP behaviour of hybrid joints at $1.08T_g$, $1.25T_g$ and $1.33T_g$.

Figure 6. Debonding map of GFRP-Ti adhesive joints in terms of average FRP force and normalised temperature. The T_g of the modified epoxy film adhesive is 120°C.

Figure 7. ILFC of the hybrid joint tested at $0.21T_g$.

Figure 8. Hybrid joint tested at T_g showing cohesive failure.

Figure 9. Cohesive failure of the joints tested at $1.33T_g$.

Figure 10. Crack path selection as a function of temperature: (a) before loading, (b) at $0.21T_g$, (c) at $0.75T_g$ and (d) from T_g to $1.33T_g$.

Figure 11. Comparison of the storage modulus of bulk adhesive and initial FRP load of hybrid adhesive joints.