Measurement of the mechanical properties of structural adhesives in tension and shear over a wide range of temperatures

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Abstract—The aim of this study was to select suitable adhesives for use in a mixed adhesive joint and to measure their mechanical properties over a wide range of temperatures. Three adhesives were selected: a bismaleimide adhesive (Redux 326) for high temperatures and two paste-form epoxies (Hysol EA 9359.3 and Supreme 10HT) for low temperatures. The mechanical properties in tension (dogbone specimens) and in shear (thick adherend shear test) are presented from −55 to 200°C for all three adhesives. Dynamic tests were also carried out to confirm the static modulus results. The correlations between the tensile and the shear properties are reasonable in terms of stiffness and strength but are poor in terms of ductility. In a single-lap joint, Redux 326 is stronger at high temperatures as it becomes more ductile. However, it is very stiff and brittle until 100°C. Supreme 10HT has very good performance up to 100°C. The combination of Redux 326 and Supreme 10HT formed a joint that had a good load carrying capacity from −55 to 200°C.

Keywords: Epoxy; high temperature adhesives; dynamic mechanical analysis; lap-shear; mechanical properties of adhesives.

NOMENCLATURE

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>E</td>
<td>Young’s modulus</td>
</tr>
<tr>
<td>f</td>
<td>natural frequency</td>
</tr>
<tr>
<td>G</td>
<td>shear modulus</td>
</tr>
<tr>
<td>I</td>
<td>second moment of area</td>
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</table>

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1. INTRODUCTION

Adhesive joints used in supersonic aircraft need to withstand low temperatures (down to \(-55^\circ\text{C}\)) when travelling subsonically at high altitude and high temperatures (200\(^\circ\text{C}\) or so) when travelling at Mach 2 or above \([1]\). Adhesives suitable for high temperatures are generally very brittle at low temperatures. On the other hand, adhesives for low temperature use are too weak or degrade at high temperatures. Current commercial adhesives, therefore, are by no means ideal from \(-55^\circ\text{C}\) to 200\(^\circ\text{C}\). Therefore, a solution would be a suitably-designed joint with a combination of a low-temperature adhesive and a high-temperature adhesive.
At high temperatures, the low-temperature adhesive may be very ductile and creep, but it must not degrade. If the high-temperature adhesive fails at low temperatures, then it cannot carry any load at high temperatures. There is an uncertainty for each adhesive outside its range of application. Therefore, the mechanical properties need to be measured, from low to high temperatures, for both types of adhesives in order for a suitable joint to be designed.

Below the glass transition temperature \( (T_g) \), as the temperature decreases, both the modulus and strength increase while the ductility decreases. The lap shear strength increases with the adhesive ductility up to the best compromise between ductility, strength and stiffness. At \( T_g \) there is a rapid reduction in both the modulus and strength as the temperature increases, and the adhesive can no longer carry a substantial load. An optimal joint for both low and high temperatures can be obtained as long as the adhesive is loaded in the temperature range for which it has been designed. By reducing the peak stresses and choosing the right adhesive or a mixture of adhesives, it may be possible to overcome the problem. The mixed modulus concept described by Hart-Smith [2] is that at high temperatures, a brittle adhesive (high modulus) in the middle of the joint retains the strength and transfers the entire load, while at low temperatures a ductile adhesive at the ends of the overlap (low modulus) is the load-bearing component, making the high-temperature adhesive relatively lightly stressed.

The optimisation of the geometry is carried out by finite element analysis (FEA) to make sure that the joint performs well at both low and high temperatures, as well as at intermediate temperatures. This analytical evaluation needs the mechanical properties of the proposed adhesives for both low and high temperatures. FEA is a well-established technique [3], but is still being developed and refined [4–10].

2. EXPERIMENTAL

2.1. Adhesives selected

For high-temperature applications (above 200°C), the adhesives used are either bismaleimides or polyimides, and are generally supplied as films. Film adhesives can be supported, i.e., with a carrier, or be unsupported, i.e., without a carrier. In general, carriers can be of nylon, polyester or glass (usually preferable at high temperatures) fibres and their structure can be non-woven, woven or knitted. The carrier enables the film to be handled, cut to shape and laid up easily. The carrier also serves as a bondline thickness limiting device and also controls the adhesive flow during the cure phase. Although polyimides have good high-temperature properties, bismaleimides offer advantages such as the fact that they do not generate volatiles during cure, thus resulting in less porosity.

The low-temperature adhesive must be ductile and have a higher modulus than the high-temperature adhesive, so that the load is transferred mainly through the low-temperature adhesive. If this is not possible, then the joint can be designed to
Table 1.
Adhesives investigated

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Manufacturer</th>
<th>Adhesive type and curing process</th>
<th>$T_g$ (°C) (manufacturer’s data)</th>
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<tbody>
<tr>
<td>Redux 326</td>
<td>Hexcel</td>
<td>Modified bismaleimide (supported), curing at 175°C for 2 h, post-curing at 230°C for 2 h</td>
<td>280</td>
</tr>
<tr>
<td>Hysol EA 9359.3</td>
<td>Loctite Aerospace</td>
<td>Two-part, modified epoxy paste, curing at 82°C for 1 h</td>
<td>82</td>
</tr>
<tr>
<td>Supreme 10HT</td>
<td>Master Bond</td>
<td>One-part, modified epoxy paste, curing at 120°C for 1 h</td>
<td>135–140</td>
</tr>
</tbody>
</table>

stiffen the ends of the joint. At high temperatures, provided the low-temperature adhesive does not degrade, the high-temperature adhesive will carry the load. Thus, the low-temperature adhesive must be ductile, stiff and strong from $-55^\circ$C to at least $100^\circ$C, which is approximately when the high temperature adhesive starts to become ductile. This means that it must have a $T_g$ around $100^\circ$C or even higher if possible. A rubber would give an excellent ductility, but its stiffness and strength would be too low. A toughened epoxy is probably a better solution since it has a stiffness comparable to that of a high-temperature adhesive and has better ductility.

Three commercially available adhesives were selected. One was a modified bismaleimide film adhesive (Redux 326) for high temperatures and the other two were modified paste epoxies for low temperatures (below $100^\circ$C). The adhesive characteristics are presented in Table 1. In order to understand the influence of the carrier, Redux 326 was studied with and without a carrier, but in both cases the adhesive was solid at room temperature; the carrier was woven glass. As the bismaleimide adhesive has a very high $T_g$, its maximum service temperatures are, according to the manufacturer, 230°C for long periods and 270°C for short periods. The epoxies have a $T_g$ close to $100^\circ$C.

2.2. Tests

It was decided to test the adhesives both in tension and in shear in order to determine the adhesive behaviour under different modes of loading. The adhesives were tested at four different temperatures ($-55$, $22$, $100$ and $200^\circ$C) to obtain a strength profile over this temperature range. Four specimens were tested at each temperature.

Tensile specimens (see Fig. 1) were tested according to BS 2782 standard [11]. The width of the parallel portion was 10 mm, the radius was 60 mm and the thickness was 2 mm. This geometry is recommended when working with rigid adhesives. For flexible adhesives, tensile specimens are about two times smaller [11, 12]. The specimens were originally tested using grips but there was too much slippage. Loading by a pin was also considered but the adhesive failed at the hole,
especially for the brittle bismaleimide. To overcome these problems, special clamps were designed, loading the specimen in the radius region as shown in Fig. 1b. The specimens now always failed in the gauge length. The strain was measured using strain gauges bonded to the specimen. A half bridge with a dummy specimen was used for temperature compensation. To minimise the effect of reinforcement by gauge installation, strain gauges especially designed for plastics were used. The strain gauge technique has previously been used by one of the authors [13] with good results.

To measure the shear properties, the Thick Adherend Shear Test (TAST) was used according to ISO 11003-2 [14]. Note that the extensometer measures not only the displacement of the adhesive, but also the displacement of the adherend. Therefore, it was necessary to apply a correction to the measured displacements. According to ISO 11003-2 [14], the correction should be deduced from the measurement of the shear strain on a ‘dummy’ specimen consisting of the adherend material alone. However, Kadioglu et al. [15] showed that the correction could not be deduced accurately from such a specimen. They modelled the TAST specimen as well as the ‘dummy’ specimen. The predicted profile of shear stress along the centreline of the adhesive was almost constant. Conversely, the shear stress distribution in the ‘dummy’ was not uniform. Kadioglu et al. [15] showed that the adherend correction was better derived from a FEA. Öchsner and Gegner [7, 16] also corrected the deformation using a finite element approach. Alternatively, a simple elastic analysis, assuming that the adherends experience pure shear only, also provides a better
correction than a dummy specimen [15]. This method was used here. The bondline thickness was nominally 0.5 mm.

As regards the specimen fabrication, ISO 11003-2 [14] recommends machining (cutting) the specimens from two plates bonded together. Kadioglu et al. [15] proposed that a better solution is to machine the adherends to the correct dimensions before bonding, which enables the reuse of the adherends and avoids risking damaging the adhesive by machining. A specially built jig was used for aligning and holding the specimens. To control the adhesive fillet, steel shims were inserted into the gaps once the adherends had been brought together. The shims were inserted at the ends of the bond and, after the adhesive was cured, the shims were removed by gently tapping them out of the gap, leaving an accurate termination of the adhesive in the overlap with no fillet.

In addition, single-lap joints (SLJs) were fabricated and tested to assess the adhesive performance in a joint. Hard steel adherends were used. The width was 25 mm, the overlap 12.5 mm and the bondline thickness 0.12 mm.

Dynamic tests to assess the tensile and shear moduli were also performed to confirm the static results. Their description is presented at a later stage in this paper.

2.3. Fabrication

The fabrication of adhesive joints and bulk specimens has been treated in detail in a previous paper [17]. The technique described in the French standard NF T 76-142 [18] for producing plate specimens without porosity was used (see Fig. 2a). It consists of curing plates of adhesive in a mould with a silicone rubber frame under a high pressure (2 MPa or 20 atm). The dimensions of the adhesive plate after cure were 200 mm × 200 mm, with a thickness of 2 mm, which corresponds to the

![Diagram]

Figure 2. Fabrication of adhesives in the bulk (plate) form and in a joint.
internal dimensions of the silicone rubber frame. A quantity of adhesive slightly greater than the volume corresponding to the internal part of the silicone rubber frame is first placed in the centre part of the mould. There is a gap, initially, between the adhesive and the silicone rubber frame. At the moment of the application of pressure, this gap enables the adhesive to be displaced, so as to avoid air entrapment. The lid eventually touches the silicone rubber frame and subjects the adhesive to a high pressure because the mould is perfectly sealed. The high pressure causes the remaining voids to be compressed and also gives a good surface finish. The specimens were then cut to shape.

As regards the bismaleimide joints, the first joints we produced contained many voids. To fabricate joints free of voids, the vacuum release technique proposed by Bascom and Cottington [19] was modified and adopted (see Fig. 2b). The joint was placed in an oven under vacuum without the upper adherend (point 1 in Fig. 2b). The temperature was increased to a point at which the adhesive was most fluid, so that when the vacuum was released the voids collapsed to a negligible volume (point 2 in Fig. 2b). The vacuum release technique was applied without the upper adherend to facilitate the evacuation of air. After the vacuum had been released, the adhesive was largely free of voids and at its most fluid state so that it could wet the upper adherend adequately. The upper adherend was then placed on top and the joint allowed to cure.

3. RESULTS AND DISCUSSION

3.1. Stiffness

3.1.1. Static results. The results are presented in Fig. 3. In tension (Fig. 3A), the bismaleimide in the supported film form is stiffer than in the paste form. The difference between the film tensile modulus and the matrix tensile modulus is approximately constant over the temperature range, being about 1.8 GPa. This difference is due to the presence of the glass carrier. The increase in Young’s modulus due to the fibres varies from 35% at $-55^\circ C$ to 118% at $200^\circ C$. At $200^\circ C$ the Young’s modulus of the paste is only 1.43 GPa and the fibres are much more influential. The modulus of a fibrous composite when loaded along the fibre direction is a linear combination of that of the fibres and the matrix [20]:

$$E_{\text{film}} = V_f E_{\text{glass}} + (1 - V_f) E_{\text{paste}},$$

(1)

where $E$ is the Young’s modulus and $V_f$ the fibre volume fraction. However, the carrier is a cross-weave of fibres, so that the actual volume fraction is twice that used in equation (1). Supposing that the modulus of glass is 76 GPa [20], the value given by equation (1) multiplied by two gives a volume fraction of 4.7%. The fibre volume fraction can also be estimated by the density measurement method [21]. The density of the Redux 326 supported film was found to be 1330 kg/m$^3$, the density of the Redux 326 paste 1280 kg/m$^3$ and the density of the glass fibre was taken.
Figure 3. Adhesive moduli; (A) tensile (measured with dogbone specimens); (B) shear (measured with the TAST).

The density of the film was found to be 2560 kg/m$^3$ [20]. This yields a volume fraction of 3.7%, which compares reasonably well with the 4.7% given by equation (1).

In shear (Fig. 3B), the film is also stiffer than the paste but to a lower extent than for the longitudinal direction. The difference was approximately constant over the temperature range, being this time only about 0.15 GPa. The increase in shear modulus due to the fibres varies from 12% at $-55^\circ C$ to 44% at 200$^\circ C$. At 200$^\circ C$, the shear modulus of the paste is only 0.37 GPa and the fibres are much more influential. Results from the literature [22] show that the fibres are very influential in the axial direction because the longitudinal stiffness is a weighted mean between the moduli of the matrix and the fibre but the shear stiffness is very close to that of the
matrix up to a relatively high volume fraction. This is in accordance with the results presented here, when the matrix is relatively stiff at \(-55^\circ C\) \((E_{\text{paste}} = 5.49 \text{ GPa})\), \(22^\circ C\) \((E_{\text{paste}} = 4.44 \text{ GPa})\) and \(100^\circ C\) \((E_{\text{paste}} = 3.56 \text{ GPa})\). But at \(200^\circ C\) \((E_{\text{paste}} = 1.43 \text{ GPa})\), where the adhesive has a low stiffness, the fibres give a particularly high increase even in shear. Hysol EA 9359.3 is stiff up to \(22^\circ C\), but there is a dramatic drop in stiffness at \(100^\circ C\) as it is above its \(T_g\) \((82^\circ C)\). Supreme 10HT has a reasonable stiffness at \(100^\circ C\) \((2.2 \text{ GPa in tension})\) but at \(200^\circ C\) the modulus is very low because the adhesive is in its rubbery state \((T_g\) is \(135^\circ C)\).

The tensile modulus trend given by the static results contains only four points. To confirm and complete these results, dynamic tests were performed under flexure from \(-55\) to \(200^\circ C\). These results are presented in the next section.

### 3.1.2. Dynamic results.

#### 3.1.2.1. Hysol EA 9359.3 and Supreme 10HT.

Dynamic tests were performed on beams vibrating in flexure in the free-free mode [23]. Both Hysol EA 9359.3 and Supreme 10HT have a \(T_g\) below \(200^\circ C\), so they need to be supported when the temperature is above the \(T_g\). A layer of adhesive was deposited onto a steel beam using the technique described in the French standard NF T 76-142 [18]. The resonance frequency was measured as a function of temperature and the Young’s modulus was deduced from this. A Dynamic Mechanical Thermal Analysis (DMTA) type apparatus was also used which gives the damping as a function of temperature to corroborate the changes in the modulus. The results are presented in Fig. 4. The damping was measured at lower temperatures than the dynamic modulus so as to have a clearer understanding of the low temperature transitions.

For the compound beams (steel + Hysol EA 9359.3 and steel + Supreme 10HT), the adhesive Young’s modulus is related to the compound beam resonance frequency using the following equation:

\[
f = \frac{3.558}{l^2} \sqrt{\frac{\Sigma EI}{\rho_a w t_a + \rho_s w t_s}},
\]

where \(f\) is the natural frequency, \(l\) the length, \(I\) the second moment of area, \(\rho\) the density, \(w\) the width, \(t\) the thickness; the subscript \(a\) stands for adhesive and the subscript \(s\) for steel. The flexural rigidity of the beam, \(\Sigma EI\), is given by [24]:

\[
\Sigma EI = \frac{w l_s^2 t_a E_s E_a}{12(t_a E_a + t_s E_s)} \left[ 4 + 6 \frac{t_s}{t_a} + 4 \left( \frac{t_s}{t_a} \right)^2 + \frac{E_a}{E_s} \left( \frac{t_a}{t_s} \right)^3 + \frac{E_s}{E_a} \frac{t_s}{t_a} \right].
\]

Equation (3) means that the Young’s modulus of the steel must be known in order to compute the adhesive stiffness. Therefore, before bonding a layer of adhesive on top of the steel, the steel alone was tested from \(-55\) to \(200^\circ C\) in order to determine its Young’s modulus as a function of temperature. The adhesive Young’s modulus was then calculated by an iterative procedure. Note that the position of the neutral axis is not constant with temperature, especially below the \(T_g\) of the adhesive. However,
Figure 4. Dynamic flexural modulus of composite specimens and damping using a DMTA type apparatus; (a) Hysol 9359.3; (b) Supreme 10HT.

if it is assumed that the neutral axis of the compound is the same as the neutral axis of the steel due to the much lower stiffness of the adhesive, the computation of the adhesive modulus is more straightforward and does not require an iterative process. It can be obtained from the following equation, which is a simplified version of
Table 2.
α- and β-transition temperatures for Hysol EA 9359.3 and Supreme 10HT

<table>
<thead>
<tr>
<th>Material</th>
<th>$T_\alpha$ ($^\circ$C)</th>
<th>Damping</th>
<th>$T_\beta$ ($^\circ$C)</th>
<th>Damping</th>
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<tbody>
<tr>
<td>Hysol EA 9359.3</td>
<td>108</td>
<td>99</td>
<td>−27</td>
<td>−18</td>
</tr>
<tr>
<td>Supreme 10HT</td>
<td>140</td>
<td>135</td>
<td>−34</td>
<td>−32</td>
</tr>
</tbody>
</table>

equation (3):

$$E_a = E_s \frac{\left( \frac{f_c}{f_s} \right)^2 \left( \frac{\rho_a}{\rho_s} t_s t_a^2 + t_s^3 \right) - t_s^3}{4t_a^3 + 6t_a^2 t_s + 3t_s^2 t_a},$$

(4)

where the subscript c stands for compound. The differences in the modulus results from that calculated by the exact equation were found to be negligible (2.6% for Hysol EA 9359.3 and 2% for Supreme 10HT) for the experimental parameters used in this investigation.

In polymers, the transition from the glassy state to the rubbery state is accompanied by a rapid fall in modulus and a peak in the damping. The (principal) transition is usually referred to as the α-transition and is associated with large scale motions of the polymer chains. Other secondary transitions (β and γ) occur at lower temperatures, which are due to local motions of units of the polymer molecules. The α-transition and the low temperature transition given in Fig. 4 by the damping are close to the ones given using the modulus-temperature curves (point of inflexion method) as shown in Table 2. The change in modulus is lower for the β-transition than for the α-transition by at least an order of magnitude, which is in accord with the literature [25]. According to the damping curve for Supreme 10HT, there is only one secondary transition, at $−32^\circ$C. For Hysol EA 9359.3, there seems to be three peaks, one at $−72^\circ$C, one at $−38^\circ$C and one at $−18^\circ$C. To interpret these results, it should be borne in mind that both epoxies contain rubber (manufacturer’s information). In general, the adhesives industry uses carboxyl terminated butadiene-acrylonitrile (CTBN) co-polymers to increase toughness. According to the literature, the rubber may be dissolved in the matrix (as plasticizer), incorporated into the network (as flexibilizer) or occur in the phase separated state [26–28]. If a polymer is a mixture of two separate phases, then two principal glass transitions are observed instead of one. The CTBN α-transition is of the same order as the epoxy β-transition, which makes it difficult to separate the two phenomena, even though the CTBN peak is sharper. For Supreme 10HT, there is only one peak at low temperature, which is probably the β-peak of the epoxy (there is no overlapping peak, which would be the CTBN α-transition). For Hysol EA 9359.3, the peak at $−72^\circ$C would most probably be the γ-peak. The peak at $−38^\circ$C is sharper and less broad than the peak at $−18^\circ$C. Therefore, at $−38^\circ$C, it is probably the α-transition of
Figure 5. SEM micrographs of fracture surfaces of dogbone specimens tested in tension at room temperature. (a) Supreme 10HT \((\times 5000)\) and (b) Hysol 9359.3 \((\times 5000)\).
the rubber and at $-18^\circ\text{C}$ it is the $\beta$-transition of the epoxy. The Hysol EA 9359.3 tensile specimens showed a large amount of whitening when stressed to beyond its elastic limit, which could be due to cavitation caused by the rubber particles. This was not apparent for Supreme 10HT. The rubber in Supreme 10HT would, therefore, seem to be acting as a plasticizer or a flexibilizer. To support the damping results, the fracture surface of a dogbone specimen tested at room temperature was observed in a scanning electronic microscope (SEM) in order to check for the rubber particles. Supreme 10HT (Fig. 5a) shows no evidence of holes, whereas Hysol EA 9359.3 (Fig. 5b) has many holes, corresponding to the rubber particles.

3.1.2.2. Redux 326. The manufacturer’s value for the $\alpha$-transition of Redux 326 is $280^\circ\text{C}$, so a plain beam was used in this case for testing both the film and paste forms of the adhesive.

The Young’s modulus calculation from the frequency is quite straightforward. The frequency is related to the adhesive modulus by the following equation (23):

$$f = \frac{1.027 w}{l^2} \sqrt{\frac{E_a}{\rho_a}}.$$ (5)

The difference in moduli between the film and the paste is approximately constant over the temperature range ($\approx 1.7$ GPa) as shown in Fig. 6a. This is due to the stiffening caused by the presence of the glass carrier seen also in the static results (1.8 GPa). However, the difference starts to decrease at $150^\circ\text{C}$, due a more rapid decrease in the modulus of the film adhesive, and is only 1.4 GPa at $200^\circ\text{C}$. This suggests that the glass transition temperature of the film is lower than that of the paste. The $T_g$ values of Redux 326 paste and Redux 326 supported film were measured with a DMTA type apparatus to confirm that the film had a lower $T_g$. After cure ($175^\circ\text{C}, 2\ h$), the film $T_g$ is $200^\circ\text{C}$ and the paste $T_g$ is $210^\circ\text{C}$. And after a post-cure at $230^\circ\text{C}$ for $2\ h$, the film $T_g$ is $260^\circ\text{C}$ and the paste $T_g$ is $285^\circ\text{C}$. A literature review shows that the presence of fibres in composites generally increases the $T_g$ although there are cases where a decrease has also been seen [29]. The arguments for an increase are that the fibres decrease the effective free volume, cause a restriction of the molecular motion, and reactions within the interphase cause an increase of the cross-links. The main argument for a decrease is a reaction within the interphase, which causes a decrease of the cross-links or inhibits curing of the resin. Therefore, in the present case, the glass fibres might create an interphase region with a different composition than the bulk matrix thus lowering the $T_g$. However, in the present case, the volume fraction of the fibres is very low (about 4%), so its influence on the matrix damping properties was expected not to be so significant.

An ESEM (Environmental Scanning Electron Microscope) analysis was carried out to check the microstructures of the Redux 326 film and paste, as well as their chemical composition. Figure 7a and 7b, respectively, show the microstructures of Redux 326 film and Redux 326 paste on polished bulk specimens. First, it is interesting to note that, in both cases, there seems to be two phases. One is
Figure 6. Redux 326 flexural dynamic modulus on plain beam specimens; (a) film and paste; (b) film (static moduli included); (c) paste (static moduli included).
Figure 7. Microstructures of polished specimens of (a) Redux 326 supported film (×175) and (b) Redux 326 paste (×1000) (ESEM).
Table 3.
Chemical analysis (ESEM) of Redux 326 supported film and Redux 326 paste

<table>
<thead>
<tr>
<th>Material</th>
<th>Ratios of displayed energy within a spectrum with respect to C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
</tr>
<tr>
<td>Redux 326 film away from fibres</td>
<td>1</td>
</tr>
<tr>
<td>Redux 326 film next to fibres</td>
<td>1</td>
</tr>
<tr>
<td>Redux 326 paste position 1</td>
<td>1</td>
</tr>
<tr>
<td>Redux 326 paste position 2</td>
<td>1</td>
</tr>
<tr>
<td>Redux 326 paste position 3</td>
<td>1</td>
</tr>
</tbody>
</table>

Also see Fig. 7.

homogeneous and the other one heterogeneous with a honeycomb type of structure. This confirms previous suspicion [17] about a secondary phase in the resin since, in the fabrication phase, one part of the adhesive seems to gel at a lower temperature than the rest of the adhesive. A chemical analysis was done on both phases (points 1, 2 and 3 in Fig. 7b) as shown in Table 3. Points 1 and 2 are on the secondary phase and they show a higher sulphur content than point 3 which is on the main homogeneous phase. The manufacturer indicated that it was likely that the secondary phase was sulphur and oxygen rich which could explain the significant differences seen in the sulphur analysis. A chemical analysis was also done on the film away from and close to the fibres (see Table 3). There is Ca and significantly more Si next to the fibres than away from the fibres. The glass fibres, therefore, probably interfere with the composition of the resin and create an interphase with a composition different from the bulk matrix. According to the manufacturer, both Redux 326 film and paste contain spun rock wool at a loading of less than 5%. There is no guarantee that this will be uniformly distributed on the microscopic scale being used in the ESEM analysis. This material will contain an abundance of Si amongst other trace elements. The glass fibres and the rock wool will be the only sources of Si and Ca.

3.1.3. Comparison between the static and dynamic results. The static moduli measured at −55, 22, 100 and 200°C (Fig. 3) show a trend similar to the curve given by the dynamic modulus (Fig. 4). The dynamic properties of a polymer depend on both frequency and temperature. The temperature at which a transition occurs is related to the frequency at which it is measured. As the frequency is increased, so does the transition temperature [26]. The Arrhenius relationship is commonly applied to relate the frequency and the temperature [25]:

\[ f = f_0 \exp\left(-\frac{\Delta H}{RT}\right), \]

where \( f_0 \) is a characteristic of the material, \( \Delta H \) is the activation energy and \( R \) the universal gas constant. The static test was conducted at 1 mm/min. It took approximately 25 s for the specimen to be loaded to its linear elastic limit.
Table 4. Static $\alpha$- and $\beta$-transition temperatures prediction of Hysol EA 9359.3 and Supreme 10HT from the dynamic $E$ vs. $T$ curve measured on steel/adhesive composite beams, using the Arrhenius relationship.

<table>
<thead>
<tr>
<th>Transition</th>
<th>Dynamic</th>
<th>Hysol EA 9359.3</th>
<th>Supreme 10HT</th>
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<tbody>
<tr>
<td>$\alpha$-transition</td>
<td>$f_1$ (Hz)</td>
<td>369.4</td>
<td>360.5</td>
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<tr>
<td>$T_1$ ($^\circ$C)</td>
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<td>Static</td>
<td>$f_2$ (Hz)</td>
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<tr>
<td>$T_2$ ($^\circ$C)</td>
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<td>$\Delta T$ ($T_1 - T_2$)</td>
<td>28</td>
<td>33</td>
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<td>$\beta$-transition</td>
<td>$f_1$ (Hz)</td>
<td>384.8</td>
<td>383.3</td>
</tr>
<tr>
<td>$T_1$ ($^\circ$C)</td>
<td>$-27$</td>
<td>$-34$</td>
<td></td>
</tr>
<tr>
<td>Static</td>
<td>$f_2$ (Hz)</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>$T_2$ ($^\circ$C)</td>
<td>$-78$</td>
<td>$-82$</td>
<td></td>
</tr>
<tr>
<td>$\Delta T$ ($T_1 - T_2$)</td>
<td>51</td>
<td>48</td>
<td></td>
</tr>
</tbody>
</table>

Supposing that this is a quarter of a sinusoidal wave, then the tensile test is equivalent to a dynamic test with a frequency of 0.01 Hz. Using equation (6), the temperature corresponding to 0.01 Hz can be calculated so that the dynamic curve can be shifted and compared with the static results. It should be noted that the Arrhenius relationship is not strictly valid for all transitions. An Arrhenius plot ($\ln f$ vs. $1/T$) shows that the $\beta$-transition obeys the Arrhenius relationship whereas for the $\alpha$ (glass) transition, there is a curvature because the activation energy is not constant with temperature [25]. In the present case, the curve was approximated to a line and the activation energy was supposed to be constant with temperature. For the $\alpha$-transition, $\Delta H$ is of the order of 100 kcal/mol [30] and for the $\beta$-transition it is of the order of 20 kcal/mol [31]. The temperature shifts for both transitions were calculated using equation (6) (see Table 4) and the corresponding values were used to shift the dynamic curves as shown in Fig. 8 for Hysol EA 9359.3 and Supreme 10HT. Although this treatment is only an approximation, the shifted curves fit reasonably well the static results.

For Redux 326 film and paste (see Fig. 6), the dynamic values of the modulus are closer to the static values than for the case of Hysol EA 9359.3 and Supreme 10HT. This is because Redux 326 is a brittle adhesive, and its mechanical properties are less dependent on the strain rate.

3.2. Strength

Typical stress-strain curves in tension and shear for all the adhesives at room temperature ($22^\circ$C) are shown in Fig. 9. To show the variation of strength as a function of temperature, bar charts are presented in Fig. 10. In tension (Fig. 10A), Redux 326 film is stronger than the paste. The difference between the film and the paste is approximately constant at 22, 100 and 200°C and is about 26 MPa. This is due to the glass fibre carrier. The increase in strength due to the fibres varies from
56% at 22°C to 123% at 200°C. At 200°C the strength of the paste is only 21.2 MPa and the fibres are much more influential. At −55°C, the film is still stronger than the resin but to a lesser extent (only 9.6 MPa). At this temperature, the paste is very brittle and may fail prematurely.
For the shear strength (see Fig. 10B), the film is also stronger than the paste but to a lower extent than for the case of the tensile strength, the difference being approximately constant over the temperature range, being this time only about 6 MPa. The increase in shear strength due to the fibres varies from 14% at $-55^\circ$C to 23% at $200^\circ$C. At $200^\circ$C, the shear strength of the paste is only 19.7 MPa and
the fibres are much more influential. Similar comments can be made for the film behaviour in terms of stiffness as for the strength. The glass fibre carrier is very influential in the longitudinal direction but has very little effect in shear. The axial strength follows the rule of mixtures, whereas the shear strength is quite close to that of the matrix, unless the fibre volume fraction is very high [20]. However, when the $T_g$ is approached, the adhesive modulus and strength reduce and the fibres become more influential, even in shear.

The strength trend for Hysol EA 9359.3 and Supreme 10HT (see Fig. 10) is the same as for the stiffness. At 100°C, Hysol EA 9359.3 has very little strength,

![Tensile strength](image1)

**Figure 10.** Adhesive strengths; (A) tensile strength (measured with dogbone specimens); (B) shear strength (measured with the TAST).
whereas Supreme 10HT is still relatively strong. At 200°C, both adhesives are very weak. These results are in accord with the $T_g$ values. Below the $T_g$, the adhesives are stiff and strong; above the $T_g$, they are very soft and cannot carry a substantial load. The $T_g$ of Supreme 10HT is higher than that of Hysol EA 9359.3, and it will, therefore, be able to carry the load to a higher temperature than when used in combination with Redux 326.

### 3.3. Ductility

The results of the ductility tests are presented in Fig. 11. Both Redux 326 film and paste are brittle up to 100°C, but their ductility is higher at 200°C (as the $T_g$ is

![Figure 11.](image-url) Adhesive ductility; (A) tensile (measured with dogbone specimens); (B) shear (measured with the TAST).
approached). This is especially evident in shear (Fig. 11B). If a ductile adhesive is to be used in combination with Redux 326, it must, therefore, be able to carry the load up to 100°C. While Hysol EA 9359.3 is stiff, strong and ductile up to 22°C, at 100°C, it is near its \( T_g \) and is, therefore, not suitable to be used with Redux 326 in a bi-material joint. Supreme 10HT is stiff, strong and ductile up to 100°C. Therefore, Supreme 10HT is suitable for use with Redux 326. Note that the maximum shear strain for both Supreme 10HT and Hysol EA 9359.3 increases with temperature up to 100°C, but drops at 200°C as if they have become brittle. We must, therefore, be careful, as the tensile strain to failure might be a problem even though it has 25% shear strain to failure. This result was surprising, but is in accord with the literature [30] where it is said that at high temperatures (above the \( T_g \)), the polymers are extremely soft, but the strain to failure may again decrease and give an apparently brittle behaviour.

3.4. Single-lap joint tests

The test was described in Section 2.2 and the results are shown in Fig. 12. For Redux 326 single-lap joints, there is not much difference in strength between −55 and +100°C; the adhesive remains brittle until at least 100°C. At 200°C, the joint has a higher lap shear strength. From −55 to 100°C, the failure takes place at the end of the overlap and, once a crack has initiated, the adhesive has no ductility to absorb the fracture energy. As the temperature increases and gets closer to the adhesive \( T_g \) (280°C), the adhesive becomes more ductile and the overlap contributes more to the strength as the adhesive yields. The yielding of the adhesive redistributes the stresses, making more use of the less strained parts of the overlap. There is no noticeable difference in the average lap shear strength between the film with the carrier and the neat paste, although the carrier tends to act as a toughening agent. Some additional tests were carried out at room temperature for Redux 326 paste

![Figure 12. Lap shear strengths of the adhesives.](image-url)
without post-cure. The lap shear strength obtained was 14.9 MPa, which is 81% of the lap shear strength of Redux 326 paste with post-cure (18.4 MPa). This result was rather surprising as one would have expected the adhesive without post-cure to be more ductile and, consequently, to give a higher lap shear strength. However, this result shows that the post-cure is a very important stage and must not be omitted.

Hysol EA 9359.3 has the best average failure stress at $-55$ and $+22^\circ$C. At these temperatures it has the best combination of stiffness, strength and ductility. However, its $T_g$ is too low to be used in conjunction with the high-temperature adhesive as it is unable to cover the intermediate temperature range where Redux 326 is brittle.

Supreme 10HT has very good performance up to $100^\circ$C. In a mixed adhesive joint with Redux 326, it can be the load-bearing component until at least $100^\circ$C. At $200^\circ$C, it is above its $T_g$, but Redux 326 is now more ductile and can carry the load safely.

3.5. Tension vs. shear

3.5.1. Stiffness. For isotropic bulk materials, the shear modulus is related to the tensile modulus by the following equation:

$$G = \frac{E}{2(1 + \nu)},$$

(7)

where $G$ is the shear modulus. From tests on bulk specimens in tension, the shear modulus can be derived and vice versa. The value of Poisson’s ratio typically varies between 0.3 and 0.5; it was measured on bulk tensile specimens with two strain gauges, one in the direction of loading and the other rotated through 90°. At $22^\circ$C, $\nu$ was 0.35 for the Redux 326 paste, 0.38 for Hysol EA 9359.3 and 0.36 for Supreme 10HT.

Applying equation (7), it was found that the shear modulus measured with the TAST was lower than the predicted shear modulus from the bulk tensile test (see Fig. 13). This is especially the case for Redux 326 paste and Hysol EA 9359.3.

It should be noted that equation (7) might not be adequate for the film adhesive because it is not an isotropic material (due to the presence of the glass fibres). Note also that, at $100^\circ$C, Hysol EA 9359.3 enters its rubbery state and, at $200^\circ$C, Hysol EA 9359.3 and Supreme 10HT are in the rubbery state. The Poisson’s ratio will certainly be different from the one measured at room temperature and will approach or equal 0.5. The shear modulus was predicted at $100^\circ$C and $200^\circ$C for Hysol EA 9359.3 and at $200^\circ$C for Supreme 10HT using a Poisson’s ratio of 0.5.

To confirm the moduli obtained with the TAST and bulk specimens statically, bulk specimens were tested dynamically in both flexure and shear at room temperature. The Young’s modulus ($E$) was obtained by vibrating a beam in bending at its first natural frequency. An electrodynamic shaker was used to excite the beam and a laser to measure the resonance amplitude. Dogbone beams were also tested dynamically to check if the dynamic modulus could be measured on such beams so that the
same specimen could be tested both dynamically and statically. Note that there is no closed-form solution for the resonance frequencies of vibration of the dogbone specimen. However, if the dogbone specimen is made of a known material (e.g., aluminium), then $E$ can be computed, since the geometric factors are the same for all materials. Thus,

\[ f = k \sqrt{\frac{E}{\rho}}, \]

\[ \frac{f_a^2}{f_{al}^2} = \frac{k^2 \frac{E_a}{\rho_a}}{k^2 \frac{E_{al}}{\rho_{al}}}, \]

or

\[ E_a = \frac{f_a^2 \rho_a E_{al}}{f_{al}^2 \rho_{al}}, \quad (8) \]

where $k$ is a geometric factor and the subscript al stands for aluminium. An aluminium dogbone specimen was fabricated with exactly the same dimensions as the adhesive dogbone. The Young’s modulus of the aluminium was first determined with a plain beam and it was found to be 71.033 GPa. The density of this aluminium was 2689 kg/m$^3$. The first flexural natural frequency for the aluminium dogbone beam was 568 Hz. Then, according to equation (8), it is only necessary to measure the natural frequency of the adhesive dogbone and its density to compute its Young’s modulus. It was found that the dogbone specimens gave a modulus quite close to the one given by the beam with a uniform shape as shown in Fig. 14a. This means that, before testing a dogbone specimen destructively, the dynamic modulus can first be measured on that same specimen.
The dynamic shear modulus of the bulk specimens was obtained using the torsion pendulum developed by Adams and Singh [31]. TAST specimens were also tested dynamically in order to find their dynamic shear modulus, according to the procedure proposed by Maheri and Adams [32]. They measured the resonance of free-free TAST specimens in the axial and width-wise lateral modes. Dynamic models based on a closed-form analysis were found to be inadequate to measure
Figure 15. Dynamic $G$ measured on TAST specimens according to the Maheri and Adams method [32] vs. static $G$ measured on TAST specimens.

$G$ accurately. Therefore, empirical formulae, validated with static tests, were given for predicting the shear modulus. For the axial mode:

$$G/t = 4.8222 \times 10^{-11} f^3 - 7.3454 \times 10^{-7} f^2 + 3.0045 f.$$  \hspace{1cm} (9)

For the width-wise mode:

$$G/t = 2.7166 \times 10^{-10} f^3 - 1.7967 \times 10^{-6} f^2 + 3.4513 f,$$  \hspace{1cm} (10)

where $t$ is the bondline thickness. The dynamic shear modulus obtained from the torsion pendulum test and from vibrating the TAST specimens are presented in Fig. 14b. It confirms that in the TAST form, the shear modulus is lower than when in the bulk. It should be borne in mind that the TAST results should be regarded with caution because the equations used to compute the dynamic shear modulus are empirical and were obtained for a single adhesive. Nevertheless, the shear modulus measured statically is relatively close to that measured dynamically by the Maheri and Adams procedure [32] as shown in Fig. 15. Therefore, the Maheri and Adams dynamic method is a good guide and can give an estimate of the shear modulus using TAST specimens before these specimens are tested destructively.

A good correlation between the dynamic shear and the tensile moduli of bulk specimens was found as shown in Fig. 16. The predicted and the experimental values are very close and give a Poisson’s ratio between 0.3 and 0.5. Dolev and Ishai [33] also found an excellent relationship between the static shear and the tensile moduli of bulk specimens with an epoxy film adhesive. They also measured the shear modulus when the adhesive was in a joint (napkin ring test) and found that the predicted shear modulus from the experimental Young’s modulus in tension
Effect of temperature on mechanical properties of adhesives

Figure 16. Predicted shear modulus from the dynamic flexure modulus of a beam vs. dynamic experimental shear modulus measured in the torsion pendulum, at room temperature.

on bulk specimens was higher than the experimental shear modulus on bonded specimens. The apparent Poisson’s ratio (as calculated from equation (7)) is 0.73. They attributed this discrepancy to the fabrication and showed that when the joint was fabricated in a closed mould to prevent adhesive flow under pressure, the relationship between $E$ and $G$ was better (Poisson’s ratio of 0.5). When fabricated under hydrostatic pressure, the adhesive has fewer defects such as voids that would reduce its stiffness. Lilleheden [34] studied the same adhesive as Dolev and Ishai [33] and used Moiré interferometry to measure the adhesive strain in a TAST type joint and related the adhesive properties in situ and in bulk. Both the TAST and the bulk specimens were cured in an autoclave using hydrostatic pressure. He found an excellent correlation between the shear modulus in the bulk form and in the TAST joint. The fabrication technique used by Lilleheden [34] is comparable to that used by Dolev and Ishai [33], since, in a closed mould, the adhesive is subjected to hydrostatic pressure. Jeandrau [35] also related the mechanical properties of adhesives in situ (TAST) and in bulk. The bulk specimens were fabricated using the same technique as used here (sealed mould under high pressure) and the TAST specimens were produced by bonding two pre-cut panels from which TAST specimens were cut and machined afterwards. This method of fabrication does not put the adhesive under hydrostatic pressure, but nevertheless the adhesive is more compressed than in the fabrication method that we used, where individual specimens are bonded. Jeandrau [35] reported results on one epoxy film, three one-part epoxy pastes, and one two-part epoxy paste. He found that the Poisson’s ratio
given by the experimental Young’s modulus in bulk specimens and the experimental shear modulus with the TAST was 0.36 for the epoxy film, 0.6 for the one-part epoxy paste and 0.66 for the two-part epoxy paste. Therefore, depending on the type of adhesive, the relationship between $E$ in a bulk specimen and $G$ in a joint varies, the worst case being for the two-part adhesive. Two-part adhesives require mixing which leads to voids if the adhesive is not under hydrostatic pressure, as was the case in the TAST specimen fabrication. In the present work, it was found that the relationship between $E$ in a bulk specimen and $G$ in a joint was not so good for the two-part adhesive Hysol EA 9359.3 (Poisson’s ratio of 0.74 at room temperature) nor for the bismaleimide adhesive Redux 326 (Poisson’s ratio of 0.88 at room temperature). The bismaleimide adhesive is a one-part adhesive but its manufacture is not as easy as for an epoxy adhesive and leads to more defects. The relationship is better for the one-part adhesive Supreme 10HT (Poisson’s ratio of 0.4 at room temperature) as it does not require mixing and this leads to fewer voids in the joint. Another one-part adhesive, AV 119 from Vantico (formerly Ciba, Cambridge, UK), was also tested with the TAST ($G = 1.00$ GPa) and in tension ($E = 3.05$ GPa) with a bulk specimen, giving a Poisson’s ratio of 0.5. Another point to bear in mind is the nature of the test. Although the extensometer described in ISO 11003-2 [14] enables very precise and accurate measurements of adhesive deformations, there are several sources of error, such as the measurement of specimen dimensions, the position of the extensometer and the adherend correction. Vaughn [36] estimated that the effect of the potential sources of error on the measurement of the shear modulus could be as much as $\pm 12.7\%$. The calculated Poisson’s ratio using equation (7) is, in fact, affected by test and fabrication limitations and, thus, is not a real Poisson’s ratio. It would be better if the TAST specimens were produced with the adhesive under hydrostatic pressure, to guarantee that the adhesive was in the same state both in the bulk form and in the TAST joint. One possibility would be to use a fabrication method based on the French standard NF T 76-142 [18] previously mentioned, where two plates could be bonded together with a silicone rubber frame and then specimens could be cut according to the TAST standard dimensions.

3.5.2. Strength and ductility. When the effect of the volumetric component of the stress is negligible, the shear properties (strength and ductility) can be predicted by the following two equations [35], which are derived from the von Mises criterion,

$$\tau = \frac{\sigma}{\sqrt{3}}, \quad (11)$$

$$\gamma = \frac{2\varepsilon(1 + \nu)}{\sqrt{3}}, \quad (12)$$

where $\tau$ is the shear stress, $\sigma$ the tensile stress and $\gamma$ the shear strain. However, unlike metals, polymer behaviour depends on both the hydrostatic and deviatoric stress components. The modelling of adhesives is a complex problem that is still under development [12, 37]. There are three main pressure-dependent yield criteria
Figure 17. Ratio of shear strength measured by the TAST method and the shear strength predicted from the tension test at different temperatures; (a) using the von Mises criterion; (b) using the Dolev and Ishai criterion [33].

described in the literature. These are the Drucker–Prager [38], the Raghava [39] and the Dolev and Ishai [33] criteria. Using the Dolev and Ishai [33] method, the strength and ductility in shear can be predicted according to equations (13) and (14):

\[
\tau = \frac{\sigma}{\sqrt{3}} \frac{2\lambda}{(\lambda + 1)},
\]

(13)

\[
\gamma = \frac{2\varepsilon(1 + \nu)}{\sqrt{3}} \frac{2\lambda}{(\lambda + 1)},
\]

(14)

where \(\lambda\) is the ratio of the yield stress in compression to the yield stress in tension. \(\lambda\) varies typically between 1.2 and 1.4 [3]. Taking a value of \(\lambda\) of 1.3, the shear stress and strain given by equations (13) and (14) are higher than those given by
Figure 18. Ratio of shear failure strain measured by the TAST method and the shear failure strain predicted from the tension test at different temperatures; (a) using the von Mises criterion; (b) using the Dolev and Ishai criterion [33].

Equations (11) and (12) for the von Mises criterion, and equations (13) and (14), for the Dolev and Ishai criterion with a value of $\lambda$ of 1.3, were used to predict the strength and ductility in shear from the results in tension. The ratio of the experimental value to the predicted value is presented in Fig. 17 for the strength and in Fig. 18 for the strain to failure. Equations (11), (12), (13) and (14) may not be applicable when the adhesive is in the rubbery state, since the adhesive is hyperelastic and does not have a yield stress. Therefore, the ratio was not calculated at 100°C and 200°C for Hysol EA 9359.3 and at 200°C for Supreme 10HT. While the predicted shear strength is reasonably close to the experimental value, especially
Effect of temperature on mechanical properties of adhesives

Figure 19. Experimental tensile stress–strain curve vs. predicted tensile stress–strain curve from the experimental TAST curve for Supreme 10HT at 22°C.

when the hydrostatic component is taken into account as in the Dolev and Ishai criterion, the experimental shear strain to failure is much higher (2–9 times) than the predicted value, even for the Dolev and Ishai criterion. Furthermore, the more ductile is the adhesive, the higher is the discrepancy. For Redux 326, the ratio of shear strain to failure measured by the TAST method and the shear strain predicted from the tension test is 2 at −55, 22 and 100°C where the adhesive is very brittle, but increases to 9 (Dolev and Ishai criterion) at 200°C when the adhesive is ductile. The same ratio is 8 for Hysol EA 9359.3 at −55 and 22°C and 5 for Supreme 10HT at −55, 22 and 100°C. Both Hysol EA 9359.3 and Supreme 10HT are relatively ductile at these temperatures (see Fig. 11). This could be explained by the different behaviour of the adhesive when loaded in shear and in tension in the presence of defects such as voids. In tension, once a crack is triggered next to a void, the specimen fails due to the high stress concentration. In shear, even if a crack is triggered, the remaining area is capable of further deformation, especially if the adhesive is ductile. To illustrate this, take, for example, the case of Supreme 10HT at 22°C as shown in Fig. 19. In shear, the adhesive shows ductile behaviour, while in tension it is much more brittle than predicted from equations (11) and (12) or (13) and (14). The predicted curve fits well the initial part of the experimental curve, but gives a much larger strain to failure than is actually measured. This suggests that the adhesive failed prematurely in tension. When loaded in tension, the adhesive is extremely sensitive to any defects and does not give its full potential.
4. CONCLUSIONS

1. Supported film adhesives behave like low volume fraction composites where the fibres are very influential in tension but not so much in shear. Note that, in a joint, the adhesive is primarily loaded in shear or peel, especially for the single-lap joint. Whether in shear or in peel, supported film adhesives will behave much like a paste adhesive. In peel, the adhesive is loaded in the transverse direction; so in this case, the modulus is close to the modulus of the paste adhesive. Therefore, the failure will be mostly governed by the properties of the adhesive. The single-lap joint results prove this, as the differences between paste and film are very small.

2. The correlation between the tensile tests on bulk specimens and the TAST is reasonable in terms of both modulus and strength.

3. The adhesive is much more ductile in shear than in tension; therefore, the complete shear curve cannot be deduced from the tensile curve.

4. In a mixed adhesive joint with Redux 326 and Supreme 10HT, it is expected that, at high temperatures, Redux 326 in the middle of the joint will retain the strength and transfer the entire load and, at low temperatures, Supreme 10HT at the ends of the joint will be the load-bearing adhesive. This proposition needs to be investigated through simulation and testing and will be the subject of future work.

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