

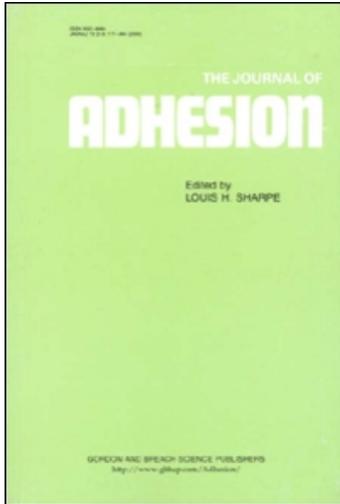
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Comparison of the Shear Stress-strain Behaviour of some Structural Adhesives

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The shear stress-strain behaviour of structural adhesives provides important data for the designer. Shear modulus, strength, and elastic and plastic strain to failure have been determined using a torsional butt joint technique which is relatively quick to perform and is believed to be very accurate. A range of structural adhesives have been compared, which has highlighted some important differences in their behaviour. Increasing the bond line thickness of an adhesive lowers the plastic strain to failure.

INTRODUCTION

Modern structural adhesives are toughened and show considerable plastic as well as elastic behaviour. The shear stress-strain properties, such as shear modulus of elasticity, elastic and plastic shear strain to failure, are as important to the designer as the widely published shear strength data when concerned with high performance structural bonded joints, yet these data are not readily available in the literature. One reason for this is the practical difficulties involved in measuring strains in material as thin as a structural adhesive glue-line, typically only 0.1–0.3 mm in thickness.

A standard method¹ of test has, in fact, existed for many years, based on the 'napkin-ring' specimen originally proposed by de Bruyne.² This consists of two circular coaxial thin walled cylinders bonded end to

end by the adhesive under test, and subjected to a torsional shear force which produces a peripherally uniform stress distribution and is self-aligning. An alternative method which has been used is the thick adherend lap shear specimen,^{3,4} which although perhaps allowing easier measurement of the glue-line thickness, experiences a complexity of stress distribution and loading alignment.

The present work adopted a modification of the first method, but, instead of cylinders, round bar specimens were used for ease of machining and specimen fabrication. The stress field is not uniform across the bar as in the case of the napkin-ring specimen, but is still of pure shear only and can easily be calculated. The results presented are part of a larger programme of work to generate design data on the best structural adhesives currently available having a curing temperature no greater than 120°C, and the work has highlighted important differences in their elastic-plastic properties and also the effect on certain of these properties of varying the bond line thickness.

EXPERIMENTAL PROCEDURE

Materials and Specimen Fabrication

Seven adhesives from five different manufacturers are compared in this report and are designated A to G respectively. The adhesive type and curing details are given in Table I. The room temperature epoxies were given a post-cure at a moderate temperature (50°C) for consistency with the overall programme of work. The bonded specimens were fabricated as round aluminium alloy (HE 30) butt joints with a diameter of 15 mm. In order to characterise the adhesive rather than the metal/adhesive interface it was necessary to obtain "cohesive" failure

TABLE I
Types of Structural Adhesive Investigated

Adhesive	Manufacturer	Adhesive Type
A	1	two-part, cold-curing, modified epoxy paste
B	2	two-part, cold-curing, modified epoxy paste
C	1	one-part, hot-curing (120°C) epoxy paste
D	1	hot-curing, (120°C) modified epoxy film (supported)
E	3	hot-curing, (120°C) modified epoxy film (mat carrier)
F	4	hot-curing, (120°C) modified epoxy film (nitted carrier)
G	5	cold-curing, two-part, toughened acrylic

in the joint, and past experience had shown phosphoric acid anodizing⁵ to be the most suitable surface pretreatment for the aluminium adherends. This resulted in almost entirely cohesive failure in all the joints. The bond line thickness of the paste adhesives was controlled by the addition of 0.5 wt.% ballotini glass spheres (105–210 μm diameter), whilst the acrylic adhesive, F, is intended for use without a bond line spacer. In later tests which investigated the effect of varying the bond line thickness, the latter was increased in the paste adhesives by including a very short (< 1 mm) piece of wire of the desired thickness at the centre of the joint as a spacer. The film adhesives were increased in thickness by the application of several film layers in the bond lines. Axial alignment of the specimens during bonding was achieved by mounting the joints vertically in a purpose designed jig, and the required pressure was applied by placing the correct weight on the end of each specimen. For the hot curing adhesives the specimen jig was placed in an oven at the required temperature (120°C).

The accuracy of the shear strain and modulus determinations is very dependent on the accuracy to which the bond line thickness of each specimen can be measured. Direct measurement at the circumference of the joints was found to be inadequate, and the method finally adopted was to measure the distance between scribed lines on the adherend end pieces before and after bonding using a digital component measuring system with a resolution of 1 micron. Much time and effort was expended in perfecting this technique, to the extent of optimising the scribing tool for perfect alignment of the measuring equipment's cross-hairs. The accuracy of the technique was checked by direct measurement of the bond line thickness on some bonded specimens which had been sectioned across the joint and highly polished; the results were generally within 2% of each other for a bond line of 0.15 mm. Even so, each adhesive was always tested using four replicate specimens, to increase the accuracy of the data.

Torsional Testing Equipment

The joints were shear loaded in a purpose built torsional testing machine manufactured by the Additional Equipment Co. (see Figure 1). The torque was monitored by a 70 Nm-capacity torque cell fed into the Y-axis of an X-Y plotter. A "twistometer" measured the angle of twist and was attached to the specimen across the bond line by three pointed screws. Two LVDT's (Sangamo type NER, ± 2.5 mm stroke) attached

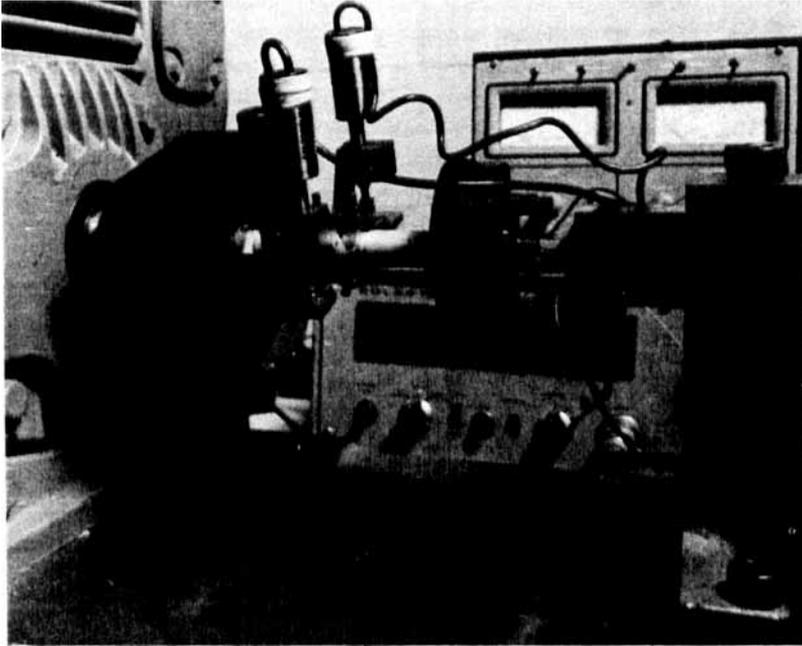


FIGURE 1 Close-up of butt joint being tested with twistometer attached.

to the arms of the twistometer monitored the twist and the signal was fed into the X-axis of the plotter. In this way torque/twist curves were obtained for each specimen. The instrumentation described has a potential resolution of 1×10^{-5} of a degree, but in practice a resolution of 0.002 deg. was found to be satisfactory. This is because the accuracy of the shear modulus and strain values are determined to an equal extent by the uniformity and accuracy of the bond line thickness, and this was generally to within about 10% for a 0.15 mm bond line. An angle of twist resolution of 0.002 deg. was well within this figure and had the further advantage of allowing all of the torque/twist curve up to the point of failure to be included on the same XY plot.

Allowance was made for the adherend twist by performing tests on a dummy alloy specimen with no bond line, and subtracting this from the total twist of each bonded joint. Although the contribution from the adherend to the total twist is relatively high, this is not disadvantageous as long as the equipment is capable of measuring each

very accurately, as was the case here. Preliminary experiments comparing aluminium alloy bonded joints with those of steel bonded joints for the same adhesive showed no difference in the resulting properties of the adhesive layer, even though the steel adherends reduced the adherend twist significantly due to its greater stiffness.

The torque speed of the testing machine could be varied in steps of 0.001 r.p.m. The preliminary experiments included a variation of this torque speed in order to vary the strain rate on the adhesive joints. The ASTM standard¹ specifies a loading rate to produce failure in 2–5 minutes. Varying the speed from 0.001–0.061 r.p.m. gave loading rates both within and on each side of this failure time, but there was no significant effect on the resulting stress-strain curves. The torque speed was therefore maintained in all the subsequent tests at 0.021 r.p.m., which produced failure within the specified time, and resulted in an average strain rate (before yielding) of 0.5 min^{-1} on a bond line of 0.15 mm.

RESULTS and DISCUSSION

Calculation of Shear Properties from the Torque-Twist Curves

The properties which are of interest in the present work are illustrated in Figure 2; they are shear strength (τ_{max}), shear modulus (G), elastic shear strain (γ_e) and plastic shear strain (γ_p). The total strain to failure is therefore given by $\gamma_e + \gamma_p$. The equations used to construct shear stress-strain curves from the experimental data (torque-twist) and to obtain the values of τ_{max} , G , γ_e and γ_p are listed in Table II. Their derivations can be found in classical mechanics texts such as Timoshenko⁶ for the elastic properties and Nadai⁷ for the plastic behaviour.

It will be noted from Table II that the definition of shear strain used is that of the sheared displacement divided by the adhesive thickness and therefore has the units of mm/mm. Some confusion arises when quoting plastic shear strains to failure, where the angle of shear can be quite large. In this case the relationship $\tan \gamma = \gamma$ no longer holds and the angle of shear (in radians) is not then the same as the value of shear strain as defined above. Shear strain values in mm/mm can be converted to shear strain angles by taking the arc-tan of $\gamma(\text{mm/mm})$ and converting to radians.

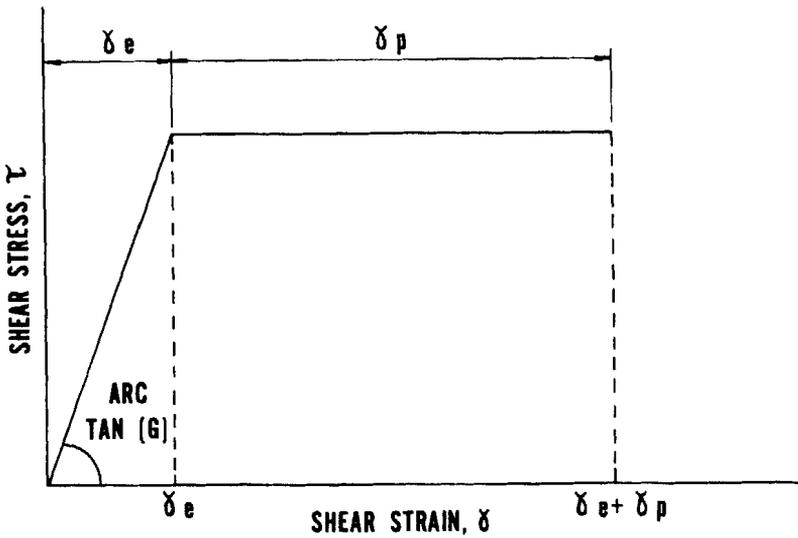


FIGURE 2 Model stress-strain curve.

TABLE II
Equations Used to Calculate Shear Properties from Torque-Twist Curves

	Notation	Units
	θ = relative twist across the bond line	degrees
	T_θ = torque to produce a relative twist θ	N.mm
	t = adhesive bond line thickness	mm
	r = radius of specimen	mm
	G = adhesive shear modulus	MPa
	τ = shear stress on adhesive	MPa
	τ_{max} = adhesive shear strength	MPa
	T_{max} = maximum torque	N.mm
	θ_e = maximum linear twist	degrees
	θ_p = maximum plastic twist (i.e. at T_{max})	degrees
	γ_e = elastic shear strain	mm/mm
	γ_p = plastic shear strain to failure	mm/mm
(1)	$G = \frac{360 T_\theta t}{\pi^2 \theta r^4}$	
(2)	$\tau = \frac{1}{2\pi r^3} \left(\frac{\theta \delta T}{\delta \theta} + 3T \right)$	
	At τ_{max} $\frac{dT}{d\theta} = 0$, then $\tau_{max} = \frac{3 T_{max}}{2\pi r^3}$	
(3)	$\gamma_e = \frac{\pi r \theta_e}{180 t}$	
(4)	$\gamma_p = \frac{\pi r \theta_p}{180 t}$	

Comparison of Shear Properties

The individual shear stress-strain curves for the four replicate specimens of one of the adhesives, B, have been plotted out in Figure 3. The

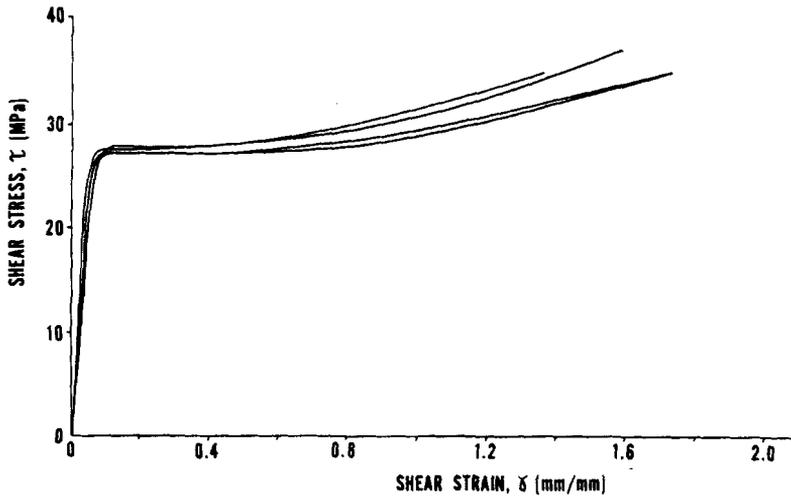


FIGURE 3 Shear stress-strain curves of replicate specimens for adhesive B.

consistency of behaviour and reproducibility of the test technique shown by this adhesive is typical of all the adhesives investigated with the exception of one (adhesive G). For the others, the only parameter to show any significant variation between specimens of the same adhesive was the plastic strain to failure (γ_p). The reason for this variation in γ_p was due to differences in the bond line thicknesses of the replicate specimens, and this will be further discussed in the next section. Adhesive G is the acrylic, and this type of adhesive is known to have somewhat less consistent behaviour compared to epoxies. This has been found to be the case here also, where Figure 4 shows some variation in shear strength, τ_{max} , as well as γ_p , and these variations do not appear to be related to bond line thickness.

For direct comparison the four replicate stress-strain curves for each adhesive have been averaged out and all plotted on the same graph in Figure 5. The corresponding values of G , τ_{max} , γ_e and γ_p have been tabulated for each adhesive in Table III. It is interesting to compare the results where possible with those of Althof,⁴ who included two of

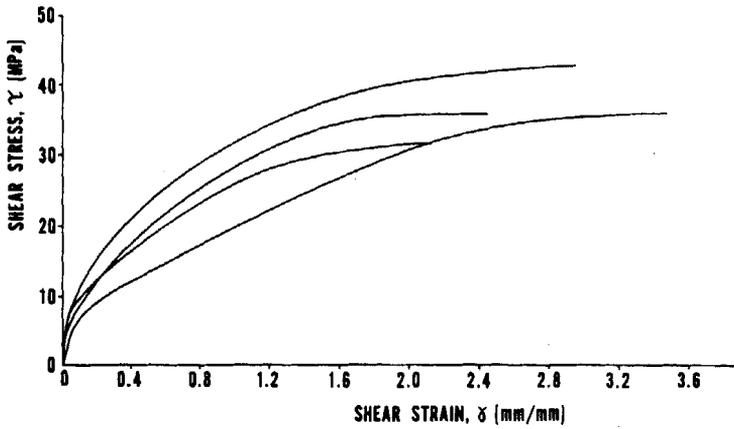


FIGURE 4 Shear stress-strain curves of replicate specimens for adhesive G.

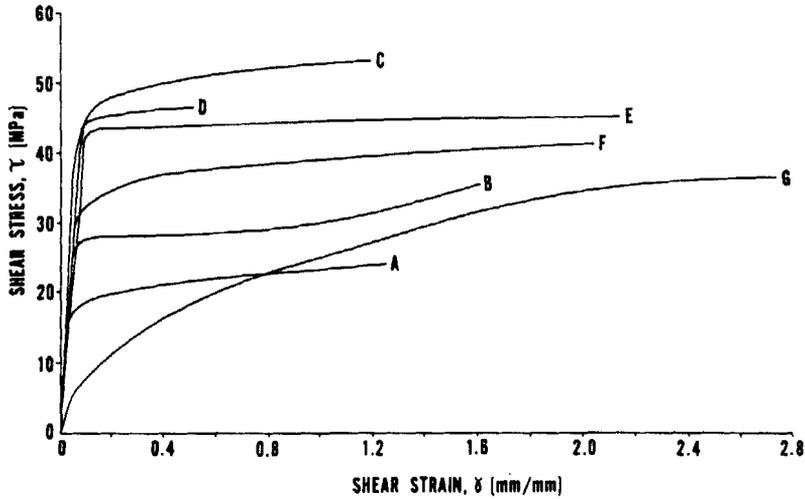


FIGURE 5 Comparison of adhesive shear stress-strain curves.

the same adhesives but obtained his results using the thick adherend lap shear specimen. The values for G and τ_{max} are virtually identical to those determined in the present work, and although the shear strain to failure varies slightly, this variation is in agreement with the differences in bond line thickness used in the respective investigations.

TABLE III
Adhesive Shear Stress-Strain Data
(Results are averages of 4 replicates with standard deviations)

Adhesive	G(MPa)	τ_{\max} (MPa)	γ_e (mm/mm)	γ_p (mm/mm)
A	479 ± 27	24.2 ± 0.9	0.033 ± 0.002	1.215 ± 0.07
B	511 ± 101	35.6 ± 0.6	0.049 ± 0.011	1.550 ± 0.17
C	643 ± 26	53.0 ± 2.2	0.061 ± 0.002	1.100 ± 0.22
D	722 ± 203	46.3 ± 0.4	0.048 ± 0.010	0.462 ± 0.07
E	424 ± 10	45.0 ± 2.9	0.100 ± 0.010	2.035 ± 0.43
F	569 ± 146	41.3 ± 1.1	0.045 ± 0.016	1.98 ± 0.34
G	155 ± 46	36.6 ± 47	0.040 ± 0.010	2.685 ± 0.55

From Table III it can be seen that all the epoxy adhesives, A–F, have similar shear moduli and lie within the range 400–750 MPa. However, there are distinct differences between their values of γ_p , the plastic strain to failure; the epoxy film adhesives E and F exhibited over four times the plastic strain of epoxy film adhesive D. As their shear strengths are approximately the same, these γ_p values represent a significant difference in toughness. Hart-Smith,⁸ in his closed form analysis for bonded joints, defines the area under the stress-strain curve as the adhesive strain energy, A_s , where $A_s = \tau \max \left(\frac{\gamma_e}{2} + \gamma_p \right)$. In the analytical solution the load carrying capacity of the joint is determined as being proportional to the square root of A_s . A fourfold increase in adhesive strain energy would therefore double the strength of the joint.

The two room temperature curing epoxies, A and B, not unexpectedly have lower shear strengths than those cured at 120°C; however, their plastic shear strain to failure is quite large. The stress-strain curve of the two-part acrylic adhesive, G, differed in shape significantly from all the epoxies. A much lower shear modulus was found, but its strain energy was high due to its strain to failure being the largest of all the adhesives tested. However, this would be aided by its thin glue line (no bond line spacer was used).

Effect of Bond Line Thickness on Adhesive Shear Properties

The shear strength of a given adhesive and its ability to maintain that strength in service is very much dependent on the surface pretreatment given to the adherends and is therefore out of the hands of the designer. However, one adhesive parameter which to some extent is in the

TABLE IV
Effect of Bond Line Thickness on Adhesive Shear Properties

Adhesive	Thickness (mm)	G(MPa)	τ max (MPa)	γ_e (mm/mm)	γ_p (mm/mm)
A	0.178	456	24.1	0.033	1.309
	0.212	457	23.1	0.036	1.159
	0.190	507	25.1	0.031	1.230
	0.211	497	24.6	0.032	1.163
	0.458	533	23.6	0.029	1.000
	0.443	538	27.9	0.035	1.446
	0.440	483	22.4	0.034	1.075
	0.438	459	24.3	0.030	1.295
	0.836	456	17.1	0.030	0.735
	0.894	488	16.8	0.028	0.744
	0.864	454	16.4	0.029	0.714
B	0.153	649	35.0	0.034	1.327
	0.124	407	36.4	0.060	1.511
	0.107	506	35.4	0.050	1.681
	0.119	482	35.4	0.051	1.672
	0.427	675	28.9	0.031	0.887
	0.410	592	27.0	0.034	1.091
	0.399	599	23.8	0.033	0.531
	0.428	562	30.2	0.037	1.177
	0.879	674	26.6	0.027	0.310
	0.839	604	25.4	0.029	0.522
	0.898	588	27.3	0.025	0.969
0.895	550	24.8	0.029	0.870	
F	0.085	622	41.5	0.033	1.727
	0.085	417	40.5	0.067	1.643
	0.062	747	42.8	0.033	2.274
	0.062	489	40.5	0.046	2.268
	0.147	518	42.2	0.051	0.902
	0.168	1081	42.4	0.021	0.720
	0.142	643	42.7	0.035	0.807
	0.323	730	41.8	0.030	0.422
	0.267	705	46.7	0.041	0.628
	0.234	755	43.8	0.043	0.487
	0.274	727	36.2	0.034	0.337
0.337	689	37.1	0.036	0.641	

designer's control is the thickness of the bond line. Reference has already been made to the effect on shear strain to failure of small variations in bond line thickness. The investigation was extended to include larger variations up to 0.85 mm (nominal) on two of the cold curing epoxies (adhesives A and B) and one hot curing film (adhesive F), and the results are listed in Table IV. Adhesives A and B show a 27-30% fall off in shear strength when the bond line thickness is increased to about

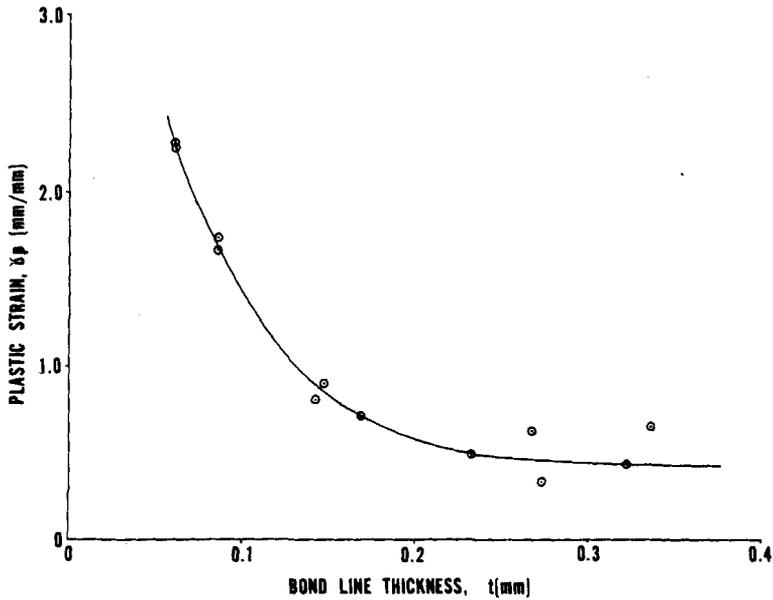


FIGURE 6 Variation of plastic strain with bond line thickness (adhesive F).

0.8 mm and an even greater decrease in failure strain. The film adhesive, F, shows no change in shear strength when the number of film layers in the joint is increased from one to six, but shows a very substantial decrease in plastic strain to failure (see Figure 6). Therefore with this adhesive, because the shear strength does not change, the Hart-Smith joint analysis would predict a larger load carrying capacity by increasing the adhesive thickness. In practice, however, this may not be the case because of the corresponding decrease in adhesive strain energy as a result of a reduced failure strain.

CONCLUSIONS

Adhesive shear stress-strain data have been produced by a torsional butt joint technique which is relatively quick to perform and is believed to be very accurate. Comparison of results with another worker using the alternative method of thick adherend lap shear testing has shown extremely good agreement.

The shear stress-strain behaviour of various structural adhesives have been compared, which has highlighted some important differences in their strain energies. The latter is also affected by the bond line thickness of the adhesive which lowers the plastic strain to failure as the thickness increases.

Acknowledgement

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