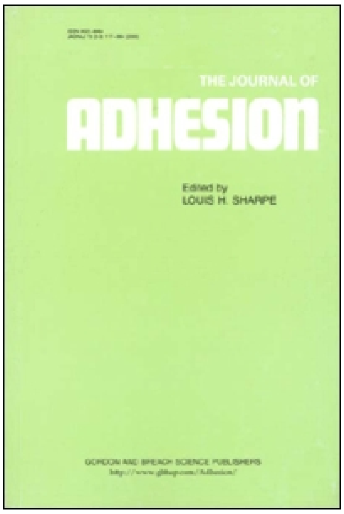

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Direct Measurement of Longitudinal Strains and Stresses within Single Lap Shear Adhesive Joints Using Neutron Diffraction

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The neutron diffraction technique has been used to investigate the longitudinal stresses in the adherend produced as a result of cure and due to the application of a tensile load in a single lap shear joint. A comparison has also been made between the stress distributions in loaded "aged" and "unaged" joints. The neutron diffraction technique is the only viable method of investigating these stresses within metal adherends and enables comparisons between predicted and measured stresses to be made. The results of these experiments cast doubt on some of the predictions from finite element modelling of adherend stress levels.

Keywords: Adhesively bonded joints; ageing; curing stress; epoxide

INTRODUCTION

A great deal of work using analytic and finite element techniques has been carried out to predict the distribution of strains and stresses within single lap shear (SLS) adhesive joints [1–5]. Of particular note is a recent overview of this topic by Adams *et al.* [1] and the reader is referred to the references contained therein. The previously-reported

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studies have concentrated on modelling the effects of adherend modulus and thickness, adhesive type and spew-fillet geometry on the strains and stresses within the SLS joint with externally applied loads. Existing models to explain the SLS joint performance utilise the fundamental bulk mechanical properties of the adhesive and adherends and, principally, consider the strains and stresses in the adhesive layer. There is, however, less available literature detailing such distributions in the adherend and a distinct lack of direct measurements of the stresses and strains within the adherend. This information is necessary to validate the existing models of joints.

To date, photoelastic studies have been used to provide an indication of the strains and stresses within polymeric adhesives and adherends; these experiments do not replicate the situation within much higher modulus metallic adherends. Strain gauges and Moiré interferometry have been used to evince such information but these techniques have their limitations since they only provide direct measurements of the surface strains.

Neutron diffraction is a technique which has been used to study residual and other strain and stress distributions within bulk metal pieces [6, 7]. With this technique a monochromatised neutron beam is incident upon the material and the Bragg angle at which constructive interference of the emerging neutron beam occurs for a selected metallic lattice plane is measured. The position of the Bragg diffraction peak provides a measure of the lattice spacing of the chosen plane in a particular direction within the sample. The equation:

$$\lambda = 2.d.\sin\theta \tag{1}$$

where λ is the neutron wavelength, θ is the Bragg angle and d is the lattice spacing. Differentiating Eq. (1) and introducing the lattice spacing, d_0, from a strain-free reference leads to an expression for the strain:

$$\varepsilon = (d - d_0)/d_0 = -\cot\theta.\Delta\theta \tag{2}$$

with $\Delta\theta$ the difference in Bragg angle between the strained and unstrained states. A particular difficulty in the use of diffraction methods for strain measurement is the determination of the zero-strain lattice parameter d_0 since, except in the case of annealed powders, it is difficult to be sure a specific sample is truly free of residual stress. In the present study, only comparative data are considered so the determination of an absolute d_0 is not critical. From a knowledge of the appropriate modulus for the selected lattice plane the local stresses in a particular direction can be determined.

The advantage of using neutrons as a diagnostic tool for this application is their high penetration depth in metals compared with alternatives such as X-rays which are widely used to measure stresses in surface layers [8]. This penetration permits in-depth rather than solely surface information to be obtained. Slits and collimators are used to define a finite gauge volume within the material and by rastering the sample a three-dimensional map of the stress distribution within the sample can be determined. The gauge volume is the volume within the sample which is both illuminated by the neutron beam and from which diffracted neutrons can reach the detection system. The measured diffraction angle is, therefore, an average of the diffraction angles from all the crystallites within the gauge volume which are oriented so as to diffract neutrons towards the detector. In principle, the smaller the gauge volume the more accurately can the strain within a small region of the sample be defined but the resulting reduction in diffracted neutron beam intensity due to the use of very small gauge volumes considerably worsens the signal-to-noise ratio and lengthens the experimental time. Gauge volumes of the order of 1 mm^3 are generally accepted to be a reasonable compromise in neutron diffraction work.

The main objective of the present study was to evaluate the usefulness of neutron strain-scanning applied to the SLS adhesive joint. Experiments were carried out to investigate the following: (a) the presence of residual strains within the adherends of SLS joints introduced by shrinkage during the high temperature cure phase of an epoxide adhesive and (b) the longitudinal (*i.e.*, in the direction of the long axis of the adherend) stress distributions within "unaged" and "aged" SLS joints in the loaded condition.

EXPERIMENTAL

SLS adhesive joints were prepared using 50 mm × 16 mm × 3 mm CR1 mild steel adherends (see Fig. 1). The adherends were grit blasted and subsequently degreased in acetone prior to assembly. Additionally, the

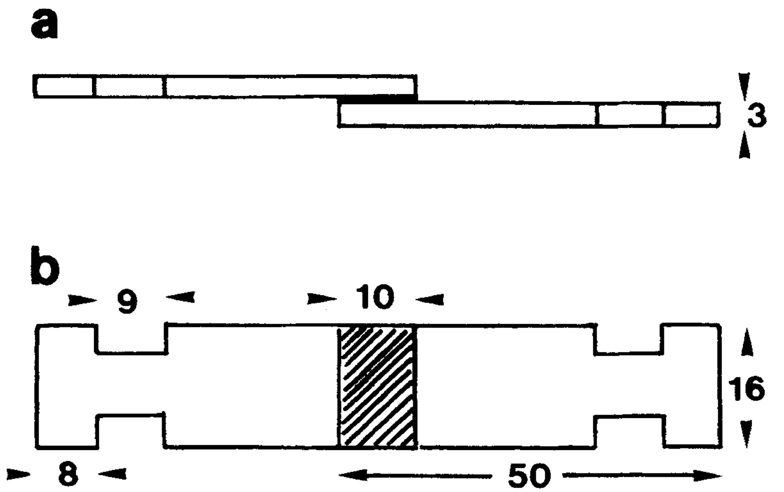

FIGURE 1 Dimensions of the joints used in the study. All dimensions in mm. (a) side view, (b) plan view.

samples used in the residual strain experiment (a) were vacuum annealed for 4 hours at 700°C and then slow cooled and demagnetised in order to reduce the level of residual stress in the sample before bonding. The adhesive used was Araldite AV119, a 120°C curing single-part epoxide supplied by Ciba Polymers with a 1% addition of 250 micron "Ballotini" glass spheres for bondline thickness control. The joint overlap area was 16 mm × 10 mm. For the ageing experiments (b) two types of joints were analysed. These were freshly prepared "unaged" samples and "aged" samples. The "aged" samples were prepared by immersion in deionised water at 60°C for approximately 10 days with a simultaneously-applied load of 1 kN.

The ideal set-up for maximum spatial resolution in a neutron strain-scanning experiment is to have the angle between the input and diffracted beams as close as possible to 90°, *i.e.*, $2\theta = 90°$. The D1a diffractometer at the Institut Laue-Langevin used for these experiments has a number of user-selectable neutron wavelengths ranging from 1 Å to 4 Å. The Bragg peak with maximum intensity for the steel used in this experiment peak was found to be the 110 peak (in all that follows the terminology for describing peaks is the standard crystallographic Miller indices terminology). However, constraints imposed by the use of the *in-situ* stress rig meant that the most suitable peak to use in the ageing experiment was the 211 peak. The neutron wavelength used for the 211 peak was 1.514 Å, giving a 2θ of 80.6°, and for the 110 peak $\lambda = 2.993$ Å, giving a 2θ of 95.2°. In all cases the joints were oriented in such a configuration so as to determine the strains in the longitudinal direction. The symmetry of the SLS joint is such that, for maximum efficiency in the use of the neutron beam, only part of each sample was analysed. Figure 2 indicates the region studied as well as the x, y, z coordinate directions used in the study. In summary, the x-direction is measured through the thickness of the joint from the glueline, y is along the length of the joint in the direction stress is applied, and the z-direction is along the joint height. The region chosen for study which is illustrated in Figure 2 should, by symmetry, give a picture of the stresses throughout the joint.

RESULTS

(a) Residual strains within SLS joints: For this experiment a guage volume of 2 mm (in the z-direction)$\times$0.5 mm $\times$ 0.5 mm was selected. A summary of the longitudinal stress (stress in the y-direction) for

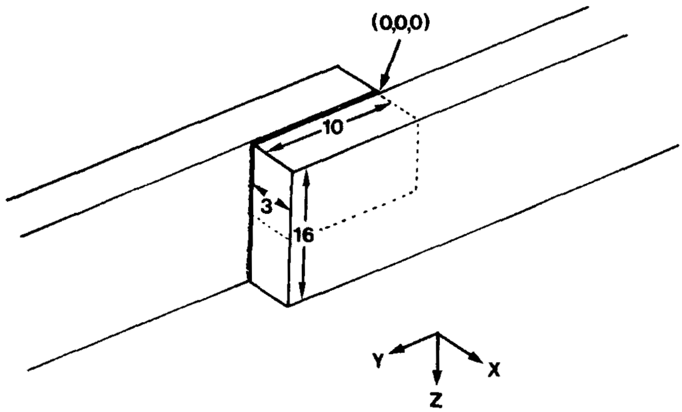

FIGURE 2 Origin and directions of coordinate system. The origin is the point on the metal/adhesive interface on the upper side of the metal adherend in line with the corner of the second adherend as indicated. The upper section of the front adherend, as indicated by the dotted lines, was the region analysed in this study. All dimensions are in mm.

the x, y, z points measured is given in Table I. Stresses were evaluated using the Kroner elastic constant (227 GPa) for the 110 plane. The stresses are calculated from strains measured relative to the point (2.6, 8.8, 7) which is close to the centre of the adherend and is generally accepted to be free of longitudinal stress. A simple substantiation of this in the case of loaded joints is given later in the text. The $x = 0.6$ plane is adjacent to the central plane of the joint (the metal polymer interface), $x = 1.6$ is in the centre of the adherend and $x = 2.6$ close to the major free surface. Throughout the study the diffraction peaks were fitted with a Gaussian function on a linear sloping background and very good fits to all peaks were obtained; typical values of the goodness of fit coefficient, r^2, were 0.98 – 0.99. The centre of the Gaussian was taken as a measure of peak position and the accuracy of the fit generally gave an error of the order of $\pm 0.005°$ in 2θ. Points were chosen such that the complete gauge volume was within the sample for all measurements since complications may arise when the gauge volume bisects the free surface which lead to asymmetric peaks and possible large errors in the determination of the peak centre.

TABLE I Curing stress (MPa) as a function of position in adherend. For definitions of position see Figure 1, all dimensions in mm. Accuracy of the stress measurements is ± 10 MPa. Positive values indicate tension, negative values compression

	$z = 1$			$z = 4$			$z = 7$		
y	$x = 0.6$	$x = 1.6$	$x = 2.6$	$x = 0.6$	$x = 1.6$	$x = 2.6$	$x = 0.6$	$x = 1.6$	$x = 2.6$
0.5	−28			1			−25		
1.2	7			−14			−3		
1.9	42			−3			7		
5	−7			19			15		
8.1	42			−9			18		
8.8	−25			−13			0		
0.5		2			30			−2	
1.2		7			−6			16	
1.9		39			33			23	
5		39			33			19	
8.1		−8			−16			−4	
8.8		5			−32			−3	
0.5			−1			20			24
1.2			11			−15			−2
1.9			31			−6			27
5			27			21			14
8.1			6			5			4
8.8			0			−12			0

(b) The "aged" and "unaged"samples were both loaded to 2.5 kN in a stress rig which enabled measurements to be made under load *in-situ* in the neutron beam. For these measurements a larger gauge volume of 2 mm (z-direction)$\times$ 1 mm $\times$ 1 mm was used in order to allow the measurements to be carried out in the time available using the 211 Bragg peak. Space constraints imposed by the use of the stress rig meant that the more intense 110 peak could not be used as previously. The elastic modulus for the 211 Bragg peak was obtained by carrying out a subsidiary experiment in which the peak position in a portion of the sample well away from the joint was monitored as the sample was loaded in increments of 600 N to 2.4 kN. A straight line fit to the stress-strain curve obtained gave a modulus of 224 ± 30 GPa; this is in good agreement with the theoretical value of 219 GPa. A strain gauge with a 1 mm gauge length bonded on the surface close to the position $(3, 8, 7)$ in the coordinate system used in these experiments, and in such an orientation so as to detect longitudinal strains, showed no change in resistance as the load on the joint was increased from 0 to 2.5 kN. It can, therefore, be concluded that this position remains stress-free. The d spacing at the measurement position $(2, 8, 8)$ was, therefore, used as the d_0 and strains, and hence stresses, measured relative to this point. Because of the use of a larger gauge volume, two rather than three x-planes (planes parallel to the adherend surface) were examined in these experiments. The central planes of these gauge volumes were at $x = 2$ and $x = 0$. This latter measurement plane will include contributions from metal on both sides of the adhesive but the longitudinal stress should be almost the same at points just on either side of the glue line, so the measurement will give a good indication of the stress in the metal close to the glue line. The results are set out in Table II. The $x = 0$ results summarised in Table II show very large values of stress. However, if the 2.5 kN load is averaged over an xz-plane, and the reasonable assumption that the stress falls away rapidly with distance from the glue line is made, the stresses are not unreasonable. Since the purpose of this experiment was to make comparative tests of the stress distributions in loaded "aged" and "unaged" joints the differences between the stress values in the joints is required. Differences along the glue line are illustrated in Figure 3 and clearly show a redistribution of load occurring with ageing. The load carried by the aged sample is considerably decreased in the material along the upper surface of the glue line ($x = 0, z = 2, y = 2, 4, 6, 8$) and, in consequence, increased in the central region ($x = 0, z = 8, y = 2, 4, 6$). This is likely to be due to plasticisation of the adhesive in the outer areas of the joint and a consequent reduction in load-carrying capacity and relaxation in the strain of the adjacent metal.

TABLE II Stress (MPa ± 15 MPa) in "aged" and "unaged" joints under a 2.5 kN load. All x, y, z positions in mm

x	y	*Unaged* $z = 2$	*Aged* $z = 2$	*Unaged* $z = 5$	*Aged* $z = 5$	*Unaged* $z = 8$	*Aged* $z = 8$
0	8	230	184	177	176	155	152
0	6	218	187	160	165	129	149
0	4	223	213	177	162	114	134
0	2	249	233	206	200	190	208
2	8	57	11	2	−11	0	0
2	6	71	53	70	27	54	−8
2	4	68	53	81	46	61	31
2	2	54	38	68	57	65	42
2	0	61	44	79	62	70	38

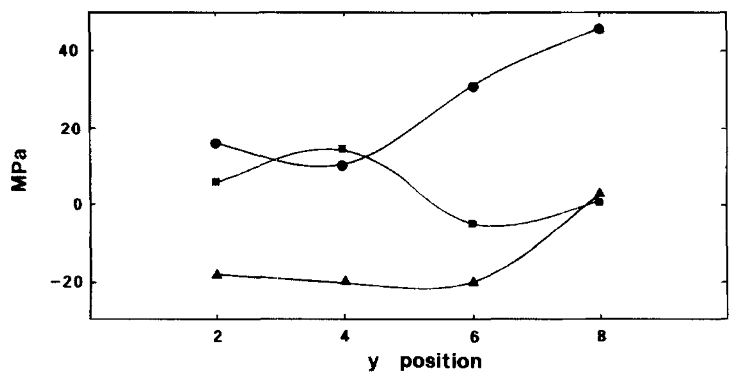

FIGURE 3 Plot of the stress difference between unaged and aged joints in a loaded SLS joint close to the glue line ($x = 0$). The stresses are $\sigma_{\text{unaged}} - \sigma_{\text{aged}}$. ● $Z = 2$; ■ $Z = 5$; ▲ $Z = 8$.

DISCUSSION

The results of experiment (a) summarised in Table I can be compared with the modelling predictions of Adams *et al.*, for curing stresses in a lap joint [5]. Adams' predictions are for shrinkage and cooling to produce tensile stresses throughout the adhesive layer which are of constant magnitude along the central region of the joint but decrease towards the joint edges.

These should be balanced by compressive stresses in the adherend whose general distribution follow the same pattern as the adhesive stresses *i.e.*, greater and of constant magnitude along the central region and decreasing towards the edges. The results for $x = 0.6$ which is the region that should be compared with the modelling results are illustrated in Figure 4. The results of these experiments do not agree with the predictions of Adams *et al.* The stress close to the interface is not uniform along the central region for either $z = 1$ or $z = 4$ and, in fact, tends to be tensile in the centre and compressive at the edges. These results, therefore, disagree in almost all respects with Adams' predictions. However, it should be noted that the accuracy of the experimental results are $\pm$ 10 MPa and that the system (AV119/steel) differs from that used by Adams *et al.*, so a direct comparison of stress magnitudes is not valid although the general form of the results should be similar.

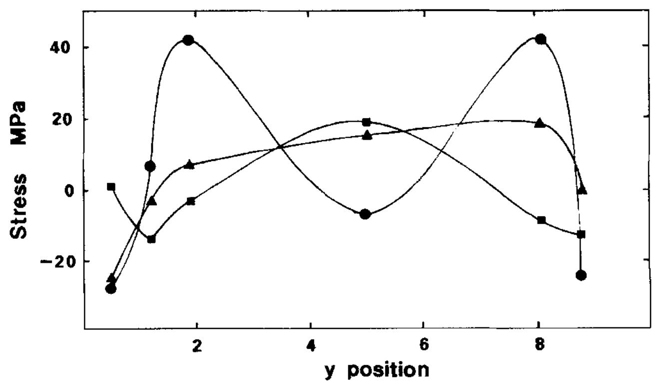

FIGURE 4 Stress in the adherend as a functional of longitudinal position close to the glueline in an unloaded SLS joint. The curve $Z = 1$ (●) illustrates measurements close to the free surface, $Z = 4$ (■) measurements one-quarter way into the joint and $Z = 7$ (▲) measurements close to the centre line.

Tsai and Morton [4] have modelled the stresses in the adherends in a loaded SLS joint and predict that the stresses are maximum along the joint edge ($x = 0, y = 0$ plane) and decrease to a low and almost constant value within the first 10% of the joint length. They also predict that the load should remain almost constant over xy-planes before dropping off as the edge of the joint is approached. These predictions can be compared with the "unaged" data from Table II. The general form of the stresses agree with the finite element predictions in that the stresses close to the joint ends are greater than the values in the central region. However, there is not sufficient resolution in the y-direction to see if the detail agrees with that of the model but the magnitude of the stresses in the central region do seem too large compared with the model. Stresses in the region of 200 MPa may seem unrealistically large but they are of the same order as those reported by Kawada and Ikegami [9] from strain gauge measurements on steel embedded in epoxide and we, therefore, have confidence in their validity. The data over a given xy-plane adjacent to the glue line does not agree with the model predictions. The stress is consistently greater close to the free surface and decreases as the central line is approached. This is opposite to the model predictions.

CONCLUSION

The studies described here have been of a preliminary nature and have demonstrated the use of neutron strain-scanning in the provision of information on adhesively-bonded SLS joints. To date, only limited neutron beamtime has been available for data acquisition and, in consequence, relatively large gauge volumes have been used in order to provide suitably high quality data for analysis. A reduction in gauge volume giving better spatial resolution and the opportunity to investigate a greater number of points within the adherend would clearly be of benefit. In addition, three-dimensional strain distributions could be obtained so that peel forces close to the metal-polymer interface could be evaluated. All of these improvements would be at the expense of much increased experimental time.

The present study has provided useful information on the residual stress pattern caused by adhesive cure and the loading of both "aged" and "unaged" joints. The data obtained have cast doubt on the results of recent finite element models and provided evidence for load redistribution during ageing of joints. More work in this field is clearly required since it provides the only method of obtaining three-dimensional strain distributions within the adherend and the possibility of validating current finite element models.

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