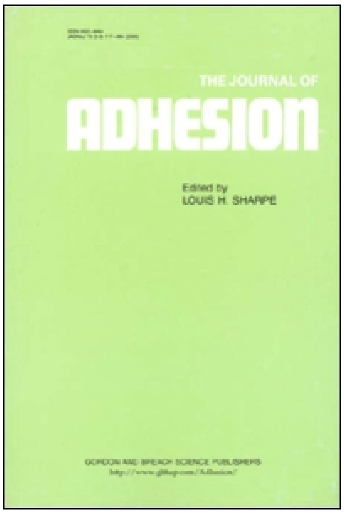

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Study of the Crack Deviation Angle in Adhesive Ruptures of Ceramic Adhesively-Bonded Assemblies

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Study of the Crack Deviation Angle in Adhesive Ruptures of Ceramic Adhesively-Bonded Assemblies

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Mechanical tests are commonly used to characterize structural adhesively-bonded assemblies since they give rise to typical values such as rupture load, stress, strain or energy. It is shown herewith, that for a single lap compression-shear test, it can also be of great interest to analyse the bonded assembly after rupture. The investigated fracture surfaces can reveal the presence of normal stresses in the joint and a valuation of their intensity can be done, from the crack deviation angle value. Actually, for adhesive ruptures, a transition zone exists where the crack propagates from one interface to the other. It is demonstrated from the failure occurrence that the deviation angle value does not depend on the adhesive itself but on distributions in the joint of different kinds of stresses (shear and normal ones). In addition to experimental angle measurements, numerical modelling was performed with the finite element computer code ANSYS. Experimental values match numerical values.

KEY WORDS: Single lap compression-shear test; structural adhesive; ceramic adherends; failure criterion; angle of deviation; finite element simulation.

INTRODUCTION

Mechanical rupture tests can be used to study the adhesive-substrate adherence. In the case of adhesive bonding of metals, the standardised single lap tensile-shear test (ASTM D1072-83) is commonly used. The problem is the unsuitability of this test for brittle substrates such as ceramics since it very often leads to cohesive failure of the substrates. It is the reason why we have developed[1–3] a single lap compression-shear test where specimens consist of adhesively-bonded parallelepipedic adherends, blocked for the lower adherend and pushed for the upper one (Fig. 1). This test gives very good results when used with brittle ceramics such as silicon carbide (SiC) or alumina (α-Al_2O_3).

Test purity is generally not compatible with a bonded assembly having a simple shape and, as a result, a parasite mode I appears in the case of our compression-shear

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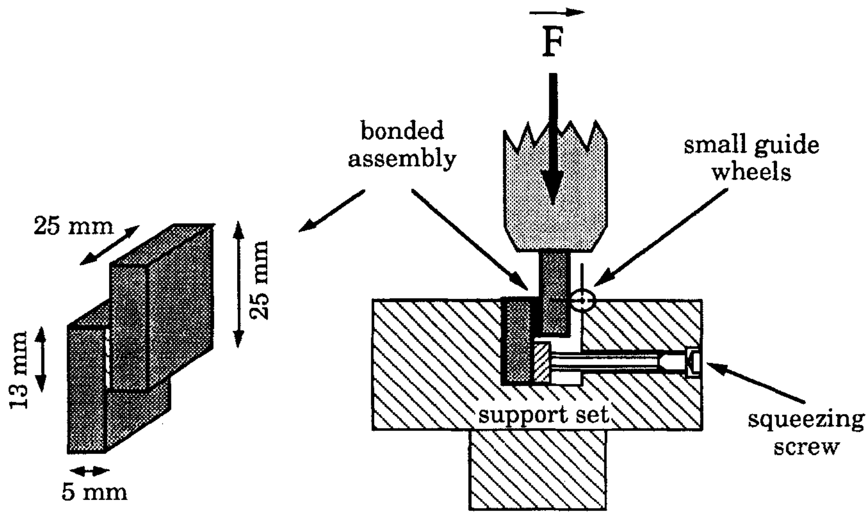

FIGURE 1 Description of the single lap compression-shear test and specimen geometry.

test. This fact is well known since the single lap tensile-shear test has been largely studied and analytical expressions of normal stresses in the joint have been proposed.[4] The only difference between these two tests (with identical experimental conditions) is the mathematical sign of normal stresses.[2,3] They are compression stresses for the compression-shear test and tensile stresses for the tensile-shear test.

We already showed[2,3] that these normal stresses are of great importance in joint deformation and failure processes. The aim of this paper is to demonstrate experimentally the presence of these stresses by means of a careful study of fracture surfaces and especially with the measure of the crack deviation angle as we will see further. Thus, this study will be divided in two parts: the first section will be devoted to experimental (materials description, experimental measurement of the crack deviation angle and appraisal of normal stresses), and the second section to comparative calculations of that angle by means of failure criteria. Finite element modelling of the crack tip will be performed using ANSYS code.

EXPERIMENTAL

Substrates used in this work are made of silicon carbide (98.5% purity) provided by *Céramiques & Composites* (FRANCE). Two epoxy adhesives are used: EC286 (*Emerson & Cuming*) and Hysol EA9321 (*Structil*). Some properties of these three materials are shown in Table I.

When both adhesives are compared it is noticed that they are quite different as far as rigidity is concerned and this difference can be explained by the amount of filler powder in each adhesive:

- 43% (w/w) for EC286 (fillers are silica and alumina)
- 24% (w/w) for Hysol EA9321 (fillers are aluminum and silica).

TABLE 1
Elastic properties (Young's modulus E, Poisson's ratio ν) and density (ρ) of silicon carbide. EC286 and Hysol EA9321

	E (GPa)	ν	ρ
SiC	420	0.16	3.21
EC286	5.8	0.34	1.65
EA9321	3.8	0.36	1.36

Adhesion between SiC and these adhesives greatly depends on the surface treatment applied to the substrates. This paper deals only with ruptures of bonded assemblies which are mainly adhesive, thus SiC substrates are only degreased (in 1, 1, 1-trichloroethane), rinsed (in demineralised water) and air dried at 80°C for 30 min. This surface treatment, which leads to a relatively low surface free energy of SiC substrates, always involves mainly adhesive failures,[2] as it is more precisely described further in this section.

The adhesive joint geometry is as follows: lap length is 13 mm and thickness 0.2 mm. The bonded area is delimited by an auto-adhesive Teflon coating to be sure that the spew filet never participates in the bonding. This enables one to obtain identical bonded area for each bonded assembly and when failure loads ($F_{failure} = 14.5$ kN)[2] are analysed, small standard deviations are observed ($\pm$ 5% for five trials) indicating a good reproducibility. It is important to notice that these failures are brittle since no ductile behaviour is observed on load-displacement curves (Fig. 2). This is true for both adhesives, the only difference being the different slope of these curves indicating a difference of rigidity.

When a failure is obtained, the fracture surface observation can lead to interesting information. First of all, it is possible to determine if the failure is really adhesive.

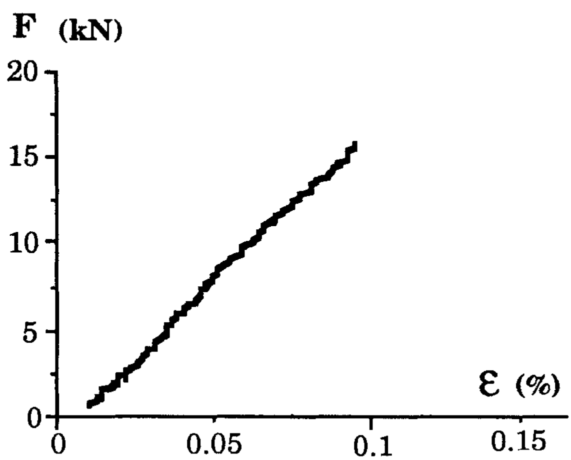

FIGURE 2 Load-displacement curve obtained for an adhesive failure between SiC and Hysol EA9321 adhesive.

For several authors[5–7] adhesive failures do not exist and fracture propagates in an interphase created between bulk substrate and bulk adhesive. However, we will see later that the referred-to ruptures can reasonably be called *adhesive failures.*

In that case, Scanning Electron Microscope (SEM) is a good way to detect the presence of adhesive remaining on substrates since silicon carbide conducts electricity. Therefore, when existing, adhesive traces would appear as very bright areas since they are insulating. In fact, nothing but SiC was observed with SEM on adherend surfaces after adhesive failures. On the other hand, complementary analysis using X-Ray Photoelectron Spectroscopy (XPS), revealed the presence of a very small quantity of adhesive remaining on the SiC surfaces.[2] This quantity is less than 5% of the analysed surface. As SiC substrates are not ideally smooth and show peaks and valleys (arithmetic rugosity: $R_a = 0.43\ \mu m$, total rugosity: $R_t = 9.3\ \mu m$), it seems that the small quantity of adhesive detected by XPS comes from the adhesive remaining in valleys after shear rupture in the interfacial zone.

There is another zone where the fracture propagates through the adhesive, since in most cases the crack starts near a substrate and is deflected towards the other. We are particularly interested in this area which is illustrated in Figure 3 of this work. The crack propagation begins at the interface and goes across the adhesive joint along an inclined plane (Fig. 3). The area of this plane represents around 2% of the bonding area.

The crack does not propagate from one substrate to the other in a hazardous way and the area where this phenomenon occurs requires careful study. In fact, here an angle α (deviation angle) can be measured when following a straight line between points

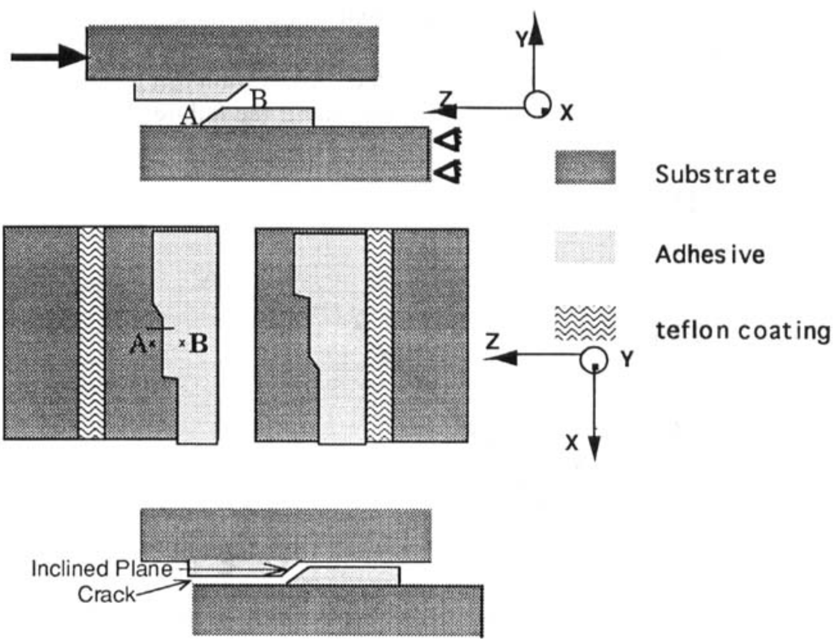

FIGURE 3 Schematic illustration of a fracture surface showing that the crack passes from one interface to the other, creating a small zone of cohesive fracture in the adhesive.

A and B (see Fig. 3) Such a measure was performed using three-dimensional profilometry. The profilometer used in this study is a Perthen S6P, the contact sensor of which is a diamond tip with a 5 micron radius of curvature. Its maximal amplitude is 250 μm (which is quite suited for our measurements since the joints are only 200 μm thick) and its vertical sensitivity is 0.01 μm.

For each adhesive, 30 measurements were made and two examples are provided in Figure 4. A good reproducibility is obtained since standard deviation represents around 5% of the average value which is equal to 57° (± 3°) for both adhesives.

The plane where the crack passes from one interface to the other is not always the same. In fact, this phenomenon can be located in almost **any** place along the joint lap length.† This result is illustrated in Figure 5 where α values are plotted against the position (**z**) where the crack is deflected. For all the studied bonded assembly ruptures, the lap length was 13 mm, thus **z** varies between 0 and 6.5 mm, the positions **z** = 13 mm and **z** = 0 mm being similar since the center of the joint is a center of symmetry (Fig. 3). We must explain that, for each adhesive, the 30 measurements are made on 30 different bonded assemblies and not on 15 assemblies where measurements would be made on both substrates.

The analysis of Figure 5 shows that the deviation angle does not depend on **z** and that no **z** value lower than 1 mm was encountered.

The Maximal Normal Stress Criterion

The fact that the same deviation angle was found for two different adhesives seems to indicate that the polymer quality of the adhesive is not involved in this phenomenon and that the explanation lies probably in the bonded assembly geometry. In this kind of test, it is well known that the lap length and the joint thickness are two major parameters which define the stress distributions in the joint.[4]

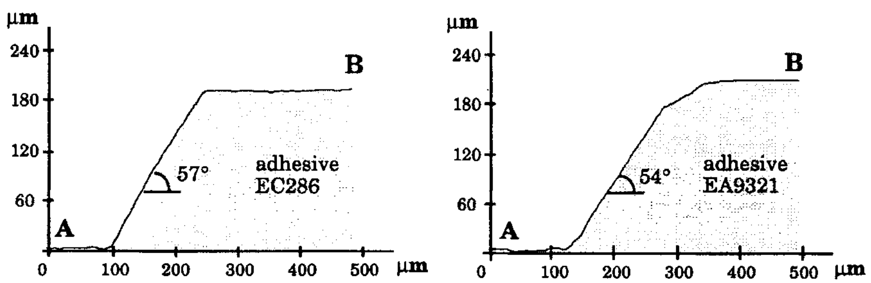

FIGURE 4 Failure profiles and deviation angles obtained by the mean of a profilometer for adhesives EC286 (left) and Hysol EA9321 (right). (points A and B are those previously defined in Fig. 3).

† If the failure is joint cohesive and far from the interface and starts everywhere in the overlap zone, the crack does not change direction (the deviation angle is nil). If it would start on or near the interface, we will have the deviation angle value of 57°.

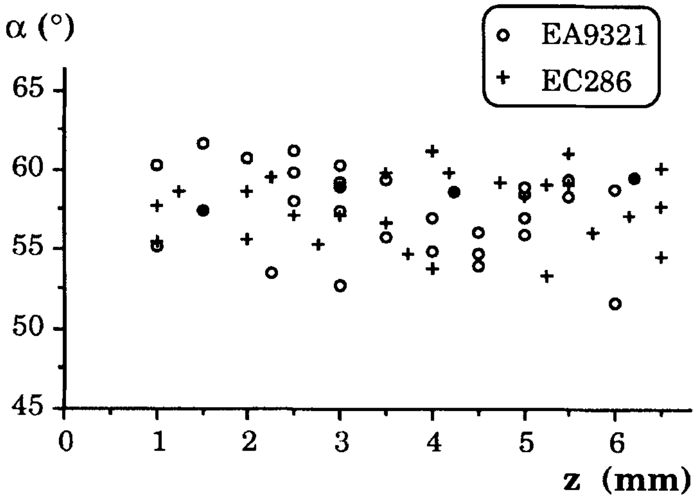

FIGURE 5 Values of the crack deviation angle (α) as a function of the position (z) where it is measured.

As will be demonstrated in the following sections, the fact that normal stresses are present in the joint in addition to shear stresses must be considered to explain the values of α measured on the fracture surface.

Fracture mechanics shows that the fracture process is completely directed by the way stresses are distributed in the material and, therefore, several rupture criteria have been developed for adhesive joints.[8-11] None of these authors give a suitable criterion to calculate the deviation angle.

A simple criterion, called the Maximal Normal Stress Criterion,[12] is well adapted to our experimental case and we use it as a first approximation for the valuation of normal stresses. This criterion, only usable for brittle and elastic behaviour of materials, specifies that the crack propagates in a plane which is perpendicular to the direction where tensile stresses are maximal. For example, in the case of a pure tensile test, failure occurs in a plane which is perpendicular to the applied load and in the case of a pure shear test it occurs in a plane forming an angle α ($\alpha = 45°$) with the direction of the applied load. This angle (45°) is calculated by determining the position of the principal reference relative to the global reference.

When a parasite mode is present (mode I in our case)[2,3] the determination of the fracture plane is the same and the general relation giving the expression of the deviation angle as a function of the shear stress (σ_{YZ}) and the normal stress (σ_{YY}) is as follows (Fig. 3):

$$\tan(2\alpha) = \sigma_{YZ}/\sigma_{YY} \qquad (1)$$

where σ_{YY} is positive for tensile stresses and negative for compression stresses.

Figure 6 shows what happens when normal compressive or tensile stresses are present in addition to shear stresses. In the case of compressive normal stresses, the deviation angle must be greater than 45° and smaller for tensile normal stresses.

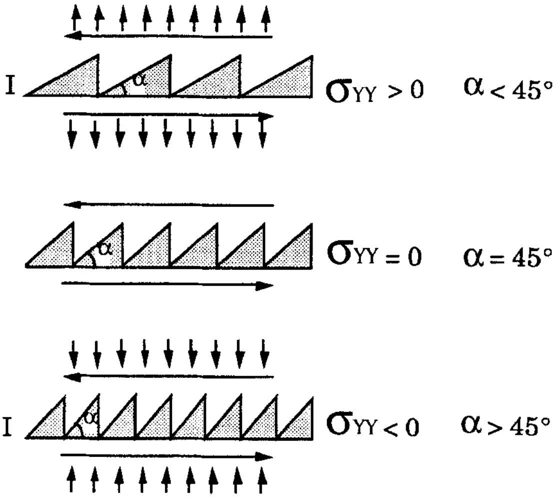

FIGURE 6 Fracture propagations in case of mixed modes I and II.

Previous information concerning the value of α indicates, when considering our experimental result ($\alpha = 57° \pm 3°$), that normal stresses are compressive stresses. It is really what happens for the single lap compression-shear test as already shown.[1–3]

By using the relation above, it is possible to evaluate normal stresses present in our joint, considering that apparent σ_{YZ} is equal to the failure load divided by the bonded area ($\sigma_{YZ} = 44.6$ Mpa). With $\alpha = 57°$, $\sigma_{YY} = 19.8$ MPa is obtained. The ratio σ_{YY}/σ_{YZ} (independent of the applied pressure) is equal to 0.44.

In the literature,[1] using the same single lap compression-shear test with α-alumina (99.7% purity) substrates and with AV 119 adhesive from *Ciba*, this ratio is equl to 0.40.

CALCULATIONS OF THE CRACK DEVIATION ANGLE AND FINITE ELEMENT ANALYSIS

To verify the accuracy of the value of this angle, a finite element simulation of the test has been made for linear behaviour of the materials. Boundary conditions are chosen close to those encountered in experimental test conditions.

A three-dimensional finite element study was undertaken to analyse the specimen behaviour under mechanical load *in the case of adhesive failure.*

Computations were made using the ANSYS[13] finite element programme in which an automatic mesh generation exists, allowing change of design parameters. The element used was a 20-node isoparametric structural solid (named Solid95 in the ANSYS library), with three degrees of freedom in translatory motion. This element was used for the substrates as well as for the adhesive joint. For a defect length equal to 5 mm, the finite element model (for the half sample) includes a total of 10837 nodes and 3240 elements (Fig. 7). There are 648 elements which span the adhesive thickness. Such a refined mesh leads to a good accuracy of numerical results. For the numerical

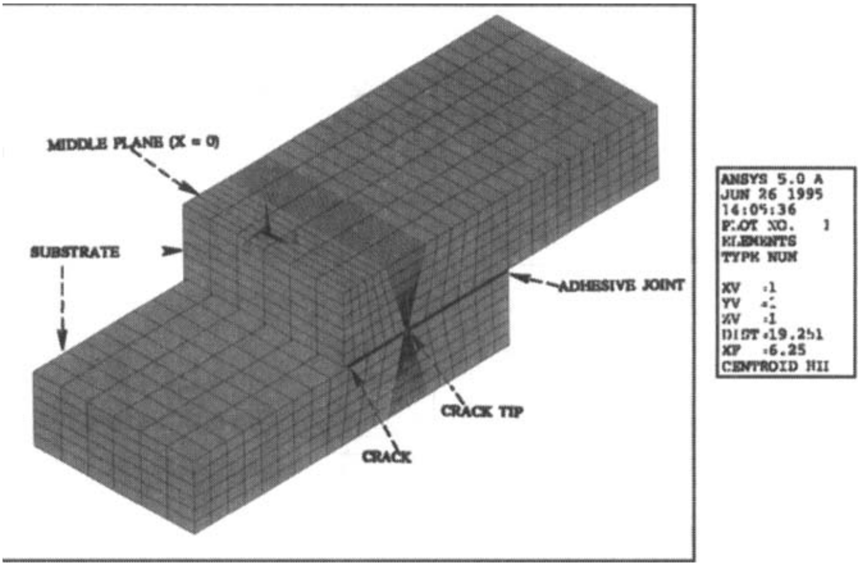

FIGURE 7 Finite element mesh of the half specimen.

simulation, only the shear mode will be studied, although it is difficult to obtain a pure mode II in our experimental test conditions, to dissociate easily the factors required in the fracture mechanics analysis. We apply a pressure to the upper substrate plane. Figure 8 gives the boundary conditions used in the simulation. The behaviour of the bonded specimen is assumed to be elastic with adhesive failure. For finite element analysis, the studied adhesive is Hysol EA9321.

Whenever a rupture criterion is used, it leads to only one direction of the crack deviation. Then, we must make two studies: the first leads us to find the first deviation angle which will be along the interface and a second study gives the second deviation through the adhesive joint.

Owing to experimental results, for the numerical simulation we directly have skipped over the first stage and we have introduced a defect at an interface.

For a given crack length introduced in the finite element model, we use a criterion to determine the deviation angle at the crack tip level.

The Erdogan Criterion

When considering a crack contained in one material, where the crack tip is in contact with a second material, Williams[14] has shown that in the general case the singularity of the stresses is of the order $r^{-(1/2)+i\varepsilon}$ where ε is the bi-elastic constant (ε will be defined further) and r the distance from the crack tip (singularity is a complex number which induces a oscillating deformation of the crack lips). The best example showing this oscillating character is when the crack follows the surface separating the two ma-

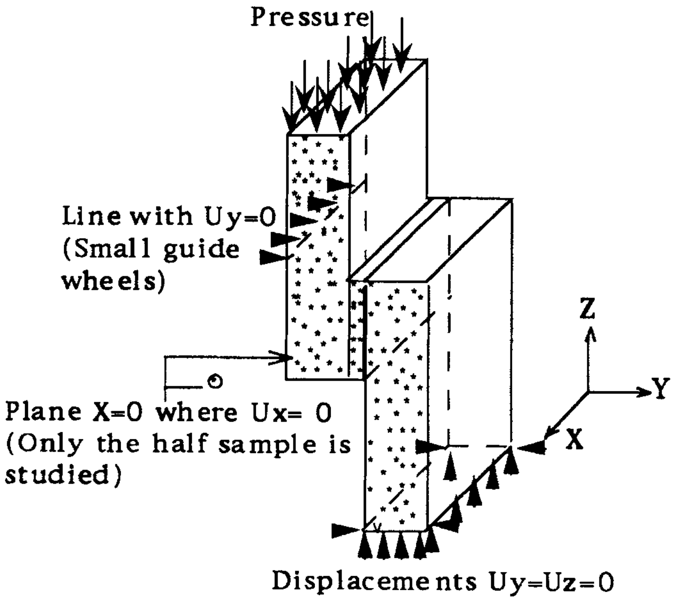

FIGURE 8 Boundary conditions applied in the simulation.

terials.[15] Comninou[16] shows that, in the neighbourhood of the crack tip when the faces of the crack are in *contact*, this oscillation effect disappears and the singularity becomes real. It is the case for the single lap compression-shear test used here. In the absence of a physically more acceptable criterion for this type of fracture problem, a simple criterion such as the Maximum Stress Criterion is adequate. This criterion, developed by Erdogan,[17] may be stated as "the fracture propagation will take place radially in the direction $\alpha = \alpha_{\max}$ for which the cleavage stress $\sigma_{\alpha\alpha}(\delta_p, \alpha)$ is maximum, where δ_p is the size of the fracture process zone around the crack tip".

The process zone depends on the microstructure and the continuum properties of the material as well as on the environmental conditions. If the "weak link" around the singular point is the interface, then the last criterion may be modified as follows:

Find the angle, $\alpha_{\max}$, which maximizes the stress, σ_d, as defined by (see Fig. 9):

$$\sigma_d = \begin{cases} [(\sigma_{\alpha\alpha})^2 + (\sigma_{r\alpha})^2]^{(1/2)} & \text{if } \sigma_{\alpha\alpha} > 0 \\ \sigma_{r\alpha} & \text{if } \sigma_{\alpha\alpha} < 0 \end{cases} \tag{2}$$

where $\sigma_{\alpha\alpha} = \sigma_{zz}\sin^2(\alpha) + \sigma_{yy}\cos^2(\alpha) - 2\sigma_{yz}\sin(\alpha)\cdot\cos(\alpha)$ and $\sigma_{r\alpha} = [-\sigma_{zz} + \sigma_{yy}]\sin(\alpha)\cdot\cos(\alpha) + \sigma_{yz}[\cos^2(\alpha) - \sin^2(\alpha)]$. The critical stress, σ_d, is considered to be "a material constant" representing the cohesive strength of the constituent materials.

When calculations are made, stresses and strains can be obtained for each angle α of mode I and it is, therefore, possible to have the values of stresses σ_{YZ}, σ_{YY}, σ_{ZZ} near the crack tip and to calculate the angle $\alpha_{\max}$ which maximizes σ_d (see Fig. 9).

For a defect length equal to 5 mm, the maximum displacement (6.37 μm) is located on the upper substrate plane under pressure. The distribution of Von Mises stresses, σ_{VM},

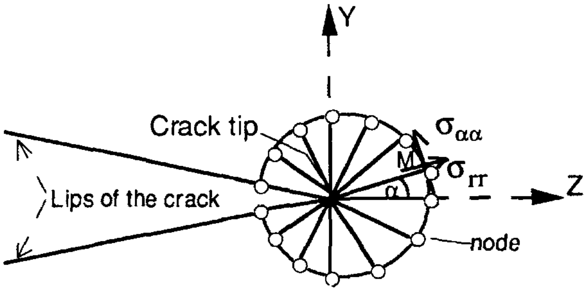

FIGURE 9 Finite element mesh near the crack tip.

defined as:

$$\sigma_{VM}{}^2 = (1/2)[(\sigma_{XX} - \sigma_{YY})^2 + (\sigma_{XX} - \sigma_{ZZ})^2 + (\sigma_{YY} - \sigma_{ZZ})^2 + 6(\sigma_{XY}{}^2 + \sigma_{XZ}{}^2 + \sigma_{YZ}{}^2)] \quad (3)$$

in the assembly is not homogeneous (Fig. 10) and their maximal value (104 MPa) is located in the adhesive joint, near the crack tip (Fig. 11). The calculation of the numerical deviation angle using the Erdogan criterion gives 52°.

To check that the deviation angle does not depend on the position, z, of the crack tip, we introduce a crack length equal to 2 mm. Figure 12 gives Von Mises stresses (in the middle plane of the sample ($X = 0$), see Fig. 7), with a distribution different from the one obtained for 5 mm. The close-up of Von Mises stresses, given in Figure 13, shows that the maximum of these values (78 MPa) is smaller than the one obtained for 5 mm, and the use of the Erdogan criterion leads to a deviation angle equal to 51°.

The small discrepancy between numerical and experimental deviation angle values is probably due to the undervaluated parasite mode I in the numerical modelling: in the numerical modelling the normal displacements, U_{yy}, are negligible or nil owing to the fact that we numerically impose a *pure controlled* vertical displacement. It is not the

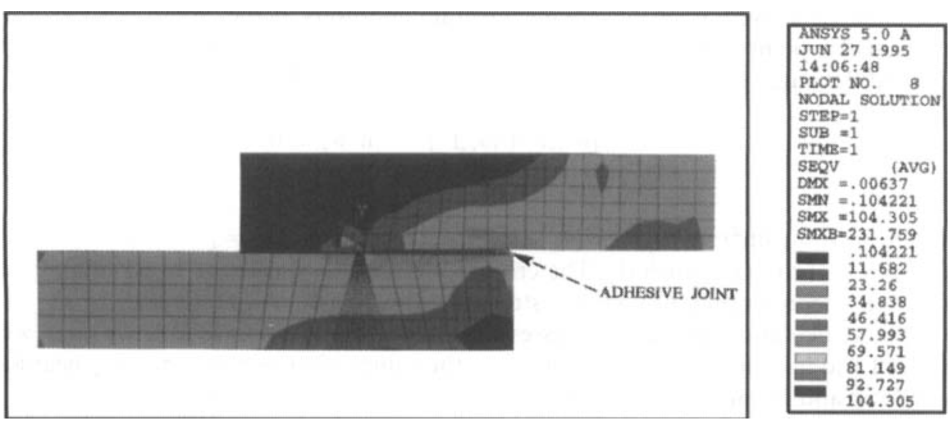

FIGURE 10 Von Mises stresses in the middle plane ($X = 0$) for a crack length equal to 5 mm.

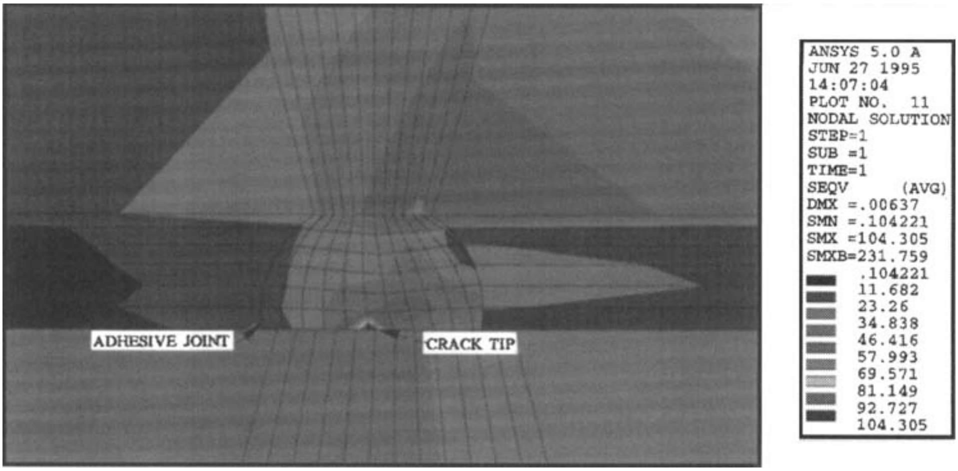

FIGURE 11 Close-up of Von Mises stresses in the middle plane ($X = 0$) for a crack length equal to 5 mm.

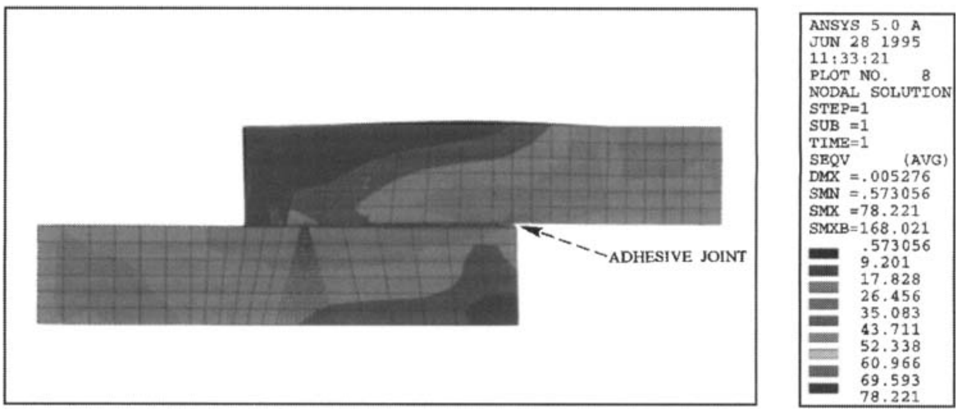

FIGURE 12 Von Mises stresses in the middle plane ($X = 0$) for a crack length equal to 2 mm.

case in the experimental test where these opening displacements are really not nil. But in all cases, the normal stresses σ_{yy} are not negligible in relation to the other stresses.

N.B.:

*The Erdogan criterion is a suitable criterion because it leads to a deviation angle which is independent of the crack length as is shown from the experimental results. In

*The Erdogan criterion is a local criterion. It must take into account the local state of stresses in the neighbourhood of the crack tip. It is the local state of stresses which leads to the deviation angle. The Maximal Normal Stress criterion is a global criterion and is not used for the determination of the deviaiton angle. It is used for the determination of the normal stress, σ_{yy}, which is not easy to estimate because it varies in the adhesive joint. Thus, an average value of σ_{yz} is easy to obtain and an experimental value of the deviation angle, α, leads to an *average* value of σ_{yy}.

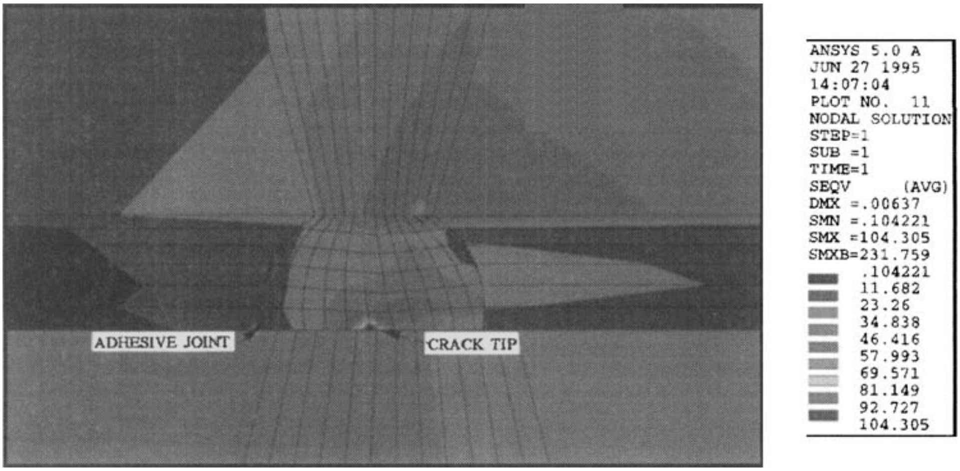

FIGURE 13 Close-up of Von Mises stresses in the middle plane ($X = 0$) for a crack length equal to 2 mm.

fact, the stresses $\sigma_{\alpha\alpha}$ and $\sigma_{r\alpha}$ are as follows:[18]

$$\begin{cases} \sigma_{\alpha\alpha} = -\dfrac{K_{\mathrm{II}}}{\sqrt{2\pi r}}\Bigg\{\exp[-\varepsilon(\pi-\alpha)]\times\left[\sin\left(\dfrac{\alpha}{2}+\varepsilon\log(r)\right)+\sin(\alpha)\cos\left(\dfrac{\alpha}{2}-\varepsilon\log(r)\right)\right. \\ \qquad \left.+\,2\varepsilon\sin(\alpha)\sin\left(\dfrac{\alpha}{2}-\varepsilon\log(r)\right)\right]+\exp[\varepsilon(\pi-\alpha)]\times\sin\left(\dfrac{3\alpha}{2}+\varepsilon\log(r)\right)\Bigg\} \\ \sigma_{r\alpha} = -\dfrac{K_{\mathrm{II}}}{\sqrt{2\pi r}}\Bigg\{\exp[-\varepsilon(\pi-\alpha)]\times\left[-\cos\left(\dfrac{\alpha}{2}+\varepsilon\log(r)\right)\right. \\ \qquad \left.-\,2\varepsilon\sin(\alpha)\cos\left(\dfrac{\alpha}{2}-\varepsilon\log(r)\right)+\sin(\alpha)\sin\left(\dfrac{\alpha}{2}-\varepsilon\log(r)\right)\right] \\ \qquad -\exp[\varepsilon(\pi-\alpha)]\times\cos\left(\dfrac{3\alpha}{2}+\varepsilon\log(r)\right)\Bigg\} \end{cases} \tag{4}$$

where ε is the bi-elastic constant as follows:

$$\varepsilon = \frac{1}{2\pi}\log\left[\frac{\dfrac{\kappa_1}{\mu_1}+\dfrac{1}{\mu_2}}{\dfrac{\kappa_2}{\mu_2}+\dfrac{1}{\mu_1}}\right] \tag{5}$$

where μ_i is the shear modulus of elasticity of material i ($i = 1, 2$), $\kappa_i = 3 - 4\,\nu_i$(ν_i is the Poisson's ratio of material i), K_{II} is the stress-intensity factor in Mode II which depends *on the crack length*, on the loading and on ε, r is the (small) distance from the crack tip.

Consequently we write:

$$\sigma_{\alpha\alpha} = \frac{K_{\mathrm{II}}}{\sqrt{2\pi r}}G(\alpha, r, \varepsilon) \quad \text{and} \quad \sigma_{r\alpha} = \frac{K_{\mathrm{II}}}{\sqrt{2\pi r}}H(\alpha, r, \varepsilon) \tag{6}$$

Where $G(\alpha, r, \varepsilon)$ and $H(\alpha, r, \varepsilon)$ are two functions *only* of α, r, and ε.

So, we have

$$\sigma_d = \begin{cases} \dfrac{K_{II}}{\sqrt{2\pi r}}[(G(\alpha,r,\varepsilon))^2 + (H(\alpha,r,\varepsilon))^2]^{(1/2)} & \text{if } \sigma_{\alpha\alpha} > 0 \\ \dfrac{K_{II}}{\sqrt{2\pi r}}H(\alpha,r,\varepsilon) & \text{if } \sigma_{\alpha\alpha} < 0 \end{cases} \tag{7}$$

We also can write:

$$\sigma_d = \begin{cases} \dfrac{K_{II}}{\sqrt{2\pi r}}F(\alpha,r,\varepsilon) & \text{if } \sigma_{\alpha\alpha} > 0 \\ \dfrac{K_{II}}{\sqrt{2\pi r}}H(\alpha,r,\varepsilon) & \text{if } \sigma_{\alpha\alpha} < 0 \end{cases} \tag{8}$$

To find the deviation angle which maximizes the stress, σ_d, amounts to finding the angle which annuls the derivative of $F(\alpha, r, \varepsilon)$ or of $H(\alpha, r, \varepsilon)$ which does not depend on the crack length.

CONCLUSION

A careful analysis of fracture surfaces can lead to very interesting information concerning the adhesion between substrate and adhesive on the one hand and on the mechanical behaviour of the bonded assembly during the test on the other hand.

The research of adhesive traces remaining on substrates enables one to know whether or not the adhesion is stronger than the cohesion of the adhesive. It has been shown that for simply degreased, rinsed and air dried silicon carbide adherends, failures are adhesive with a brittle behaviour.

In the case of a crack propagating from one interface to the other, a deviation angle was measured. It appears that this angle does not depend on the adhesive but on stress distributions in the assembly and, therefore, on the bonded assembly geometry. Moreover, the fact that the crack propagation is deflected for almost any lap length value seems to indicate that this location is hazardous and is probably due to small defects in the joint.

Good agreement was found when comparing the experimental angle with a calculated one, using a finite element model. The Erdogan criterion used in this paper is well adapted to the present case involving brittle adhesive failures.

The measurement of the deviation angle with the help of a profilometer is, therefore, a good means to verify rapidly the presence (or absence) of normal stresses during a shear test and allows one, in addition, to control their mathematical sign (compression or tension) and to estimate their average intensity with relatively good precision.

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