AN EXPERIMENTAL METHOD TO DETERMINE THE COMPLETE STRESS-DEFORMATION RELATION FOR A STRUCTURAL ADHESIVE LAYER LOADED IN SHEAR

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ABSTRACT
An experimental method to determine the complete stress-deformation relation for a thin adhesive layer loaded in shear is presented. Experiments are performed on an End-Notch Flexure (ENF) adhesive joint specimen. Balance of the energetic forces (J-integrals) of the applied load and of the adhesive at the crack tip, is used to calculate the stress-deformation relation from experimental data. The foundation of the method is an inverse solution based on beam theory. Quasi-static experiments are performed with a toughened epoxy adhesive using a servo-hydraulic testing machine. During an experiment the displacement of the loading point is increased with a constant velocity. The shape of the stress-deformation relation is found to be highly non-linear. An initial hardening behaviour is followed by a plateau and, finally, by a softening conclusion.

INTRODUCTION
Fracture of adhesive joints can accurately be predicted using a strength of materials approach based on the traction-deformation law of an adhesive layer, cf. [1-3]. Thus, in order to make reliable predictions of the strength of adhesively joined structures, the traction-deformation law for the adhesive layer need to be known. The traction-deformation relations of pure peel and pure shear are thereby of fundamental importance. Several experimental investigations of peel behaviour are found in the literature. Both direct and indirect methods are presented. In a direct measurement the idea is to subject the adhesive layer to a uniform stress distribution. A problem with this type of method is that it is virtually impossible to know in advance if the stress distribution actually is uniform. In fact, this requires that the test specimen is notch insensitive. An indirect method, on the other hand, often involves a non-uniform stress distribution. The idea is to measure the energetic force as a function of the deformation at a crack tip. The constitutive law is obtained by a subsequent differentiation, cf. [1-3]. The same technique is used in [4] to determine bridging laws of composites. In this paper an indirect method to measure the stress-deformation relation for an adhesive layer loaded in shear is presented. Experiments are performed on an ENF-specimen, cf. Fig. 1. During a test the instantaneous value of the energy release rate is recorded. An inverse method is used to obtain the constitutive law.
Fig. 1  Geometry of ENF-specimen. L = 1000 mm, W = width. Nominal adhesive thickness: t = 0.2 mm. Young’s modulus of adherends: E = 190 GPa. For numerical values, cf. Table 1.

THEORY

The shear stress distribution is non-uniform along the adhesive layer of the ENF-specimen. Hence, an inverse method is used to extract the constitutive relation, \( \tau = \tau(v) \), where \( v \) is the shear deformation. With a known \( \tau(v) \) - relation the behaviour of the ENF-specimen can be derived as the solution of the direct problem. In [5] Alfredsson derive an approximate solution to the inverse problem: Given the behaviour of the ENF-specimen, what is the \( \tau(v) \) - relation? In terms of energetic forces,

\[
J(\bar{v}) = \int_0^\bar{v} \tau(v) dv = \frac{9}{16} \frac{P^2 a^3}{E W^2 H^3} + 3 \frac{P \bar{v}}{8 W H} - \frac{9}{128} \frac{P^2}{k W^3 H^2} \tag{1}
\]

where \( \bar{v} \) is the shear deformation at the crack tip, \( E \) is Young’s modulus of the adherends and \( k = \tau'(0) \) is the initial stiffness of the adhesive layer. The other parameters are evident from Fig. 1. The first term on the right-hand side of Eq. (1) is the one ordinarily used in evaluations of the fracture energy, cf. [6]. This term is what remains if the flexibility of the adhesive layer is disregarded. Differentiation of Eq. (1) with respect to \( v \) yields

\[
\tau(v) = \frac{dJ(v)}{dv} \tag{2}
\]

In order to determine the energetic force and, subsequently, the constitutive relation; the load, \( P \), and the shear deformation at the crack tip, \( \bar{v} \), must thus be measured in a test. The initial stiffness, \( k \), is determined from data from the initial stage of an experiment using an analytical solution for a linear elastic adhesive, cf. [5]. Asymptotically for \( \bar{v} \to 0 \),

\[
\frac{P}{\bar{v}} = \frac{8 W H k}{3 \kappa a + 1} = c \quad \text{where} \quad \kappa = \sqrt{\frac{8k}{E H}} \tag{3a,b}
\]

In general, during a test, the relation \( P(\bar{v}) \) is non-linear. At the initial stage, however, \( P'(\bar{v}) \) is approximately equal to \( c = P'(0) \). The initial stiffness is thus solved from Eq. (3),
\[ k = \frac{9}{32} \frac{a^2}{EH^3} \left( \frac{c}{W} \right)^2 \left( 1 + \sqrt{1 + \zeta} \right)^2 \] where \[ \zeta = \frac{4}{3} \frac{EH^2 W}{a^2 c} \] (4a,b)

Thus, by adjusting a polynomial to the initial part of the \( P(\bar{v}) \) - curve, the initial slope, \( c \), and through Eq. (4), the initial stiffness, \( k \), are determined.

**SPECIMEN DESIGN**

The aim with the design of the specimen is to minimise its dimensions. In order not to violate the validity of Eqs. (1) and (3), however, the distance between the crack tip and the loading point must be large enough, cf. [5],

\[ \xi = \kappa (L/2 - a) \geq \xi_{\text{min}} \] (6)

where \( \xi_{\text{min}} = 10 \) for the present type of adhesive. This value is determined from numerical simulation of the experiments. Moreover, the crack length, \( a \), must be long enough to prevent instability. In [6] it is shown that, for the case of a rigid adhesive layer, the critical value of the crack length is \( a = 0.35L \). A more detailed analysis in [5], accounting for the flexibility of the adhesive layer, shows that the value predicted in [6] overestimates the critical crack length with about ten percent. In order to obtain a small specimen, still having a margin to instability, \( a = 0.35L \) is used here. Furthermore, Eqs. (1) and (3) are valid only as long as the adherends deform elastically. The adherends must thus be able to sustain the loading without reaching the yield strength of the material. In a simulation, the maximum force is always reached before the crack starts to propagate. However, the maximum force is approximately equal to the critical force prevailing at the start of crack growth, cf. [5]. The critical force, \( P_c \), is obtained by putting \( J = J_c \) and \( \bar{v} = v_c \) in Eq. (1) and solving for \( P = P_c \). The corresponding maximum bending stress in the specimen appears near the crack tip,

\[ \sigma_c = 2 \frac{EHJ_c}{\alpha H} \left( \sqrt{\gamma^2 + 1 - \gamma} \right) \] (7)

where

\[ \alpha = 1 - \frac{1}{8} \frac{EH}{ka^2}, \quad \gamma = \frac{1}{4} \sqrt{\frac{EH}{\alpha J_c} \frac{v_c}{a}} \] (8a,b)

With preliminary values of \( k, J_c \) and \( v_c \) (from test A, cf. Table 1), equations (6-8) are used to determine the dimensions \((H, L \text{ and } a)\) of the specimens. Here, the admissible value of the critical stress is set to 300 MPa. This value gives a margin to the yield strength of the material (500 MPa) in order to allow for a higher value of \( J_c \) in the following tests (B, C, D in Table 1). Table 1 gives the actual values of the length parameter, \( \xi \), and the maximum beam stress, \( \sigma_b \).
SPECIMEN PREPARATION

The adhesive, DOW Betamate XW1044-3, is a toughened epoxy used by the car manufacturer Volvo. The adherends are made of tool steel (Rigor Uddeholm). Prior to the joining the adherends are cleaned with heptane and acetone. In order to achieve the correct layer thickness, two steel wires with a diameter of 0.2 mm (nominal adhesive layer thickness) are glued on to one of the adherends. The steel wires are placed longitudinally in the interior of the adhesive layer; about 8 mm from the edges, 100 mm from the crack tip and 100 mm from the right end of the specimen. Numerical simulations indicate that the process zone, in front of the crack tip, is less than 100 mm. Thus, the wires do not interfere with the process zone. Moreover, the wires occupy less than 1% of the adhesive cross-section. The adherends and the adhesive are preheated to about 80 and 50°C, respectively. In the crack and at the right end of the specimen, PTFE-film having a thickness of 0.2 mm is positioned. The adhesive is applied and the adherends are fixed in position with clamps. The specimen is slowly heated to about 180°C, cured at this temperature during 30 minutes, and finally left to slowly cool in the furnace. Thus, residual stresses are minimised.

TEST PROCEDURE

The experiments are performed using a servo-hydraulic testing machine (MTS322). The displacement of the loading point is increased with a constant velocity of 1 mm/min. Both the force, \( P \), and the adhesive shear deformation, \( \dot{v} \), are measured during a test. The force is measured with a load cell located between the actuator and the hydraulic grip. The shear deformation is measured using an extensometer (MTS632.03F-30) attached to plates, which are fixed on each adherend, cf. Fig. 2. Two edges on the extensometer are pressed against the plates with springs and elastic bands to guarantee correct transmission of the shear deformation. During the tests, the extensometer measures the change in the distance between notches in the legs of the attachment plates. The resolution of the shear measurements is 0.05 \( \mu \)m. In order to minimize the friction between the adherends, the PTFE-film used during the joining process remains inside the initial crack.

EXPERIMENTAL RESULTS

Four different tests; A, B, C and D are performed. Geometrical data and experimentally obtained characteristic entities are given in Table 1. In Fig. 4 the energetic force, \( J \), is plotted as a function of the shear deformation at the crack tip, \( \dot{v} \). Fracture is assumed to occur at the maximum value of \( J \). It can be observed that, for tests B, C and D, \( J \) decreases beyond fracture, i.e. for shear deformation larger than the critical value. Thus, less energy is needed to fracture the adhesive layer once the crack has started to grow. For test A, the initial crack length is too small to obtain stable crack growth. Hence, for test A, catastrophic failure of the adhesive takes place before the critical shear deformation is reached. For the stable tests (B, C and D), the average values of the fracture energy and the critical shear deformation are, \( J_c = 3400 \ J/m^2 \), and \( \dot{v}_c = 0.180 \ mm \), respectively. In Fig. 3 the development of the shear deformation during the tests is shown. It is seen that the deformation rate varies considerably during the test; from about 0.1 \( \mu \)m/s at the start to about 10 \( \mu \)m/s at fracture. This is due to the fact that the loading point is given a constant velocity.
Evaluation of the constitutive law, cf. Eq. (2), involves differentiation. Numerical differentiation of the raw data inevitably leads to scatter in the $\tau(v)$ - curve. In order to avoid this, a series of functions are used to approximate the curve and then the series is differentiated. Here, a series used in [4] is choosen,

$$\frac{J(v)}{J_c} = \sum_{i=1}^{n} A_i \exp \left( -\frac{n v}{v_c} \right)$$

where the parameters $A_i$ are determined by a least squares fitting procedure. Twenty terms are used. Figure 5 shows that the resulting $\tau(v)$ - relations for tests A, B, C and D are fairly similar. The relations are highly non-linear. An initial hardening behaviour is followed by a plateau and, finally, by a softening conclusion. The peak value of the shear stress, $\tau_{\text{max}}$, is about 30 MPa. This should be compared to a maximum peel strength of about 20 MPa and a critical elongation of about 0.060 mm, obtained by similar measurements using a DCB-specimen, cf. [1,3].
DISCUSSION

It should be noted that the presence of the PTFE-film during the joining procedure, produces a blunt crack tip. No measures are taken to form a sharp crack. This is because the tests are performed to investigate the constitutive behaviour of the adhesive layer. The presence of a sharp crack would probably lead to a lower value of the fracture energy. This presumption is supported by the fact that the fracture energy decreases as the conditions at the crack tip are altered when the crack grows, cf. Fig. 4. Thus, with a sharp initial crack, the $\tau(v)$ – relation would be different than the ones shown in Fig. 5.

As mentioned previously, the rate of deformation varies considerably during the test, cf. Fig. 3. Strictly speaking, the $\tau(v)$ – relations obtained are valid only for this specific variation of the rate of deformation. In order to study a possible rate dependence of the $\tau(v)$ – relation, it is desirable to have a constant rate of deformation during a test. This will be done in future test series.

<table>
<thead>
<tr>
<th>Test</th>
<th>$H$ [mm]</th>
<th>$W$ [mm]</th>
<th>$a$ [mm]</th>
<th>$t$ [µm]</th>
<th>$v_c$ [µm]</th>
<th>$J_c$ [J/m$^2$]</th>
<th>$\tau_{\text{max}}$ [MPa]</th>
<th>$k$ [GN/m$^3$]</th>
<th>$\xi$ [-]</th>
<th>$\sigma_b$ [MPa]</th>
</tr>
</thead>
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<tr>
<td>A</td>
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<td>32.6</td>
<td>200</td>
<td>~200</td>
<td>164$^*$</td>
<td>3670$^*$</td>
<td>31</td>
<td>4300</td>
<td>22</td>
<td>230</td>
</tr>
<tr>
<td>B</td>
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<td>32.6</td>
<td>350</td>
<td>210</td>
<td>167</td>
<td>3420</td>
<td>32</td>
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</tr>
<tr>
<td>C</td>
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<td>32.8</td>
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<td>270</td>
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<td>3330</td>
<td>28</td>
<td>3200</td>
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</tr>
<tr>
<td>D</td>
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<td>32.8</td>
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<td>3470</td>
<td>30</td>
<td>4200</td>
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</tbody>
</table>

*Table 1 Geometrical data, cf. Fig 1, and experimentally obtained characteristic entities defined in the text. For the unstable test A, the critical shear deformation and the fracture energy indicate the values at failure.*

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REFERENCES


