

ON THE EXISTENCE OF A UNIQUE STRESS-DEFORMATION RELATION FOR AN ADHESIVE LAYER LOADED IN SHEAR

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ABSTRACT

An experimental method to determine the complete stress vs. deformation relation for a thin adhesive layer loaded in shear is presented. The experiments are performed by use of a classical specimen geometry, *i.e.* the end-notch flexure specimen, though the experiments are evaluated based on a novel inverse technique. With this technique, the instantaneous energy release rate is first evaluated by use of a theory for the specimen based on the Euler-Bernoulli beam theory. Effects of a flexible adhesive layer are considered in an approximate way. From the energy release rate, the stress-deformation relation is evaluated using an inverse method. In order for the theory to be valid, the adherends of the specimen are only allowed to deform elastically. Quasi-static experiments are performed using a servo-hydraulic testing machine. In the experiments, the displacement of the loading point is gradually increased to obtain a constant velocity of the shear deformation at the crack tip. Formation of micro-cracks and the propagation of a macro-crack are monitored during the experiments by use of a CCD-camera attached to a microscope. By varying the heights of the adherends, the size of the process zone in front of the crack tip changes from about 200 to 400 times the thickness of the adhesive layer. The results of the experiments give a fracture toughness of 2.5 kJ/m², a critical shear deformation of 0.13 mm, and a maximal strength of 30 MPa independent of the specimen geometry. The experiments show consistent results. The results show that if the process zone is large as compared to the thickness of the adhesive layer, the shear stress – shear deformation relation can be considered as a constitutive property of the adhesive layer.

1 INTRODUCTION

Adhesive joining is receiving increased interest during recent years due to its ability to join dissimilar materials. However, no generally accepted method for strength analysis of adhesive joints appears to exist. This is partly due to a lack of constitutive modelling of adhesive layers. Andersson and Stigh [1] have recently presented a method for the measurement of the stress-elongation relation for an adhesive layer loaded in peel. Complementing this work, Alfredsson *et al.* [2] present a method to measure the shear stress vs. shear deformation relation (τ - v -relation) for an adhesive layer. As compared to more straightforward methods, as tensile and torsion tests on butt joints, *cf. e.g.* Chai [3], the present methods do not require extremely stiff testing machines to capture a softening curve. In the present method, an end-notch flexure (ENF) specimen is used *cf.* Fig. 1. In an experiment, the displacement of the loading point, u , is gradually increased. The applied force, P , and the shear deformation, v_0 , at the start of the adhesive layer are measured continuously during the experiment. Alfredsson [4] shows that the area under the τ - v -curve, *i.e.* the J - v_0 -relation is given with high accuracy by

$$J(v_0) = \int_0^{v_0} \tau(\tilde{v}) d\tilde{v} \approx \frac{9}{16} \frac{P^2 a^2}{EW^2 H^3} + \frac{3}{8} \frac{Pv_0}{WH} - \frac{9}{128} \frac{P^2}{kW^2 H^2}, \quad (1)$$

where E is the Young's modulus of the adherends, W is the width and H the height of each adherend, and a is the initial crack length, *cf.* Fig. 1. Moreover, k is the initial stiffness of the adhesive layer. It may be noted that the first term on the right hand side corresponds to the energy release rate of the specimen modelled as two elastic beams connected by a rigid adhesive layer. Thus, the

two remaining terms corrects the expression by accounting for a flexible adhesive layer. The τ - v -relation is given by the inverse of eqn (1), *viz.*

$$\tau(v) = \frac{dJ(v)}{dv}. \quad (2)$$

The ENF-specimen is known to be conditionally stable. Estimates based on beam theory with elastic beams and a rigid adhesive layer, shows that a crack length, a , shorter than about $0.35L$ gives an unstable configuration under displacement control, *cf.* Carlsson *et al.* [5]. Alfredsson [4] gives improved estimates of the stability limit based on a theory considering the flexibility of an adhesive layer, *i.e.* the theory that yields eqn (1). For the present case, an 18 % shorter crack is allowed with $H = 32$ mm, *cf.* [4].

For eqn (1) to be valid, the adherends have to deform elastically throughout the experiment and the distance between the loading point and the crack tip ($L/2 - a$) has to be large enough, so that the shear deformation at the loading point is much smaller than v_0 . These conditions are carefully discussed in [4]. Some initial experiments are reported in [2]. A typical τ - v -curve consists of an initial elastic part where the τ increases proportionally with v . At about 30 MPa, the stress levels off. The stress subsequently decreases to zero at about 0.18 mm shear deformation.

In the present approach, we explicitly assume that the stress acting on the adherends is governed by the deformation of the adhesive layer through a “constitutive law” for the adhesive layer. Furthermore, we assume that this “law” is the same for all positions along the adhesive layer. In reality, the deformation process is non-uniform due to the non-homogeneity of the adhesive and the development of micro-cracks. Thus, we implicitly homogenize these non-uniformities and assume the existence of a unique “law” relating the homogenized stress τ with the homogenized deformation v . The purpose of the present paper is to examine if variations in the gradients along the adhesive layer influence the τ - v -relation. A new series of experiments is reported here. In this series, influences of variations in the height, H , of the specimen is studied. Moreover, all experiments in [2] were performed with a prescribed constant velocity of the loading point. This yields a variable rate of v_0 due to the non-linearity of the adhesive layer. In the present paper, the displacement of the loading point is prescribed to give a constant *shear rate* ($dv_0/dt = 1 \mu\text{m/s}$) during the experiments.

2 EXPERIMENTS

2.1 Specimen design

The adhesive, DOW Betamate XW1044-3, is a toughened epoxy. Earlier results [2] show a fracture toughness $J_c = 3.4 \text{ kJ/m}^2$. The adherends are made of tool steel (Rigor Uddeholm) with a yield strength $\sigma_y \approx 500 \text{ MPa}$ and Young’s modulus $E = 190 \text{ GPa}$.

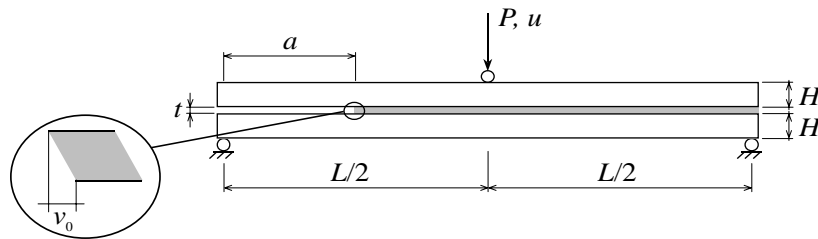


Figure 1. Geometry of ENF-specimen. $L = 1000$ mm, $W =$ width. Nominal adhesive thickness: $t = 0.2$ mm. For numerical values, *cf.* Table 1.

The conditions for the validity of eqn (1) described above have to be considered in the design of the specimens. Initial experiments indicate that $(L/2-a)$ must be larger than 150 mm, *cf.* [2] and [4]. Together with the stability condition we arrive at $L = 1000$ mm and $a = 350$ mm. The dimensions of the specimen have to be large enough to avoid plastic deformation of the adherends. Beam theory is used to estimate the maximum stress in the adherends. Here, it is assumed that the adhesive fractures at the maximum value of the force, $P = P_c$. Where P_c is calculated with eqn (1) using, $J = J_c$, and $v = v_c$. The maximum stress in the adherends at the crack tip, σ_b , is given by

$$\sigma_b = 2\sqrt{\frac{EJ_c}{\alpha H}} \left(\sqrt{\gamma^2 + 1} - \gamma \right), \quad (3)$$

where

$$\alpha = 1 - \frac{1}{8} \frac{EH}{ka^2}, \quad \gamma = \frac{1}{4} \sqrt{\frac{EH}{\alpha J_c}} \frac{v_c}{a}. \quad (4, 5)$$

Values of k , J_c and v_c are taken from the experiments reported in [2]. An analysis shows that the height, H , must be larger than 10 mm. The following values are chosen: 16, 25 and 32 mm.

2.2 Specimen preparation

The bonding surfaces of the adherends are cleaned with n-Heptane and rinsed in Acetone. After preheating the adhesive to about 60°C, it is applied to one of the adherends. Good alignment of the two adherends is secured with the use of a fixture. To achieve the correct adhesive thickness along the entire specimen, two steel wires with a diameter of 0.2 mm are glued on to one of the adherends. Numerical simulations indicate that the process zone, in front of the crack tip, is about 60 mm long. The steel wires start 100 mm from the crack tip, thus they do not interfere with the process zone. Moreover, they occupy only 1 % of the cross sectional area of the adhesive. At the crack tip a 0.2 mm thick Teflon-film is positioned. To ensure good contact between the adhesive and the adherends a small compressive force is applied to the specimens during the curing in a oven at 180°C for 30 min. After curing, the specimens are left in the oven to slowly cool to room temperature, thus, minimizing residual stresses in the adhesive layer.

2.3 Test procedure and experimental setup

The experiments are performed using a servo-hydraulic testing machine. The force, P , is measured with a load cell located between the actuator and the hydraulic grip. The displacement, u , is measured with a LVDT positioned under the loading point. The shear deformation, v_0 , is measured using an extensometer attached to two plates which are fixed on each adherend on one side of the specimen, *cf.* [2]. The crack tip region is filmed during the experiment using a CCD-camera attached to a microscope.

2.4 Evaluation and experimental results

Equation (1) gives the J - v_0 -curve for an experiment. The third term in eqn (1) contains the initial stiffness of the adhesive layer, k , which has to be estimated from the experiments before eqn (1) can be used. In [2], k is shown to be given by,

$$k = \frac{9a^2}{32EH^3} \left(\frac{c}{W} \right)^2 \left(1 + \sqrt{1 + \xi} \right)^2, \quad (6)$$

where

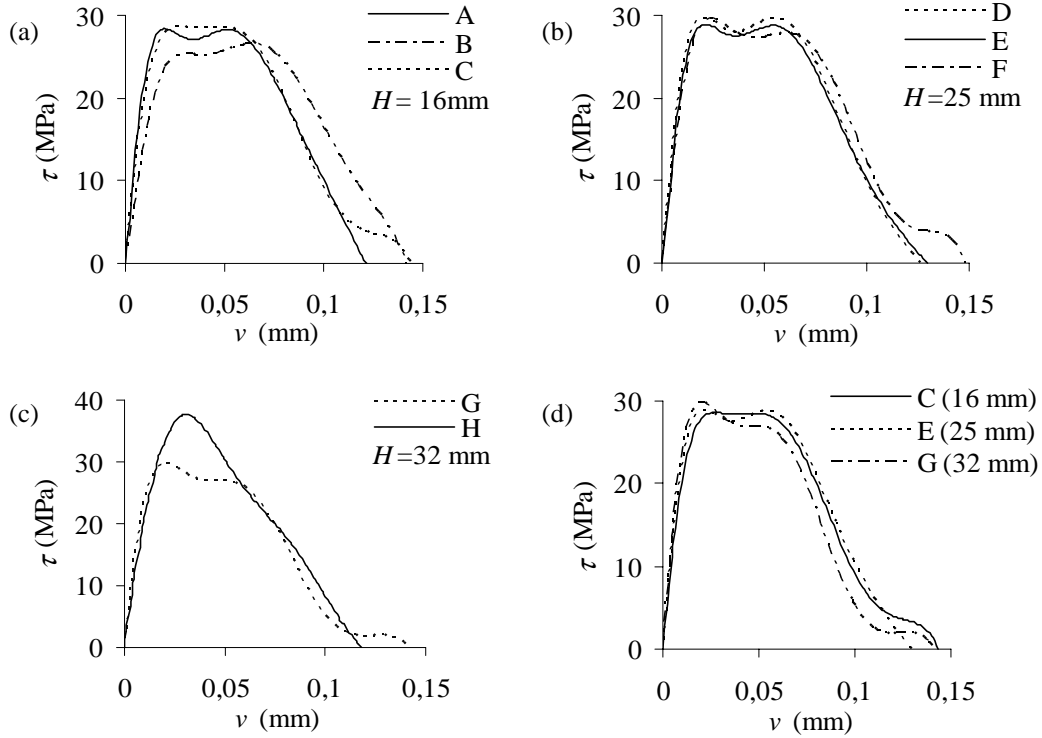


Figure 2. τ - v -curves for the eight ENF-specimens. Figures (a), (b), and (c) correspond to beam heights 16, 25 and 32 mm, respectively. Figure (d) shows a comparison of the results from different geometries.

$$\xi = \frac{4EH^2W}{3a^2c}, \quad (7)$$

and c is the slope of the initial linear part of the measured $P(v_0)$ curve. The evaluation of the J -integral involves differentiation of experimental data that leads to scatter in the $\tau(v)$ -curve. In order to minimise this, the J -curve is first approximated with a Prony-series

$$\frac{J(v_0)}{J_c} = \sum_{i=1}^{10} A_i \exp\left(-\frac{n}{i} \frac{v_0}{v_c}\right). \quad (8)$$

A least square fitting procedure is used to determine the parameters A_i . The series is then differentiated. The resulting $\tau(v)$ -relations for the eight specimens in this series are shown in Fig. 2. Geometry and characteristic data are given in Table 1. The results from two specimens differ somewhat from the other curves, *i.e.* specimens B and H. Good repeatability is obtained between the other six experiments. The average fracture toughness, J_c , is 2.49 ± 0.16 kJ/m². No systematic correlation between the height, H , and J_c is apparent. The average critical shear deformation, v_c , is 134 ± 11 μ m.

The fracture process is filmed through a microscope during the experiments. It is observed that the microscopic fracture process usually starts some distance from the PTFE insert. In all experiments the microscopic cracks are slanted, *cf.* Fig. 3. Contrary to the other results, *e.g.* [3], the cracks do not appear to open in the direction of the maximum normal stress in the

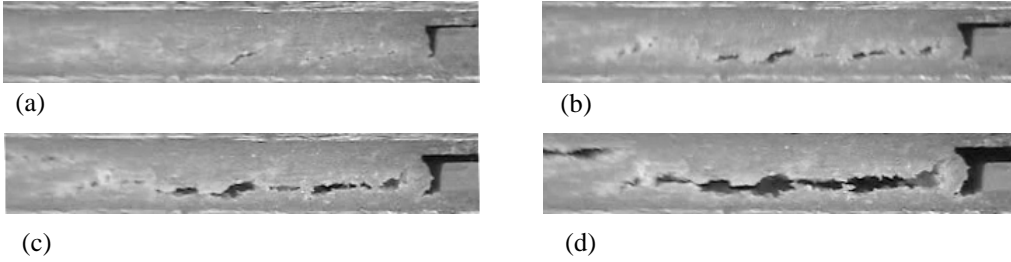


Figure 3. Crack propagation from experiment E at four different values of the shear deformation. (a) $v = v_c = 130 \mu\text{m}$, (b) $v = 178 \mu\text{m}$, (c) $v = 207 \mu\text{m}$, (d) $v = 285 \mu\text{m}$.

layer, *i.e.* 45° . It is tempting to assume that this is a result of an additional peel loading in the ENF-specimen. However, numerical analyses show that the peel stresses are virtually zero at the crack tip, *cf.* [6]. After initiating a number of slanted microscopic cracks, the cracks grow and coalescence to a long crack, *cf.* Fig. 3c.

Table 1: Geometrical data and experimental results. The value of the thickness of the adhesive layer, t , is given at the crack tip.

Test	H [mm]	t [μm]	W [mm]	J_c [kJ/m^2]	τ_{\max} [MPa]	v_c [μm]	p (mm)
A	16.7	200	32.5	2.41	28.3	121	45
B	16.7	230	32.5	2.59	26.3	141	49
C	16.7	220	32.5	2.47	28.6	144	54
D	25.6	210	32.5	2.52	29.5	126	58
E	25.6	210	32.7	2.47	28.8	130	60
F	25.6	200	32.6	2.65	28.9	148	69
G	32.6	200	32.5	2.30	28.4	141	86
H	32.6	190	32.5	2.50	37.6	118	57

The length of the process zone in front of the crack tip provides a measure of the gradient in the adhesive layer. A short process zone corresponds to a large gradient and *vice versa*. Alfredsson [4] provides estimates for the length of the process zone in the ENF-specimen using idealized τ - v -relations. Estimates of the critical length, p , of the process zone are given in Table 1. In the adjustment of the idealized model to the experiments, the parameters are determined to yield the same J_c , v_c and k as in the experiments. Table 1 shows that p varies between 45 and 86 mm. This corresponds to about 200 to 400 times the thickness of the adhesive layer. Since no influence is noticed on the τ - v -relations and the fracture energy, the stress – deformation relation appears to be a constitutive property of the adhesive layer if the process zone is large.

Inspections of the fracture surfaces of specimens B and H indicate that the weaker specimen, B, shows more interfacial cracking. Specimen H on the other hand shows a smoother fracture surface than the other experiments. None of the adherends show any sign of plastic deformation after the experiment.

3 DISCUSSION AND CONCLUSIONS

Strength analysis of adhesively joined structures based on an adhesive layer theory requires that the fine details of the stress and strain fields within the adhesive layer have a negligible influence on the stress-deformation relation for the adhesive layer. We have here showed that the shear

stress – shear deformation relation for an engineering adhesive (DOW Betamate XW1044-3) is unaffected by variations of the size of the process zone. However, the process zone is large in all experiments reported here. A method to achieve a shorter process zone is to allow the adherends to deform plastically. Equation (1) is then not applicable and other methods to deduce if the τ - v -curve is applicable have to be used. A shorter process zone can also be achieved using the alternative specimen in Alfredsson [7]. This specimen allows for an exact inverse method to measure the τ - v -curve.

The experiments presented here are performed with a constant rate of shear deformation at the crack tip. Thus, the adhesive layer experiences a nominally constant shear rate 0.005 s^{-1} at the crack tip. In the previously reported experiments, the displacement rate of the loading point was kept constant, *cf.* [2]. This gave a shear rate varying from about 0.0005 s^{-1} , at the start of an experiment, to about 0.05 s^{-1} at fracture. The τ - v -curves show some variation between the two sets of experiments. In [2], the critical shear deformation varies between 0.17 to 0.19 mm as compared to the results of the present paper, *i.e.* 0.12 to 0.15 mm. The fracture energy is also considerably smaller in the present set of experiments. In [2], J_c is about 3.4 kJ/m^2 which can be compared to 2.5 kJ/m^2 in the present investigation. However, the maximum stress is almost the same in the two sets of experiments. It is tempting to attribute these variations to the variation in shear rate. However, comparisons with a third set of experimental results indicate that it is more likely to be an effect of batch-to-batch variations of the adhesive. Similar variations are reported by Andersson and Stigh [1] for the same adhesive loaded in peel.

4 ACKNOWLEDGEMENT

The authors would like to thank our colleagues Mr. Svante Alfredsson and Mr. Anders Biel for helpful discussions during this work. We would also like to thank Mr. Bertil Enquist at Chalmers University of Technology for performing the experiments.

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