JOURNAL OF MATERIALS SCIENCE 20 (1985) 4069–4074

Measurement of crack propagation data in small slices of brittle materials in controlled bending

J. WILDE*, W. GROLL,‡, F. W. KLEINLEIN§
Institute for Materials Science, Department I, University Erlangen-Nürnberg, Martensstrasse 5, D–8520 Erlangen, West Germany

Measuring slow crack propagation data in small slices of brittle materials, elongations were cemented to both sides giving a four-point bend specimen. A method of introducing a definite sharp crack was employed to enable displacement-controlled fracture experiments to be performed. For the evaluation of the load–displacement plots of controlled fracture experiments an experimental compliance calibration was done for measuring crack length from compliance. The method to determine the curve of crack resistance against crack velocity was tested with window glass, giving good agreement between measurements on homogeneous and cemented specimens, if internal stresses from preparation are negligible.

1. Introduction

In testing the fracture behaviour of brittle materials, much smaller specimens can be used than for testing ductile ones. In some cases, however, the available dimensions of material (e.g. layer composite, single crystal) are yet too small for common testing specimens. From alternative methods the indentation test technique [1] only allows one to estimate the value of fracture toughness. Besides other shortcomings the relation between stress intensity factor K_I and crack velocity $\dot{a}$ cannot be measured till now. Another possibility is to enlarge the specimens, e.g. by cementing [2, 3], brazing or welding [4] between extensions to enable the use of standardized testing methods. However, for such heterogeneous specimens the compliance calibration is generally not known.

The aim of this work was to investigate the suitability of a cemented four-point bending specimen for measuring the crack propagation characteristics of brittle materials. First the dependence of the elastic compliance from crack length was evaluated experimentally. Then crack propagation tests in the model substance glass were performed with homogeneous and heterogeneous specimens. From both types of experiments the crack resistance against crack velocity relations were calculated using the appropriate compliance calibrations, and compared with each other.

2. Theory

The method to investigate the fracture characteristics of brittle materials by controlled crack propagation in four-point bend specimens at constant displacement rate was first analysed by Kleinlein [5, 6]. At small rates of crack propagation kinetic energies are negligible and the following relation holds:

$$G_I = \frac{P^2}{2BW}\frac{dC}{dx} = R \qquad (1)$$

G_I is the energy release rate, also called crack

*Present address: ISAF, TU Clausthal, Agricolastr. 2, D–3392 Clausthal–Zellerfeld, West Germany.
‡Fa. Degussa, FB Forschung Metall, PO Box 602, D–6540 Hanau/Wolfgang, West Germany.
§Fa. Kraftwerk Union AG, PO Box 3220, D–8520 Erlangen, West Germany.

0022–2461/85 $03.00 + .12

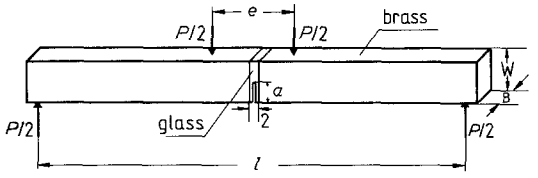

Figure 1 Cemented brass/glass/brass four-point bend specimen.

driving force, P is the applied load and R the crack resistance of the material. The compliance of a bend specimen, defined by

$$C = \frac{s}{P} \tag{2}$$

where s is the displacement under the load P, is dependent on the relative crack length $x = a/W$:

$$C(x) = C_0 + C_0 \delta Z(x) \tag{3}$$

According to the simple theory of bending the compliance C_0 of a homogeneous uncracked bend specimen is

$$C_0 = \frac{(l - e)^2 (l + 2e)}{4BW^3E} \tag{4}$$

where E is Young's Modulus of the material. (for l, e, b, W etc. see Fig. 1). The constant δ is given by

$$\delta = \frac{18 (1 - \nu^2)W}{l + 2e} \tag{5}$$

where ν is Poisson's ratio. The crack-length dependent function $Z(x)$ can be expressed by

$$Z(x) = \int_0^x Y^2 x' \, dx' \tag{6}$$

$Y(x)$ was calculated for both cases $x \leqslant 0.6$ [7] and $x \geqslant 0.5$ [8]. $Z(x)$ is a relative measure of the increase of the elastic compliance with crack length.

Since in a displacement-controlled bending experiment the crack velocity is not constant, it is very difficult to measure simultaneously and directly the crack length and load. However, the recorded load–displacement diagram allows one to calculate the actual compliance C_i for each pair of data (P_i, s_i) and the crack length a_i according to Equation 3, and further the instantaneous value of R_i from Equation 1. The crack propagation velocity $\dot{a}$ is calculated from the difference in crack length of two neighbouring points $(i + 1)$ and i, and the displacement rate of the testing machine. Here a correction for the compliance of the testing machine must be made [5].

In the case of a heterogeneous cemented specimen (Fig. 1) Equation 3 in no longer valid. It should be expected that the increase in compliance with crack length is dominated by the elastic behaviour of the material surrounding the crack.

3. Experimental details

3.1. Specimen preparation

The material tested was window glass made according to DIN 1249. Its elastic properties were measured dynamically: $E = 7.3\ 10^4$ MPa, $\nu = 0.23$. Both the homogeneous bend specimens and the layer chips for the cemented specimens were first cut from a slice of glass using a plastic-bonded diamond cutting disk, and subsequently precision-machined with a grinding machine using a plastic-cemented abrasive diamond wheel. The final dimensions of the homogeneous bend specimens were length $L = 112$ mm, $W = 10$ mm, $B = 7$ mm; the chips had a thickness $d = 2.0$ mm, $W = 9.9$ mm, $B = 6.9$ mm. The tolerances in B and W of all specimens were markedly below 0.01 mm.

The elongations consisting of brass were also precision-machined to the values of B and W of the glass slices, and cemented to them in a specially designed press which allowed the precise assembling of all parts. The cement was the rapid-hardening cyanoacrylate Agomet 280C (Degussa, Hanau, West Germany). An age-hardening of the glue was performed for 24 h at room temperature and alternatively at $T_h = 80°$ C. At the temperature of 80° C markedly higher strengths of the joints could be achieved.

For the compliance calibration, crack-simulating notches were cut in the plane of symmetry of both the glass slices and the homogeneous specimens with a high-speed circular saw using diamond cutting disks with 0.1 mm cutting width. Specimens for compliance calibration were provided with straight-through notches with lengths from 2 mm to 8 mm; specimens for controlled crack experiments were provided with chevron notches [5] with a base at $a_1 = 6$ mm. All notch lengths were measured by means of an optical microscope.

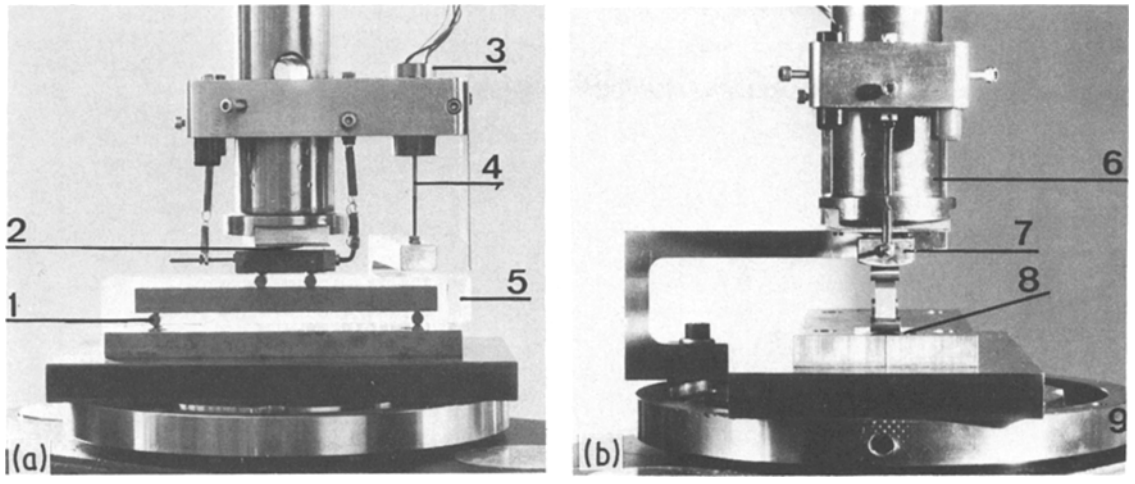

Figure 2 Four-point bend device. (a) Front view: (1) roller, (2) spherical segment, (3) displacement transducer, (4) transducer armature, (5) template for specimen adjustment (plexiglass). (b) Side view: (6) upper ram, (7) upper loading plate, (8) lower loading plate, (9) load cell of universal testing instrument.

3.2. Crack-propagation experiments.

The experiments were performed with a four-point bend device mounted on the 5 kN load cell of a universal testing machine (Instron Type 1114, see Fig. 2). All force-transmitting parts (spherical segment, loading plates, rollers) were made of hard metal to keep the device rigid. The contact plane of the upper and one of the lower loading plates had a cylindrical shape to avoid torsion momenta to the specimens.

The displacement s_{tot} between ram and lower loading plate was measured with an inductive displacement transducer and recorded together with load P on an X–Y recorder. The resulting curves were corrected with respect to the non-linear elastic characteristic of the bend device, which was measured previously with the method reported by Kleinlein [5].

In evaluating the compliance the cemented bend specimens were loaded up to about 70% of fracture load, estimated from K_{IC} data and notch length. The value of compliance was determined from the reciprocal slope of the corrected load–displacement curve.

For the controlled crack-propagation experiments all specimens were provided with a sharp crack, which was initiated at the tip and propagated to the base of the chevron notch using very small displacement rates. Arriving at the point according to this crack length in the load–displacement diagram, estimated from compliance and crack resistance values, the specimens were immediately unloaded by depressing the load cell and reversing the machine.

On the fracture surface of such specimens crack stop lines were found. Therefore extending this method to greater crack lengths a precise direct optical measurement of crack length was possible.

Finally, in the controlled-fracture experiment the specimens were loaded again at a cross-head speed v_T between 0.0833 and 0.833 mm sec^{-1}, resulting in a range of crack-propagation rates of about three decades. The environment of the crack was deionized water at room temperature. All P–s diagrams were digitalized and analysed numerically by a Fortran computer program according to the equations of Section 2 and those reported earlier [5].

4. Results and discussion

4.1. Compliance calibration

The compliance C_v of the joined brass/glass/brass specimens evaluated experimentally as a function of crack length is shown in Fig. 3. In analogy with results on homogeneous specimens the compliance is almost constant for small crack lengths, and increases strongly for high values of the relative crack length [9]. The progressive rise in $C_v(x)$ is accompanied by an increase in spread of the values which was attributed to two sources as follows. First the load range decreases with increasing crack lengths, resulting in greater values of relative error. In

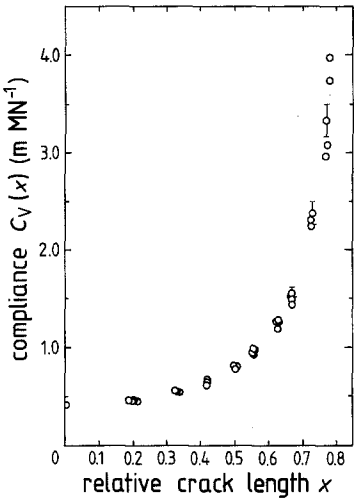

Figure 3 Compliance $C_v(x)$ of cemented brass/glass/brass four-point bend specimens plotted against relative crack length x.

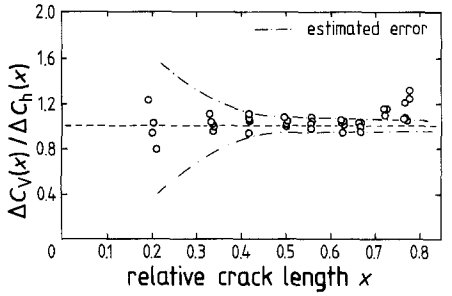

Figure 4 Ratio of increase in compliance $\Delta C_v(x)$ of heterogeneous brass/glass/brass specimens to increase in compliance $\Delta C_h(x)$ of homogeneous specimens, plotted against relative crack length x.

Fig. 3 the experimental error is shown by a bar which is smaller than the data symbols at small crack length. Secondly the nearly constant error in measuring crack lengths becomes more important with higher values of x because of the asymptotic behaviour of the compliance function $C(x)$. Fig. 4 shows, for various relative crack lengths, the ratio $\Delta C_v(x)/\Delta C_h$ of the measured compliance increase $\Delta C_v = C_v(x) - C_{v0}$ of the cemented specimen to the increase $\Delta C_h = \delta C_0 Z(x)$ of the homogeneous one.

Within the limits of accuracy this ratio is constant, symbolized by the broken line in Fig. 4. The systematic shift towards higher values could be caused by small damage in the region of the notch tip caused by the cutting process. This damage is again more effective at greater crack lengths. The few investigations of crack stop lines confirm this interpretation.

This result may be understood by a connection in series of slices perpendicular to the longer axis of the specimens. Since all slices except the crack-containing one remain unchanged, the increase of compliance with crack length is only affected by the elastic properties of the material containing the crack. Therefore the compliance of the cemented four-point bend specimen can be written in the form

$$C_v(x) = C_{v0} + \delta C_0 \int_0^x Y^2 x' \, dx' \qquad (7)$$

with the same functions $Y(x)$ and $Z(x)$ as in the homogeneous case. C_0 is the compliance of a virtual crack-free bend specimen consisting of the same material as the crack-containing one (here glass), having the same dimensions as the cemented uncracked specimen and being calculated by Equation 4. C_{v0} is the compliance of the crack-free cemented bend specimen. This value might also be calculated analytically, but because of the difficulties in evaluating thickness and elastic constants of the cement layer, measurement of C_{v0} should be preferred. Hence for analysing controlled crack-propagation experiments with heterogeneous bend specimens the same procedure applies as for homogeneous ones, except that the modified crack-free compliance must be taken into account.

4.2. Crack-propagation experiments on homogeneous and cemented specimens

For homogeneous specimens the results of two crack-propagation measurements are shown in Fig. 5a in a log $\dot{a}$–log R diagram, the straight lines representing a relation $\dot{a} \propto R^{n/2}$. Together with further results good reproducibility was obtained for the crack-propagation exponent n, which is also in good agreement with the value reported by Richter [10]. The difference in level of both curves may be due to local variations in chemical composition of the glass, especially in sodium content [10]. This interpretation is indirectly confirmed by the investigation of the crack stop lines, resulting in crack lengths differing by less than 1% from the values calculated from compliance; thus measuring should be accurate enough.

The results for the cemented specimens can be seen in Fig. 5b. However, due to the different hardening temperature T_h of the cement, two

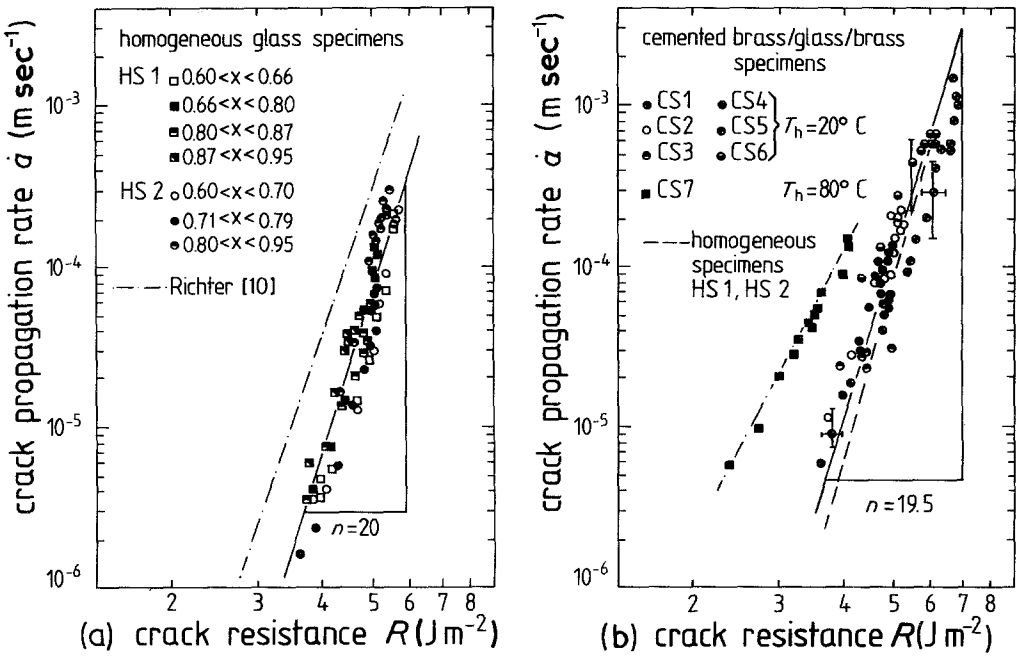

Figure 5 (a) Crack propagation rate $\dot{a}$ of window glass measured on homogeneous specimens, plotted against crack resistance R. (b) Crack propagation rate $\dot{a}$ of window glass measured on heterogeneous brass/glass/brass specimens, plotted against crack resistance R.

sets of straight lines were received, differing markedly in level and exponent of the equation

$$\frac{\dot{a}}{\dot{a}_0} = \left(\frac{R}{R_0}\right)^{n/2} \tag{8}$$

For $T_h = 20°\,C$, the exponent $n = 19.5$ is very similar to the one measured with the homogeneous specimens and the level of the log $\dot{a}$–log R curves is the same as for the homogeneous specimens, only the spread in data is slightly higher. Analysing crack stop lines indicates that the crack lengths can be calculated from load–displacement diagrams as precisely as in the homogeneous case.

For $T_h = 80°\,C$, the crack propagation exponent was evaluated as $n = 10$ which means only half the value of the former case. As temperature is the only obvious deviation in specimen preparation it must be responsible for the differences in crack-propagation characteristics. Because the thermal expansion coefficients of glass and brass are different, thermal stresses arise during cooling down from $T_h = 80°\,C$ to room temperature. Furthermore the hardening process of the cement itself may lead to residual stresses.

Two experimental observations confirm the existence of internal stresses:

(a) The curvature of the crack line is different in cemented, heat-hardened glass specimens and in homogeneous ones (Fig. 6). This may be understood if the loading stresses are superimposed on internal stresses [11].

(b) Inspection of the cemented specimens with polarized light using crossed polarization filters shows the existence of stresses in the notched specimens.

Internal stresses are combined with elastic

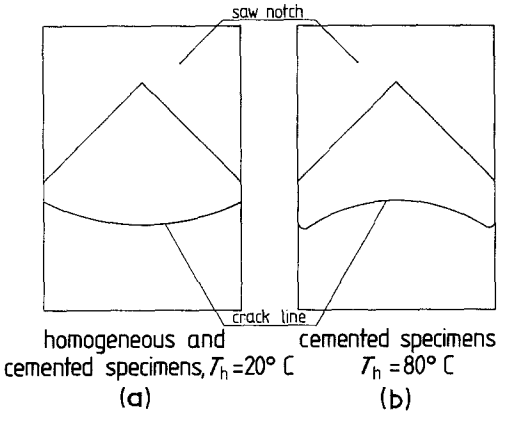

Figure 6 Curvature of crack lines during crack propagation: (a) stress-free homogeneous and cold-hardened specimens; (b) cemented specimens, heat-hardened at $T_h = 80°\,C$ containing thermal stresses.

deformation energy which may be released during crack propagation, and therefore will contribute to the total crack driving force. Hence the crack driving force G_a resulting in a certain value of crack velocity is smaller than for specimens without internal stresses. In the case of slow crack propagation this may be expressed in the following form:

$$R = G_{tot} = G_a + G_{th}$$

or

$$G_a = R - G_{th} \quad (9)$$

G_{tot} is the total elastic energy release rate, G_{th} is the portion according to internal stresses.

From Equation 9 it can be seen that internal stresses may result in reduction of apparent crack resistance: this is valid for internal stresses producing crack opening modes I, II or III. By Equations 8 and 9 one can further show the apparent crack propagation exponent n to be diminished [12].

No influence of internal stresses was seen comparing measured and computed crack lengths. It is evident because of the linear elastic behaviour, guaranteeing the superposition principle to hold.

5. Summary

It was shown that the increase of elastic compliance with crack length for the cemented four-point bend specimen is only affected by the elastic constants of the material surrounding the crack. The analytic description $C(x)$ valid for homogeneous specimens can be applied to the cemented specimen if a correction for the compliance of the crack-free specimen is made. In particular, the function Y is unchanged in the case investigated.

The applicability was proved by evaluating the crack-propagation behaviour of glass measured in controlled-bending experiments with either homogeneous glass or cemented brass/glass/brass specimens. If care is taken to avoid significant internal stresses during specimen preparation, crack propagation data are in agreement.

The method described herein makes it possible to investigate crack propagation behaviour with very small pieces of material.

References

1. A. G. EVANS, ASTM STP 678 (American Society for Testing and Materials, Philadelphia, 1979) pp. 112–135.
2. W. GROLL, F. W. KLEINLEIN and W. BLUM, *Z. Werkstofftech.* **14** (1983) 98.
3. J. WILDE, Diploma Thesis, Erlangen (1982).
4. E. KLAUSNITZER, *Schweissen und Schneiden* **26** (1974) 3.
5. F. W. KLEINLEIN, Doctoral Dissertation, Erlangen (1980).
6. F. W. KLEINLEIN and H. HÜBNER, Proceedings of the 4th International Conference on Fracture, Waterloo, Canada, June 1977, Vol. 3 (Chapman and Hall, London, 1977) p. 883.
7. W. F. BROWN and J. E. SRAWLEY, ASTM STP 410 (American Society for Testing and Materials, Philadelphia, 1966).
8. W. K. WILSON, *Eng. Fract. Mech.* **9** (1970) 168.
9. H. HÜBNER and H. SCHUHBAUER, *ibid.* **9** (1977) 403.
10. H. RICHTER, Doctoral Dissertation, Karlsruhe (1974).
11. U. SOLTESZ, H. RICHTER and E. SOMMER, "Advances in Fracture Research", edited by D. Francois *et al.* (Pergamon Press, Oxford, 1980) p. 2303.
12. W. GROLL, Doctoral Dissertation, Erlangen (1984).

Received 27 November
and accepted 19 December 1984