JOURNAL OF MATERIALS SCIENCE 23 (1988) 3970–3972

High resolution strain measurements by direct observation in the scanning electron microscope

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A technique for measuring strain with an accuracy of $\pm 100\,\mu\varepsilon$ by direct observation in the scanning electron microscope has been developed and tested. The technique involves placing a fine metallic mesh loosely on the surface of a tensile coupon and observing the motion of the surface with respect to this mesh. It was evaluated by tensile loading of aluminium and carbon fibre-reinforced epoxy specimens for strains up to $600\,\mu\varepsilon$ using an instrumented miniature loading stage. Applications and limitations of the technique have been discussed.

1. Introduction

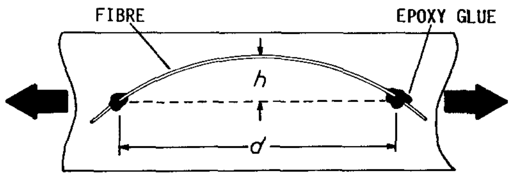
The use of minature loading stages in the scanning electron microscope (SEM) has become an important means of examining microscopic failure processes in many materials. At Cambridge University Engineering Department, loading stages for the SEM have been in use for some time [1–4]. Several workers have monitored the strain field in front of running cracks by observing the deformation of a regular array of points deposited on the sample surface by photolithography. This technique has been used to study transverse ply cracks in a glass-fibre composite [2], the deformation of particulate-filled composites [3], and to monitor the strain field and crack tip opening displacement in a brittle epoxy [4]. Elsewhere, the study of large strain deformations with this technique has been discussed by Obata *et al.* [5].

These observations have generally relied on the measurement of extremely small displacements on videotape or photographs and have been of limited use because of the lack of accuracy inherent in this approach. The general concept of strain measurement using surface patterns is well established and has been reviewed by Parks [6].

The precision of these techniques is dependent on the resolution of the display screen on which the image is presented. On a colour monitor with a horizontal resolution of 500 pixels, for example, the smallest increment of strain detectable would be 1 part in 500, or $2000\,\mu\varepsilon$. In practice, it would be difficult to achieve this, and thus the technique is unsuitable for measuring strains in high stiffness materials such as CFRP. Two approaches for improving strain measurements are proposed in this study.

2. Mechanical strain amplification

One method of achieving more accurate strain measurements involves the use of miniature levers and toggles to provide direct mechanical amplification. Fig. 1 illustrates the principle for tensile strain measurement, although it could equally be applied to the measurement of other components of strain. Using a set of fine tweezers under a binocular microscope (magnification $\simeq 20$), it was possible to manipulate fibres less than 2 mm long into the configuration illustrated. The advantage of this approach is that high resolution strain measurements can be taken from relatively low magnification images.



$$\varepsilon_{11} \simeq \frac{-\Delta h * h}{(d/2)^2}$$

Figure 1 Mechanical amplification of tensile strain with a toggle mechanism.

A more accurate, versatile and easily implemented approach is illustrated in Fig. 2. A fibre which is bonded to a sample at only one end will not deform during loading. The motion of the sample surface with respect to the unbonded end of the fibre may be viewed at very high magnification, and the "gauge length" of the test is the length of the fibre. The theoretical resolution of this technique is equal to that of the microscope divided by the length of the fibre.

The technique may be extended by replacing the fibre with a metallic mesh which rests on the surface of the sample (Fig. 3). This allows a variety of gauge lengths and directions to be selected simultaneously. In addition, because measurements are taken at both ends of the gauge length, the mesh need not be bonded to the surface at all. In practice, however, it is best to attach the mesh with a small spot of glue so that it does not move accidentally.

The mesh technique is related to the general approach of comparing a deformed surface pattern with a photograph of the undeformed pattern [6], but the use of the SEM allows the measurement of strain during the test itself. Although the measurements can be time consuming, the accuracy and versatility of the approach make it an ideal choice for detecting small

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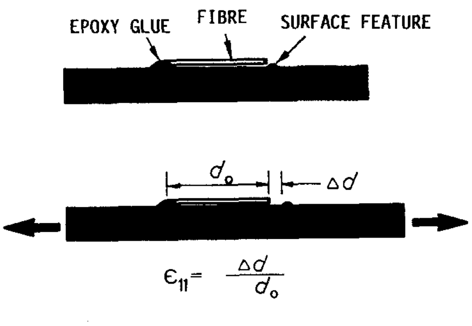

Figure 2 High magnification observation of the free end of a cantilevered fibre yields accurate strain measurements.

strains by direct observation. In the following sections the accuracy of the mesh technique is investigated, and a typical application is described.

3. Experimental procedure

The technique was tested using a Cambridge Instruments Stereoscan 200 SEM equipped with a digital noise reduction system described elsewhere [7, 8]. An instrumented miniature tensile rig capable of 5 kN loads was employed to deform the specimens [9]. Load and cross-head displacement were logged during the test with a BBC computer to an accuracy of ± 1.0%.

An aluminium alloy tensile coupon of known stiffness was used to check the accuracy of the technique at low strain. The edge of the coupon was coated with a dilute suspension of silver particles in a volatile solvent to provide surface detail. A copper mesh with a hole spacing of 65 μm was fixed to the edge of the coupon with a small amount of epoxy.

Silver particles of distinct shape were located in four mesh squares found on the same longitudinal axis at different distances from a reference square. The position of each particle within its square was measured at a magnification of about × 10 000 using the measuring capacity of the Stereoscan microscope (Fig. 4). The distance between each particle and the particle in the reference square was also measured prior to loading. After each loading increment, the same particles were located and their motion recorded. These measurements were converted into strains by subtracting the particle displacement in the reference square and then dividing the net displacement by the distance between the particles (Fig. 3). In this way strain readings were taken with "gauge lengths" of 65, 194, 741 and 1710 μm. The test was discontinued at a strain of about 2000 με.

A carbon fibre-reinforced epoxy (CFRP) sample was used to illustrate a typical application of the mesh technique. A four-ply unidirectional sample of CFRP, 10 mm wide, was double-edge notched with square notches 2.5 mm deep and 2 mm wide, giving a value of $2a/w$ of 0.5. A 2 mm × 2 mm mesh was placed at the base of the square notch and held in place with a small amount of epoxy. Two parallel sets of particles were monitored giving two separate "gauges", each with a gauge length of 894 μm.

4. Results

Fig. 4 illustrates the details of the measurement procedure. The silver particles have an identifiable shape and sharply defined edges. It would be possible to deposit an object grid or pattern on the sample's surface [3], but the deposition of silver from suspension is a simple and adequate alternative. The photograph (Fig. 4) was taken at a magnification of × 1000, but in practice, magnifications between × 7000 and × 12 000 were used when measuring particle displacements.

The results of the test on the aluminium coupon are shown in Fig. 5. The solid line represents the known stiffness of the sample as measured on an Instron 6025 load frame. The reading errors listed are calculated by dividing the uncertainty in locating the particles (typically ± 0.1 μm) by the distance between them (the "gauge length"). Increases in magnification did not reduce these errors as the particle boundaries became less distinct.

As expected, the longer gauge lengths produced more accurate results. Systematic errors probably resulted from inaccuracy in the reference readings at zero load. The effect of errors such as these would become less important at higher strains. The results were bounded by the calculated reading error, although careful measurement was required to achieve this. At each load level, it took approximately one minute to obtain the readings necessary to calculate a single value of strain.

Fig. 6 gives the strain in the ligament of the square-notched unidirectional CFRP sample. The fact that independent strain readings were nearly identical throughout the test is an indication that the notch tip strain was measured accurately.

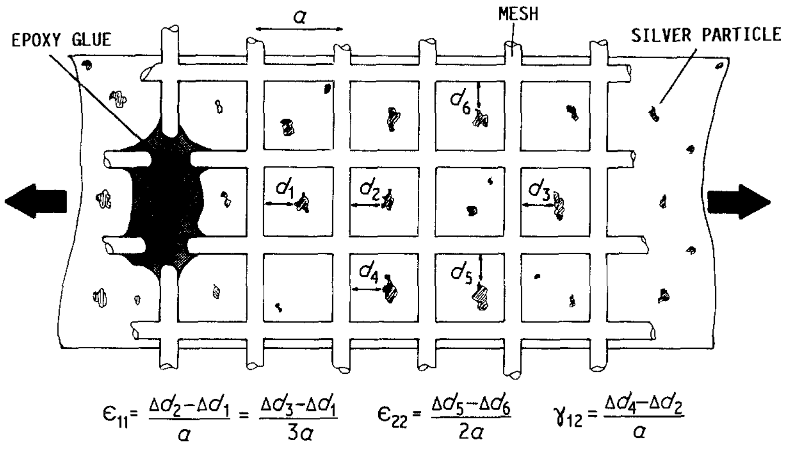

Figure 3 A metallic mesh which rests on the surface of the sample allows all components of strain to be measured accurately. The strain should be calculated using the actual distance between particles, but this is approximately equal to the distance between the relevent mesh squares.

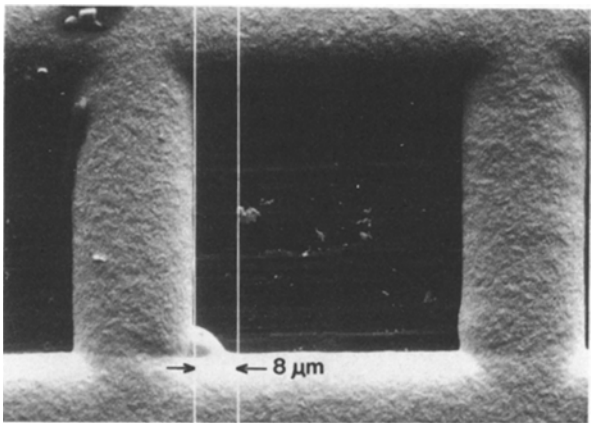

Figure 4 Photograph of typical measurement of particle displacement. Line separation is 8.00 μm. In practice, measurements were taken at about ten times this magnification.

During the test the sample split parallel to the fibres in a fashion typical of 0° laminates. The strain in the ligament was reduced by this damage, even though the load increased monotonically. The crack blunting effect has thus been measured quantitatively in the critical fibres of the ligament. It would be possible to measure the strain at the base of a square notch in each ply of a multidirectional laminate. This is an example of a measurement which would be unobtainable by normal means.

5. Conclusions

A means of using a mesh to measure small strains by direct observation in the scanning electron microscope has been described. Although the mesh technique is a labour-intensive process, it allows the user to measure a few critical strains during a dynamic SEM test with much greater accuracy than has been available previously. It is particularly useful for measuring strains in inaccessible locations, or where gradients are present, because gauge lengths as small as 100 μm can be used, depending on the accuracy required.

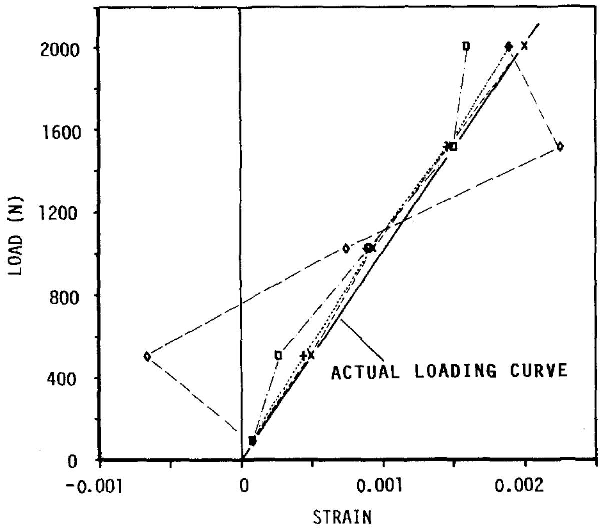

Figure 5 Strain plotted against applied load for aluminium calibration sample. The accuracy of the technique depends on the gauge length chosen and is well represented by the calculated reading errors.
Gauge length (μm): (—◇—) 65 ± 0.003;
(—·—□) 194 ± 0.001;
(--×--) 741 ± 0.0003;
(··+··) 1710 ± 0.0001.

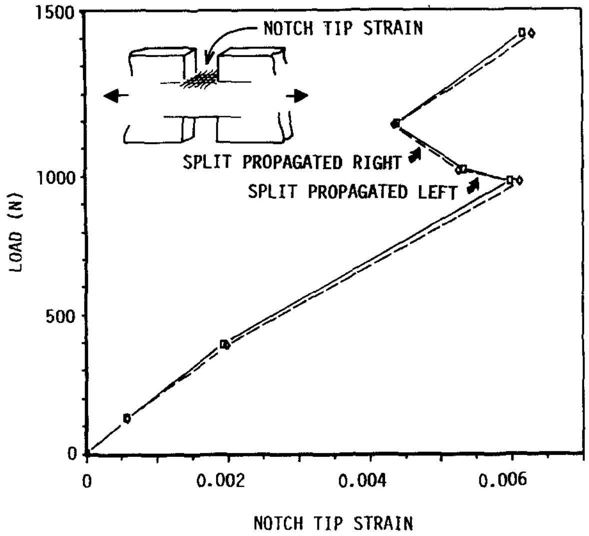

Figure 6 Strain at the root of a notch in a $(0)_4$ double-edge notched CFRP tensile coupon. The load increased monotonically but the strain was reduced by splitting parallel to the fibres. Gauge no: (--◇--) 1, (—□—) 2.

Acknowledgements

The author thanks Mr B. C. Breton, Dr P. W. R. Beaumont and Mr S. M. Spearing for guidance and assistance during the course of this work. The author also thanks the British Council for financial support. This work forms part of a larger study on the dynamic fracture of materials using *in situ* SEM in the Composite Materials Group of the Cambridge University Engineering Department.

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Received 17 September 1987
and accepted 19 January 1988