JOURNAL OF MATERIALS SCIENCE 5 (1970) 873-880

A Bend-testing Stage for the Scanning Electron-Microscope

D. R. CLARKE*
Division of Inorganic & Metallic Structures, National Physical Laboratory, Teddington, Middx, UK

P. R. BREAKWELL
Division of Materials Applications, National Physical Laboratory, Teddington, Middx, UK

G. D. SIMS
Division of Inorganic and Metallic Structure, National Physical Laboratory, Teddington, Middx, UK

The design of a stage to bend materials in a scanning electron-microscope (Stereoscan, Cambridge Scientific Instruments Ltd) is described together with examples of its use in the fracture of composite materials. The bend module†, when fitted to a large modular stage, can be used with existing scanning electron microscopes and is capable of bending, under increasing load or cyclic loading conditions, rectangular specimens of a maximum size 44 × 4 × 1.5 mm, to a maximum strain of 1.5%. Using the module with a standard display system the non-catastrophic stages of deformation and failure can be followed at higher magnification than has previously been possible. When the module is used in conjunction with a fast scanning and display system, deformation and fracture processes may be recorded at high magnifications while the specimen is being strained.

1. Introduction

The scanning electron-microscope has been used extensively in the study of materials both before and after service use or testing. The versatility of the scanning electron-microscope can be seen by studying the proceedings of the annual symposia held at the Illinois Institute of Technology Research Institute [1, 2].

A small number of mechanical testing devices have been developed [3, 4] to observe deformation *in situ*. The small size of the specimen that can be used has limited these devices to specialised material such as filaments and thin strips; this limit is partly because these devices have been made to fit, or have been adaptations of the standard specimen stage of the microscope (This stage is now being manufactured by Stereoscan, Cambridge Scientific Instruments, Ltd).

A further limitation has been that the relatively slow rate of picture formation has not always allowed the significant events to be observed and recorded (cf. ref. [3], fig. 8).

The bending module described in this paper is not so severely limited as previous stages. It is capable of testing a wide range of materials, using specimens of a more realistic size (44 × 4 × 1.5 mm) and has been designed to fit the larger modular stage recently introduced by the manufacturers (Cambridge Scientific Instruments Ltd). The stage can be used either with the instrument's normal scanning and display system, or with a fast scanning and display system developed at the National Physical Laboratory [5].

2. Design Consideration

The basic design requirement was for a module that would bend a test specimen in the scanning electron-microscope under increasing load or under cyclic loading conditions, while simultaneously allowing the deformation and fracture processes to be observed.

In order that the fullest information about the failure processes of the material may be obtained

*At present on leave of absence at Cavendish Laboratory, Cambridge.
†Patent applied for.

it was necessary for all the upper faces of the specimen to be observable. This means that the specimen must be given four degrees of freedom of motion. By designing the module to fit the manufacturer's large modular stage two degrees of freedom (the translational degrees of freedom at right angles to the electron beam) were already provided.

In an homogeneous material failure will start at the point of maximum strain, which in the case of three-point bending is above the central pivot on the face of the specimen in tension (fig. 1). For inhomogeneous materials failure can be assured at this point by suitable specimen preparation (e.g. notching). To use the maximum capability of the scanning electron-microscope it is necessary to observe fracture at high magnifications. This necessity, together with the above point on initiation of failure, requires that the central point of the tensile face of the specimen should not move during straining of the specimen. Thus the other two degrees of freedom must be rotational about the central point of the specimen's tensile face. The two chosen rotations were a circular motion (tilt) in the plane containing the electron beam and the specimen/collector direction, and a rotation in the specimen plane (fig. 2).

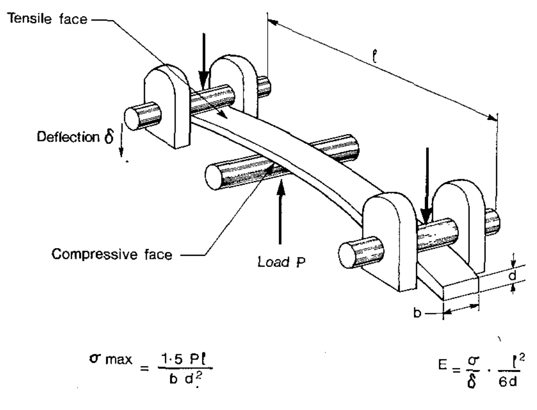

Figure 1 Three-point bend test.

Although a tilt angle, defined as the angle between the electron beam direction and the normal to the specimen plane, ranging from 0 to 90 degrees is ideally required, work on the scanning electron-microscope has shown that little is gained by increasing the angle above 60 degrees. So that every part of the fracture surface may be viewed there must be full rota-

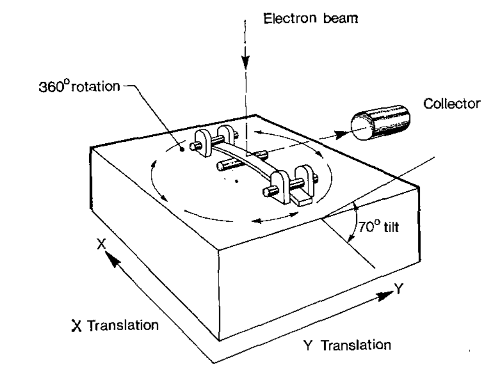

Figure 2 Tilting, rotating and translating freedoms of bend module.

tion capability of 360 degrees in the specimen plane.

The performance of the instrument improves with a decrease in the distances between the bottom of the final lens and the specimen. Unfortunately the larger the specimen used the larger the distance required for tilting and rotation without hitting the final lens. The largest desirable distance between the final lens and the specimen is 20 mm and this was chosen as the working distance in order to maximise the specimen size. The above requirements so far limit the specimen size to a length of approximately 45 mm.

The bend module was to be used primarily in the examination of composite materials and past experience had shown that attention to the span/thickness (l/d) ratio is very important in analysing the failure. If a large ratio is used (i.e. more than 20) the role of shear in the failure can be neglected, but equally if the specimen is too thin the test specimen may be unrepresentative through having too few fibres in the depth of the specimen. Thus a thickness of 1.5 mm was considered to be suitable for the reinforcing fibres currently available. An end deflection of 3 mm was chosen so that the tensile strain would be 1.5%, which would be adequate to initiate the early failure processes of most brittle materials.

Ideally, deflection sensitivity, defined as the smallest reproducible increment in end deflection, is required that would cause a change in linear dimensions of the specimen surface of the order of the resolution of the microscope. As this would be almost impossible to achieve and strain measurements in normal bend tests do not achieve an accuracy of more than a few per cent, an increment of deflection of a tenth of a per cent was thought to be reasonable.

The drive of the bending mechanism was made capable of either manual or motorised operation. The drive is suitable for a mechanical drive from a geared motor outside the vacuum system, for constant straining or cyclic straining experiments.

Apart from the obvious requirement that the module should operate with a minimum of backlash in the vacuum conditions of the microscope it is also important that the module should be robust enough to withstand shock-loading if a specimen breaks and that there should be no observable vibration of the stage, especially at high magnifications.

The design has allowance for a shield to prevent pieces of the specimen entering the electron microscope column. The shield has a small hole in it to allow the electron beam to reach the specimen.

3. Detailed Construction

The bend module is shown mounted on the manufacturer's large modular stage in figs. 3 to 5 and an exploded view is shown in fig. 6.

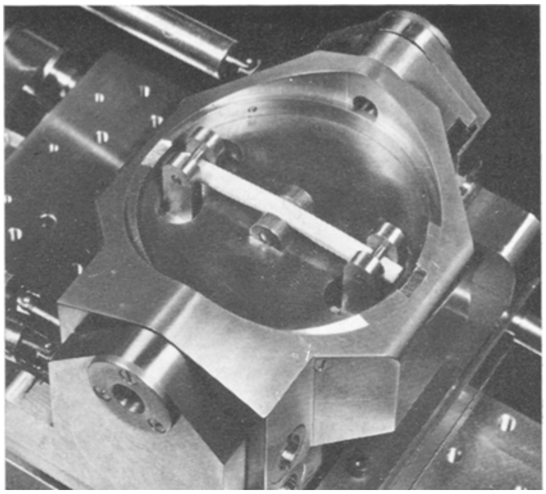

Figure 3 Bend module showing position of specimen ($\frac{3}{4}$ full size).

The specimen is mounted as shown in figs. 3 to 6, that is, above the central pin A and under the two end pins D. The specimen is bent by the end pins moving downwards parallel to the axis BB′ by the yoke C; the central pin remaining

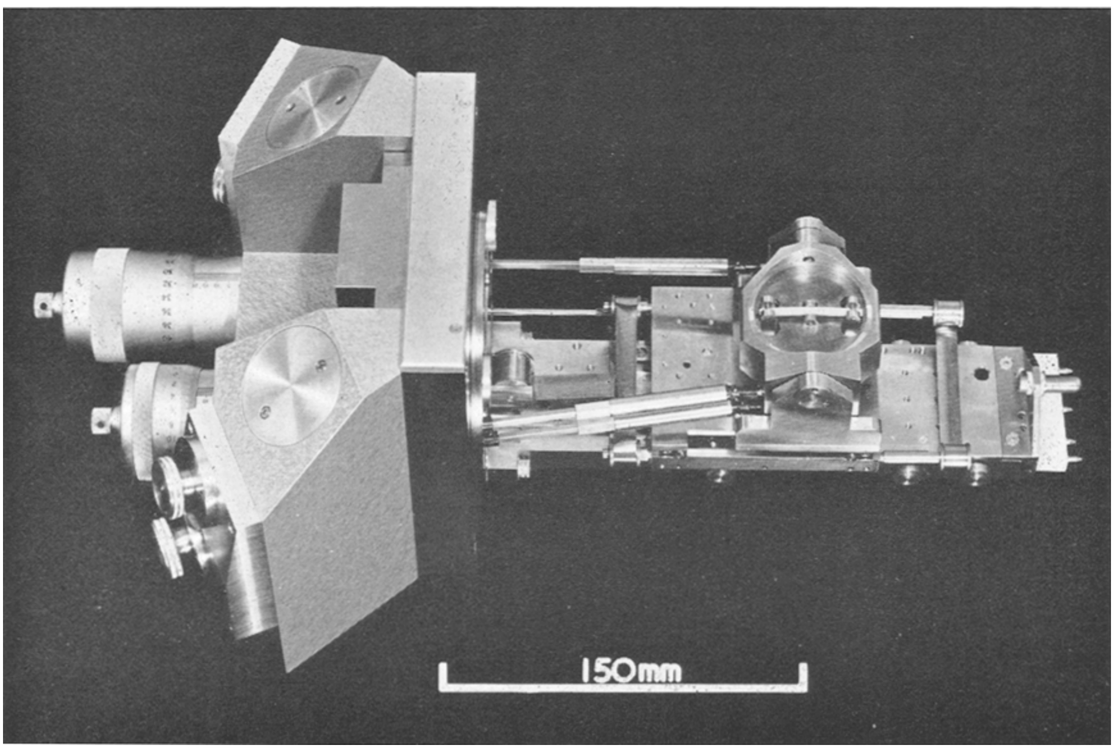

Figure 4 Bend module fitted to large modular stage showing drive controls and connections.

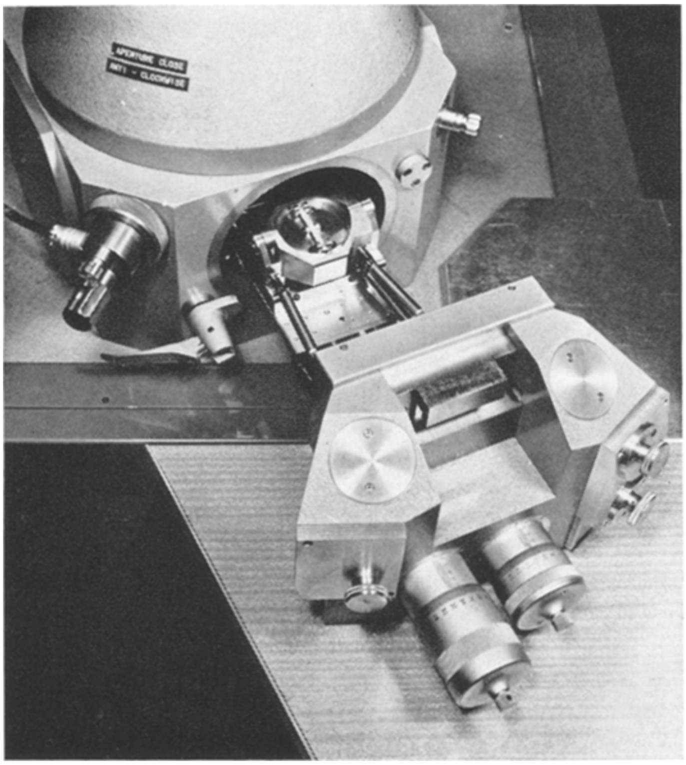

Figure 5 Modular stage positioned at the foot of microscope column.

stationary. The pin E at the end of the yoke C holds the cam follower F to the face cam G, against the spring H. The cam follower is constrained to move only in the direction BB′ by the guide I. The face cam G is bolted to the worm wheel J and the worm gear K turns the worm wheel J and the cam G, thus moving the ends of the specimen in the direction B→B′ to bend the specimen.

The whole of the bending assembly A to J can be rotated about the axis BB′, which passes through the centre point of the specimen, by turning the worm M because the central pin is an integral part of the wheel L. The yoke C rotates with the worm wheel L and bears on the PTFE washer N, which in turn bears on the recessed face of the assembly O.

The whole assembly including the worm wheel sector R is mounted on the pivots P from the supports Q, and it can be tilted by the action of the worm S on the wheel sector R. This enables the assembly A to J to be tilted with respect to the electron beam about an axis PP which passes through the centre point of the upper face of the specimen.

The worm drives are connected by telescopic rods fitted with universal couplings to the controls on the outside of the large modular stage (figs. 3 to 5). Although only the manual controls are shown in the figure a motorised drive can be used to give constant straining rates.

The very large ratio of the worm wheel J to the worm K ensures that large loads can be applied to the specimen for small input torques on worm wheel K. This large ratio also enables very small increments of deflection to be applied to the specimen ends. When manually operated a deflection of 0.1% of the total deflection can be reproducibly applied and with the geared motor even smaller deflections can be applied. One turn of the outside drive control represents a movement of 0.075 mm of the end pivots.

The stage is robust enough for a load of approximately 100 kg to be applied.

4. Experimental Results

A series of experiments were conducted to check the operation of the bend module in the scanning

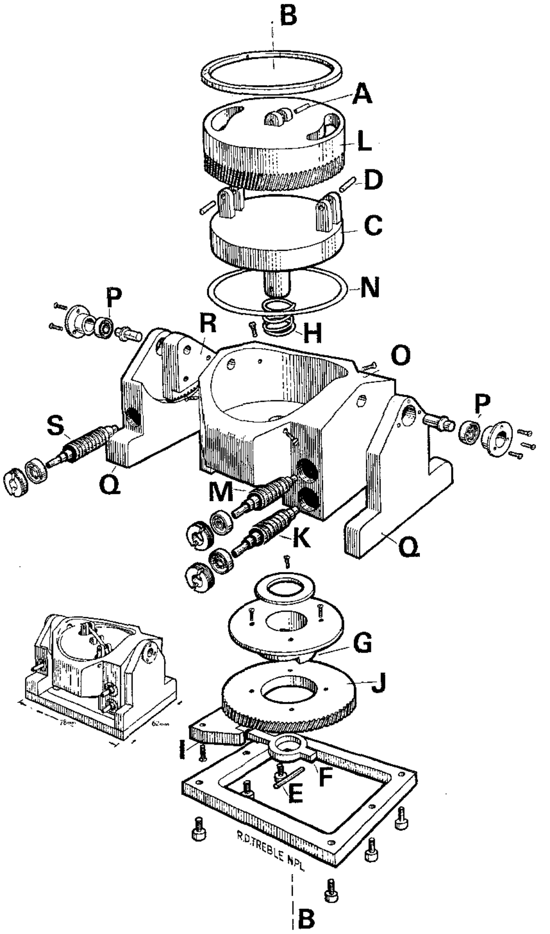

Figure 6 Exploded view showing construction of the bend module.

electron-microscope, in particular to check that all the controls operated without observable back-lash and that no vibration of the module was observable at high magnification. The materials used in these experiments were chosen so that the capabilities of the module could be tested.

4.1. Materials

Several examples of composite materials – carbon fibre reinforced resins, soft and hard woods, reinforced ceramics, and an intermetallic compound – were used. Most of the experiments were with carbon-fibre-reinforced plastics because the small diameter of the fibre (8 μm) is very suitable for high magnification work and also because a range of failure mechanisms can be obtained by varying the surface treatment of the fibre.

Two types of carbon-fibre-reinforced plastic were fabricated using a "leaky mould" technique, from surface-treated and untreated carbon fibres. The plastic used was an unplasticised epoxy resin (CIBA Ltd. 100 parts MY 750 and 10 parts HY 951 hardener). The reinforced resin specimens were coated by vacuum evaporation with a thin layer of gold-palladium to reduce the charging of the material under the electron beam. The hard and soft woods used were Makoré and Obeché, respectively. They were not coated but left to soak overnight in a 10% solution of an antistatic preparation in ethyl alcohol.

All the specimens were notched prior to testing so that the region where failure initiated was known and could be observed at high magnification. The specimens were V-notched either across the top tensile face at right angles to the tensile load or across an edge at right angles to the tensile face; in all cases the notch was above the centre pivot.

4.2. Results

The majority of the experiments were recorded on video tape and/or ciné film so that the fracture processes and the behaviour of the module could be subsequently examined in detail. The illustrations used in this report are "still photographs" taken of other specimens.

The end of a sharpened notch in a specimen, fabricated from untreated carbon fibres, before final failure is shown in fig. 7; the area to the lower left hand corner is one face of the V-notch. On straining the specimen, the crack, instead of propagating at right angles to the applied tensile stress, delaminated the material at the root of the notch by propagating along the fibre direction. The root of the notch, including the last carbon fibre cut during notching, and the delamination can be clearly seen in fig. 8. On further straining the crack ran parallel with the fibres until final failure by gross delamination of the specimen occurred with long fibre pull-out lengths (fig. 9). Such delamination behaviour illustrates the necessity of being able to view any part of the specimen, because although the initial fracture occurred at a predetermined point (the notch), final failure was not confined to this region.

The capability of following the fracture path in detail at high magnifications is shown in fig. 10.

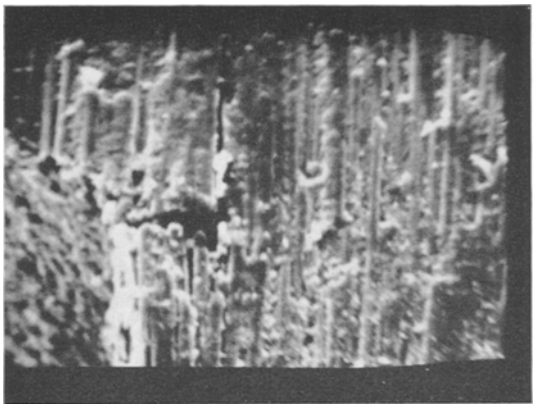

Figure 7 Delamination at the root of a notch in an untreated carbon fibre reinforced epoxy resin (× 250).

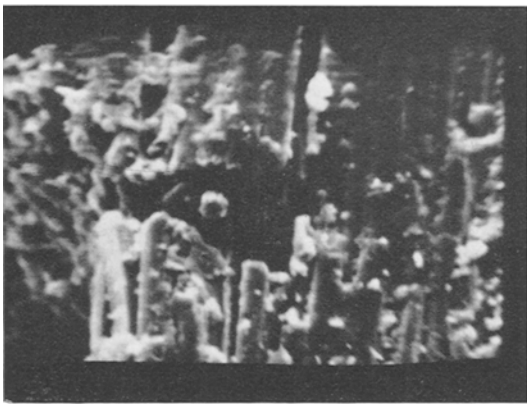

Figure 8 Higher magnification view of root of notch in fig. 7 (× 500).

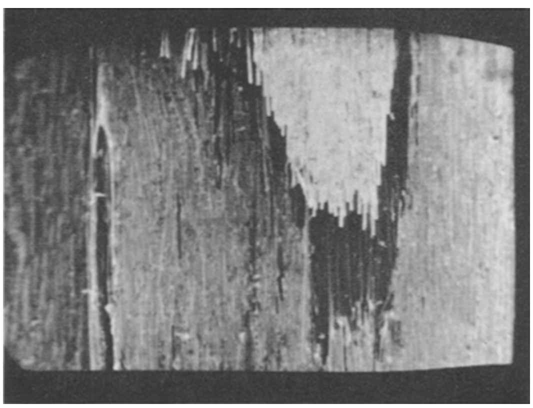

Figure 9 Failure delaminations in an area remote from notch (× 70).

Two carbon fibres lying in the tensile face of the specimen are clearly seen to be cracked and the fracture path, in the matrix, between them can just be seen. The matrix cracks were more easily seen with a different orientation of the specimen. When the specimen was strained further it was possible to examine, at this magnification, the failure processes occurring by crack propagation through fibres and matrix.

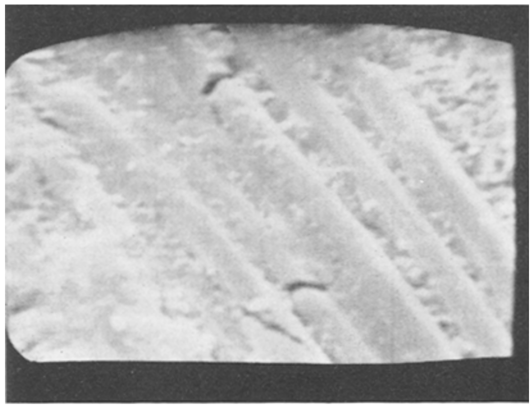

Figure 10 View of cracked fibres and connecting matrix crack (× 1000).

In contrast to this delamination type of failure, fig. 11 shows the failure crack in a surface-treated carbon-fibre reinforced epoxy specimen. The surface-treatment of the carbon fibres increases the interfacial shear strength and the fracture behaviour is consequently more brittle. There is no delamination of the specimen and any fibres not failing in the matrix fracture plane can be seen to have failed with very short pull-out lengths. Fig. 12 shows the specimen after further straining and fig. 13 is a view looking into the crack at one of the fracture faces. It is possible to observe regions such as these, which are uncoated, because they are close to the earthed metal coating which provides a leakage path for any charge produced.

Both types of wood gave similar results to the carbon-fibre reinforced epoxy specimens. In general the hard wood was slightly more brittle in behaviour but both types showed considerable delamination and pull-out. Poor contrast, partly due to an insufficiently smooth surface, hindered more detailed examination of the fracture process.

During these experiments there was no

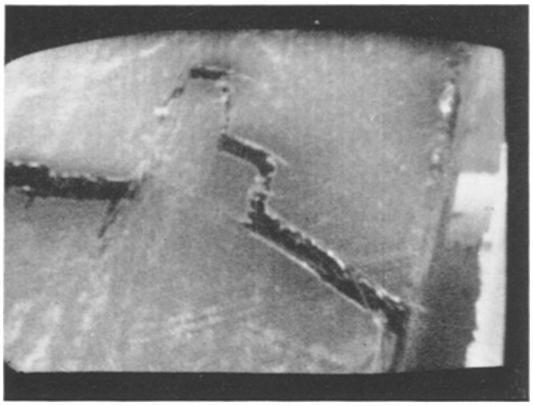

Figure 11 Brittle-type fracture in a treated carbon-fibre reinforced epoxy resin (× 70).

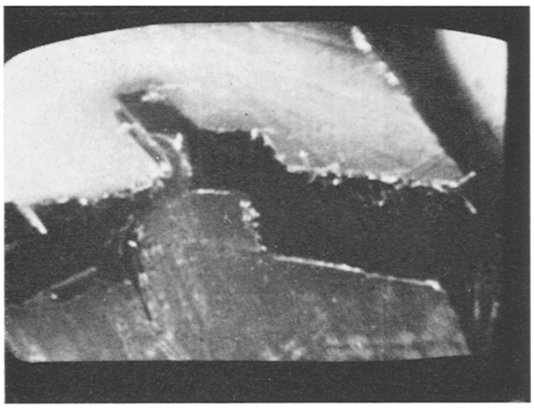

Figure 12 Specimen in fig. 11 after further straining (× 70).

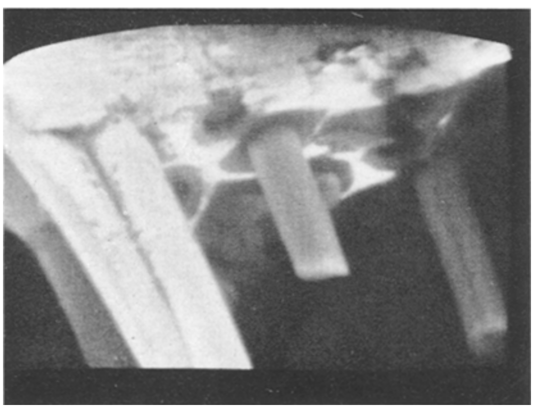

Figure 13 View of one fracture face inside crack shown in fig. 12 at high magnification (× 1000).

indication of there being any back-lash in the controls and no vibration of the stage was observed at magnifications of up to 20000 times. Above this magnification detail was obscured by parts of the uncoated regions charging up.

Though these reported experiments have been mainly on carbon-fibre-reinforced epoxies, the bending module is quite suitable for bend-testing other materials and in no way limited to composites.

An interesting observation made on uncoated material, using the fast scanning and display systems, was that the spread of charge produced by the electron beam could be observed. This was seen as a "silky" pattern, changing as the beam was moved to a fresh uncoated region. The effect is probably due to the build-up of negative charge to equilibrium, in the uncoated region of the crack, repelling the primary electron beam.

5. Conclusions

A bend-testing stage for the "Stereoscan" scanning electron-microscope has been designed and constructed. It is capable of bending realistically-sized beam specimens of materials. Its extreme manoeuvrability allows all the upper faces of the specimen to be examined during straining.

The fracture behaviour of carbon-fibre reinforced epoxy resins has been used as an example, to demonstrate the operation of the bend module. The stage is in no way limited to the testing of these materials, or to composite materials but it is particularly useful with fibrous or particulate material.

It has been possible to observe clearly the difference in failure behaviour between composites fabricated from treated and untreated carbon fibre.

When used with a fast scanning and display system, the recorded observations allowed detailed investigation of the failure process at magnifications of up to 20000.

Acknowledgements

The authors wish to thank Mr D. Pugh for allowing them to use his fast scanning and display system in conjunction with their experiments. They also wish to thank Mr D. Kynaston of Cambridge Scientific Instruments Ltd for his assistance and for the loan of a large modular stage for the scanning electron microscope.

The work described above forms part of the research programme of the Division of Inorganic

and Metallic Structure of the National Physical Laboratory.

References

1. OM. JOHARI (ed.) 1st Annual Symp. on S.E.M. Proceedings (Illinois Institute of Technology Research Institute, 1968).
2. *Idem* (ed.) 2nd Annual Symp. on S.E.M. Proceedings (Illinois Institute of Technology Research Institute, 1969).
3. A. HEPWORTH, T. BUCKLEY, and J. SIKORSKI, *J. Sci. Inst. Ser.* **2** (1969) 789-792.
4. D. DINGLEY, *Micron* **1,** No. 2 (1969).
5. D. PUGH (1970). To be published.

Received 5 June and accepted 18 July 1970