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Direct observation of compressive deformation of PBO fibre in the SEM

C. C. CHAU, M. H. THOMSEN, V. L. St JEOR
Central Research, The Dow Chemical Company, Midland, MI 48674, USA

Polybenzobisoxazole fibres were compressed uniaxially in a tension–compression stage for direct observation of deformation in a scanning electron microscope. Kink bands were observed to develop on the fibre surface at early stages of deformation. They seemed to initiate heterogeneously and propagate across a large part of the fibre. Both the length and thickness of the band increased dramatically with slight compression. Upon continued deformation, cracks or openings were seen to develop from surface irregularities near the kink bands. The cracks or openings extended along the fibre length while new bands continued to develop adjacent to them. Cracks and kink bands seemed to be mutually inducible to result in a localized bulge at later stages of deformation. In Kevlar 49 fibres, cracks or openings seemed to develop at higher compressive strains notably at intersections of kink bands.

1. Introduction

Organic fibres such as polybenzobisoxazole (PBO) and Kevlar are high-performance fibres which possess strong chemical resistance, thermal stability and tensile properties. These outstanding features make them an excellent material of choice for composite applications. One deficiency about the organic fibres, however, is their weak compressive strength. Studies have shown that the compressive strength of organic fibres is about 10% of their tensile strength and less than 20% of common graphite fibres compressive strength [1]. This weakness greatly limits their applications and methods of improving the compressive strength are highly desirable.

The origin of the weak compressive strength problem is of interest and has been studied for many years. The literature has indicated that the poor compressive strength is related to the one-dimensional nature of chain orientation in the fibre [2–4]. During compression, these fibres often show localized plastic deformations in the form of kink bands [5–7] which are in the presence of other morphological variations such as fibrillation and buckling [7, 8]. These observations suggest that strain localization under compression is a key problem in organic fibres, this prevents them from having more uniform or dispersive loading under compression.

Microstructural information generated from the deformation can help in the understanding of failure mechanisms. Structural characteristics can be revealed and implications as to methods of improvement can sometimes be indicated. Although earlier work [9] has discussed the various modes of failure under compression, little has been done to show experimentally the initiation of defects, the interaction between various defect types and its effect on final failure behaviour. In this study, an *in situ* deformation technique was used for direct observation of fibre compressive behaviour in the scanning electron microscope (SEM). The technique utilizes a deformation stage commonly used for tensile stretching of films. With slight modification of the sample holding device and sample handling technique, the stage was found to be useful for fibre compressive studies. The deformation behaviour of PBO fibres was observed with a particular emphasis on the initiation of kink bands and band interactions with existing cracks. Finally, the kinking behaviour is compared between PBO and Kevlar 49 fibres, and the implications regarding the relationship between compressive strength and kink band formation are discussed.

2. Experimental procedure

2.1. Materials and sample preparation

Fibres used in this study included both laboratory-spun fibres and commercial fibres. Multifilament fibres were spun from solutions of polybenzobisoxazole (PBO)/polyphosphoric acid (PPA) by using a dry jet–wet spinning process. The spun fibres were heat treated at elevated temperatures under tension. Commercial Kevlar 49 fibres were obtained from DuPont. Pieces of fibre, 2 in (5 cm) long, were cut from the fibre spools and suspended across supports which were placed in the centre of a Hummer X (Anatech Ltd) sputter coater. The fibres were held in place on the supports by small pieces of double sticky tape. A 3–5 nm layer of Au/Pd, measured using a quartz crystal thickness monitor, was sputtered on to the fibres. The fibres were then carefully removed from the supports without external disturbance. The fibres were epoxy glued on to copper shim stock which had been clamped in the stage clamps of the compressive stage with a 0.8–1.5 mm long distance maintained between the shim stocks. This maintained a fibre gauge length ranging from 0.8–1.5 mm during the test. A schematic view showing the assembly is illustrated in Fig. 1.

2.2. *In situ* compression test of fibres

The *in situ* compression technique used in this study has been briefly described elsewhere [10]. The stage used in the *in situ* compression is commercially available through Fullam Inc., and was originally designed for tensile stretching of films. The sample holder was slightly modified by using a pair of well cut and aligned pieces of shim stock for fibre mounting creating a substage for holding the fibres as shown in Fig. 1. The stage operates on a screw thread simultaneous motion concept which allows both sides of the sample holder in the stage to advance towards each other. It is driven by a handwheel which delivers 11.11 μm linear movement per revolution of the handwheel. After fibres were mounted, the fibres/stage assembly was installed in an Hitachi S-570 SEM. The fibres were first examined under low magnifications (× 50) for a selection of good samples which were well aligned and mounted. Studies were performed by examining the fibre morphology of the chosen sample at various revolutions of the handwheel. Each turn was completed within 5–10 s. Photomicrographs were taken at various handwheel turns. Fibre morphology and comparisons were obtained from photographs of the fibre at selected areas along the fibre and at various compression levels. To avoid fibre deformation out of plane, a shim stock was put underneath the fibre in some studies. The addition of an orientation wire under the fibres also proved helpful.

Coating SEM samples with heavy metals has long been the standard technique for reducing sample damage, sample charging artefacts and increasing secondary emission for resolution improvement. To determine if a thin coating of metal would alter the failure process of the fibres, low-voltage scanning electron microscopy techniques were used on uncoated samples and the results compared with the coated fibre results. No differences in fibre morphology and failure processes could be observed between the coated and uncoated samples. The light application of a heavy metal, estimated to be about 3–5 nm thick, was the approach used throughout this study.

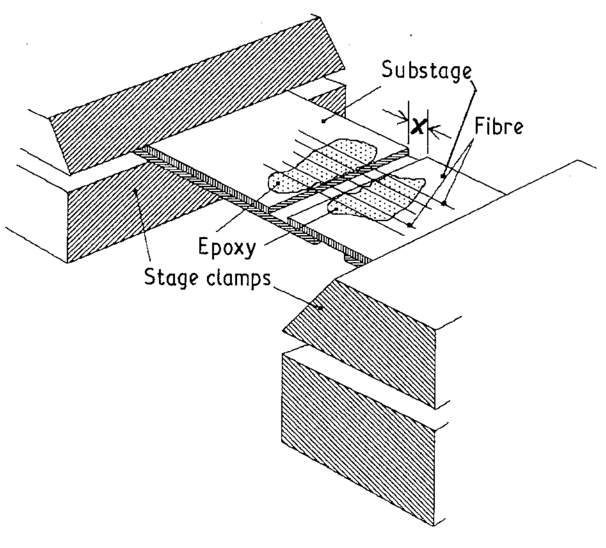

Figure 1 Schematic representation showing assembly of fibre samples on stage. The substage, made of shim stock, is seen with fibre samples mounted across a gap distance, *X*. The gap defines the gauge length of the fibres subject to compression over the space of the gap. (From [10], published with permission of the publisher.)

3. Results and discussion

3.1. Compressive deformation of PBO fibres

The formation of kink bands was considered to be strongly related to the low compressive strength of organic fibres. Exactly how the kink band is formed and how it relates to macroscopic failure are not clear yet. In this study, PBO fibres were observed directly under the SEM during compression. As shown in Fig. 2a–d, at a low magnification, the fibre was compressed by movement of the substage. When the substage was operated to move from the initial position as shown in Fig. 2a to a position at which the gap within the substage was shortened as shown in Fig. 2d, the fibre was bent to form a sharp and clear bending angle in the middle of the fibre. The bending angle changed with the substage movement. For simplicity, the compressive displacement is represented by the number of handwheel turns of the tension–compression stage throughout the study. The bending angle changed progressively with compression with no evidence of embrittlement up to a total of 60 turns, as shown in Fig. 2d, or an apparent compressive strain

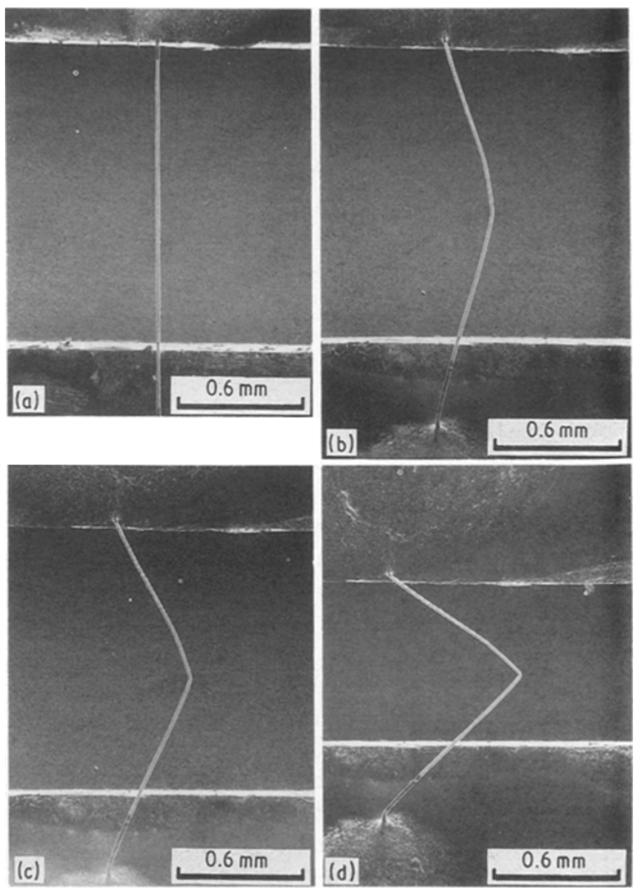

Figure 2 Compressive deformation process of a PBO fibre deformed in compressive stage within SEM (a) 0 turns (b) 8 turns (c) 18 turns (d) 60 turns.

(compressive displacement measured by substage movement divided by the fibre gauge length) about 0.46. During the compression, the fibre remained well fastened to the substage. The instability resulting from the high aspect ratio (fibre gauge length/diameter $\simeq 75$) caused the fibre to be bent easily during compression. The study was considered as a fibre bending experiment. The morphological study was then focused on the compression side of the bending fibre.

Fig. 3a–i show the morphological features of the fibre at various stages of compression. Owing to the

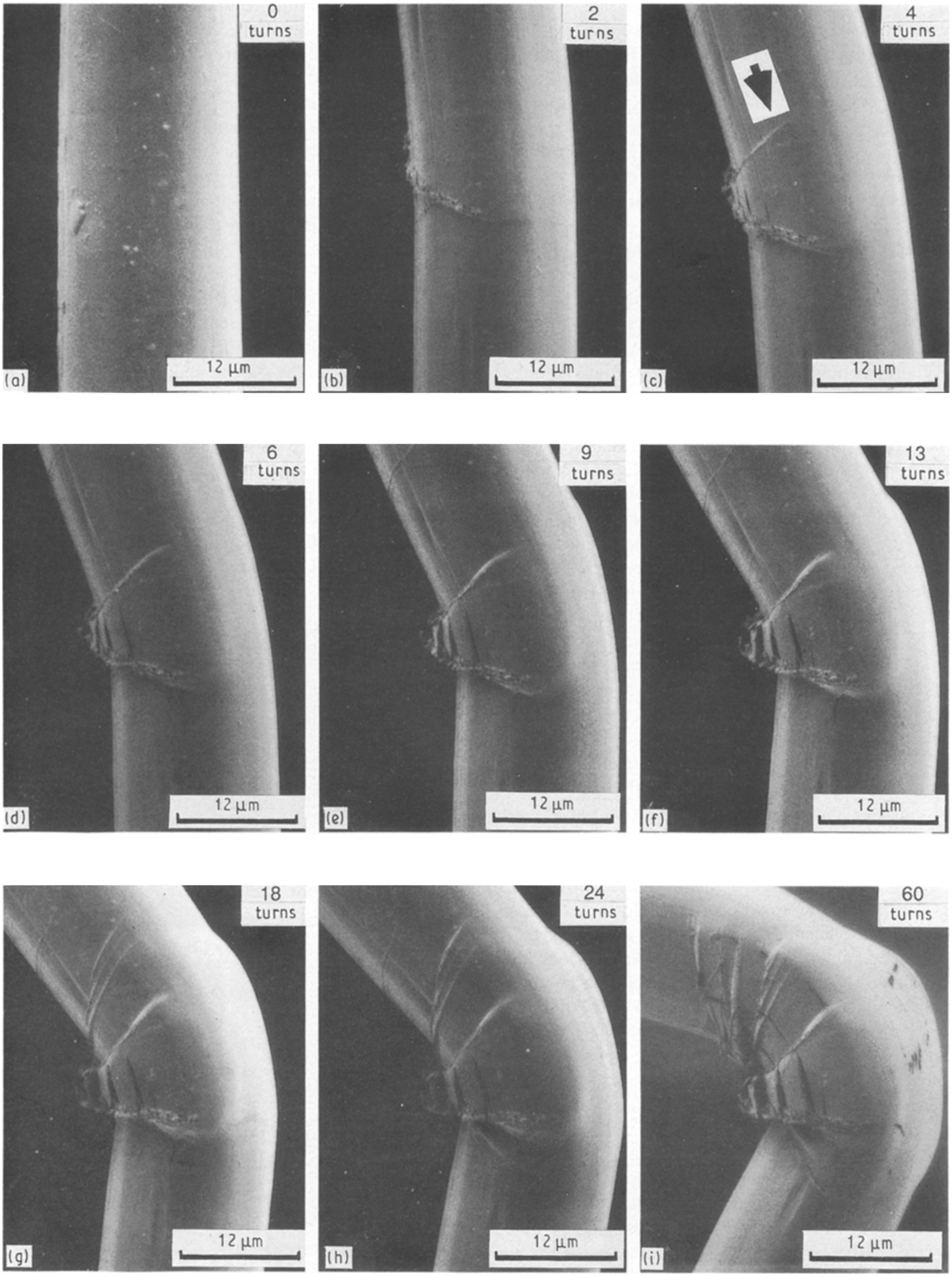

Figure 3 Sequence of PBO fibre deformation process under compression showing development of kink bands, fibrillation and buckling phenomena under different degrees of compression.

uncertainty of the locations of incipient failure at the beginning of compression, Fig. 3a, the undeformed sample is not a part of the sequential observation. It could, however, represent the general surface features of undeformed PBO fibre. The fibre was found to form localized deformation bands in less than two revolutions of the handwheel, as shown in Fig. 3b. The band appears to extend from one side of the fibre, around the circumference, toward the opposite side. The morphology of the band is not clear when examined under this magnification. However, judging from the angle of the band to the fibre axis on the surface, $\sim 67\,^{\circ}$, and the morphology of other fine bands seen along the same fibre, the band is probably a kink band, as commonly observed in organic fibres [5–7]. Localized buckling and fibrillation may be occurring with the band as shown by the bulges and openings on the fibre surface in Fig. 3b.

Further compression causes development of highly localized deformation. These developments present themselves in several aspects of microstructural variation: first, new kink bands appear to initiate adjacent to the existing bands and propagate away from the originating point. This is seen by comparison of Fig. 3b and c. With only two more handwheel turns, a new band (indicated by an arrow) was fully developed. The new band displaced surface scratches laterally which were oriented along the fibre axis, as it extended across them. It stopped shortly before reaching the opposite side, as viewed on the micrograph, of the fibre and left a diffused ending. The original band became slightly thicker with no apparent growth in its length. An outstanding feature occurring with kinking is fibrillation. At the beginning of the process, no cracking or opening could be observed along the fibre. Cracking or opening began to occur, however, as shown in Fig. 3c, with four handwheel turns. The cracks or openings developed along the pre-existing surface scratches or grooves but were localized around the bending point upon further compression. They are oriented along the fibre axial direction and can be viewed as a precursor of fibrillation as usually observed in compressive failure of a fibre in a composite. This observation is evidence of surface defects being sources of localized failure under compression.

Continued compression allowed further development of these localized deformations and an increase in bending angle. As shown in Fig. 3d and e, while the old kink bands increased in thickness with compression, new kink bands continued to form near the deformed region. The cracks or openings are seen increasing in size, and propagating some distance along the fibre at later stages of compression, as shown in Fig. 3i. The deformation at this stage appears to be highly complicated. In addition to kinking and cracking, a strong dilatational effect, as shown by the bulges, seems to develop. This could be related to the buckling of fibrillar structures in the bent region. Because the tensile stress on the outer surface of the fibre gradually increases with the increased bending angle, the kink bands are not expected to extend to this surface. This is shown clearly in this sequence of photographs. Cracks or openings appear on the outer surface as shown in Fig. 3i and are probably due to the high tensile stress developed under large bending.

3.2. The initiation and propagation of kink bands

The formation of kink bands is likely to be precursory to catastrophic failure leading to larger deformations as indicated by the above observations. Previous researchers [11, 12] have proposed models to explain the initiation and propagation of kink bands without direct observations. To see how the kink bands were initiated at the very early stages of compression, studies were performed with a particular focus on kink band formation and morphology with only slight compression. Fig. 4a–d show the morphology and behaviour of a PBO fibre within only one and one-half handwheel turns. Before the deformation, the fibre was straight. A few small precursory kink bands are seen distributed randomly along the gauge length as shown in Fig. 4a (as indicated by a thin arrow). These bands were most likely introduced during processing or handling of fibres. At one-half handwheel turn as shown in Fig. 4b, no change in morphology or in pre-existing precursory kink bands was observed. After one handwheel turn as shown in Fig. 4c, a new band (as indicated by a thick arrow) is seen developing from the surface at a previously featureless spot. The old bands did not seem obviously to increase their thickness or the length of propagation upon the minute compressive displacement. When the compression was

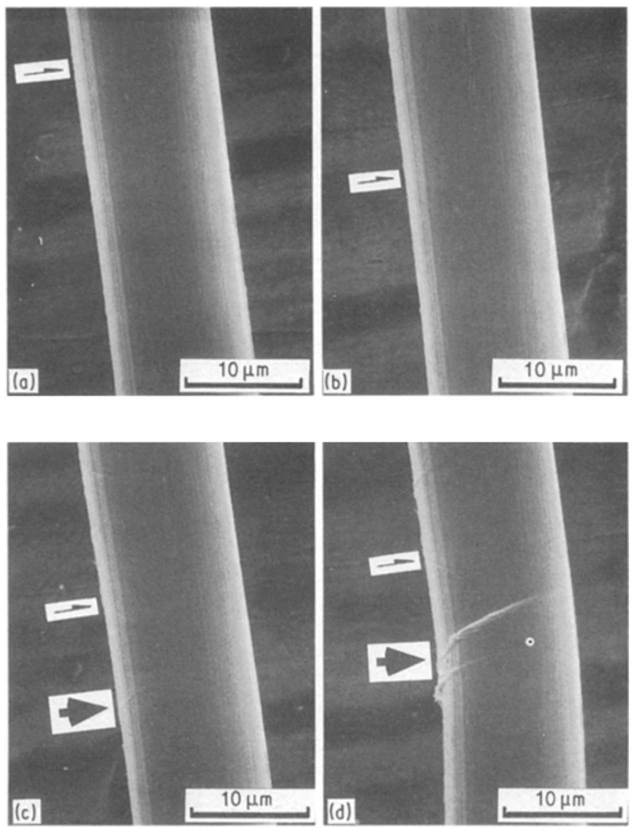

Figure 4 PBO fibre deformed slightly under compression showing the initiation and propagation of kink bands on fibre surface (a) 0 turns (b) 0.5 turns (c) 1 turn (d) 1.5 turns.

increased to one and one-half turns as shown in Fig. 4d, the new band rapidly increased its length and thickness while the adjacent old bands seemed to remain unchanged. The increases in length and thickness of the new band are remarkable, it is estimated to be about 100% in the length of propagation and 300% in the band thickness with only one-half turn of compression. In addition to the formation of the thick kink band (as indicated by the thick arrow), a new band underneath the thick band is seen to develop as also shown in Fig. 4d. This new band was oriented parallel to the thick band with a clear step or bulge on the surface. As described earlier, localized buckling may be occurring with kinking, which could result in a surface bulge. Displacement of surface scratches by the bands can be observed on these micrographs.

This observation strongly suggests that kink band formation is an initiation and propagation mechanism, much like the shear band formation in thermoplastics [13]. The speed of propagation seems quite high as indicated by the significant change in the length of band upon minute compression. This is consistent with observations on the propagation of shear bands [13] and slip lines [14] in other materials. Another observation worth noting is that kink bands can initiate without depending upon obvious pre-existing surface defects. A possible factor causing such initiatory behaviour is probably internal defects, such as voids or local variations in the chain alignment, which could not be resolved from this study.

Kink bands are often observed developing from surface defects or irregularities. Fig. 5a–h show another example in which the failure behaviour is seen to be defect dominated. The fibre surface contained striations or grooves which ran along the fibre axis. The fibre was deformed with only two and one-half handwheel turns of compression. A clear step is shown across most of the fibre diameter, the surface striations are displaced laterally. Exactly how the large shear step was developed is not clear. It is noted, however, that when a band extended across a major scratch or striation, the direction of the band is diverted to orient along the striation for some distance before resuming the original direction. This observation strongly suggests that surface irregularities such as striations, grooves or indentations are initiators of fibre failure. Further deformation, as shown in Fig. 5b–h, resulted in a complicated failure process: first, with five turns of compression, a crack was formed along the fibre direction as shown in Fig. 5b. This could be related to the thick step developed nearby. Probably due to the formation of a crack, new bands initiated from the crack and extended away from it as shown in Fig. 5b–d. These bands seemed to be unrelated to each other at the beginning; however, their interactions became apparent when they were fully developed. Continued deformation appears to cause further cracking adjacent to the existing crack and formation of new bands from the cracks, along with the increase in the bending angle as seen in Fig. 5e–h. These

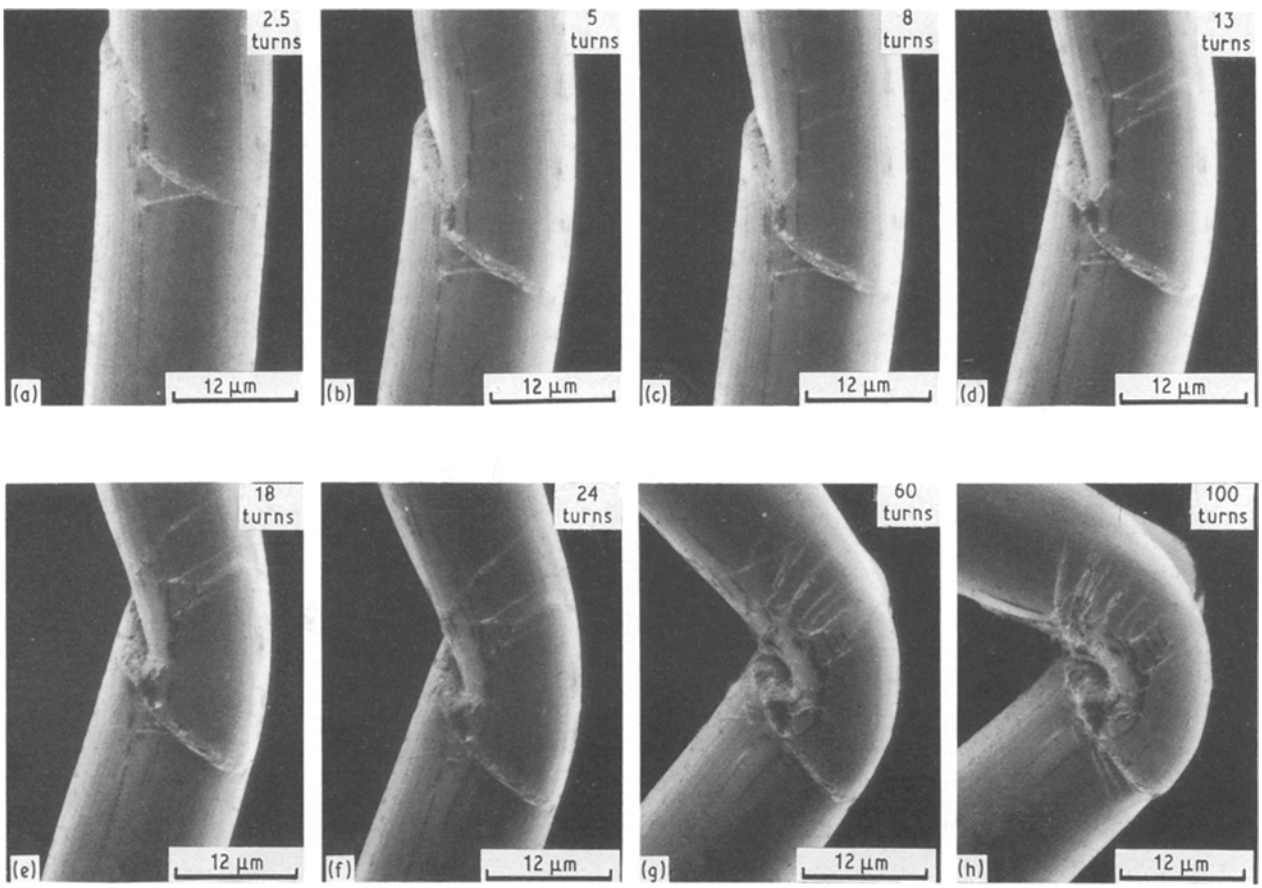

Figure 5 PBO fibre deformed under compression showing the formation of kink bands and cracks initiating from pre-existing defects and the interactions between the deformed regions upon continued compression.

modes of deformation seem to be mutually inducible until a complicated macroscopic failure has developed.

3.3. The dispersity of kink bands

Kink bands formed in PBO fibres appear to be mostly localized at the bending point where maximum compressive strain is developed. Fig. 6a–h show another example of the strain localization effect in a PBO fibre. When the fibre was compressed with five handwheel turns, kink bands were almost fully developed around the bending point. Areas away from the bending point did not show band formation. Continued compression seems mainly to contribute to the localization effect until about 60 handwheel turns as shown in Fig. 6g, at which a new bending point appears to develop adjacent to the existing bending point. Kink bands were then localized around the new bending point. The fibre was further compressed until 100 handwheel turns, as shown in Fig. 6h with only small bands being developed away from the two bends. The compressive strain was almost fully localized at the two bending points. The rest of the areas along the fibre are free of major kinks. Examination of other samples indicated that the strain localization, such as localization of kinking, is prevalent in PBO fibres.

Kevlar 49 fibre has a relatively higher compressive strength than other organic fibres, as reported previously [15]. It is interesting to observe its failure behaviour for comparison. Studies show that Kevlar 49 fibres behave slightly differently from PBO under compression. Fig. 7a–i is an example of deformation sequences. Again, due to the uncertainty of the initial failure spot, the undeformed control, Fig. 7a, is not a picture of the same spot that the rest of the micrographs depict. One outstanding observation is that the fibre can take much more compressive strain, ten handwheel turns based on the same gauge length, before any observable kink band appears, as shown in Fig. 7b. Continued compression was found to produce new and dispersed kink bands along the fibre. These bands, in their fully developed state, seem to have a similar band thickness along the fibre. Cracking or opening did not seem to happen until about 40 handwheel turns or 0.4 compressive strain, as shown in Fig. 7f. The cracks or openings developed at or near the intersections of kink bands without extending outside the band. They were in the fibre axial direction and distributed along the band. At 50 turns of

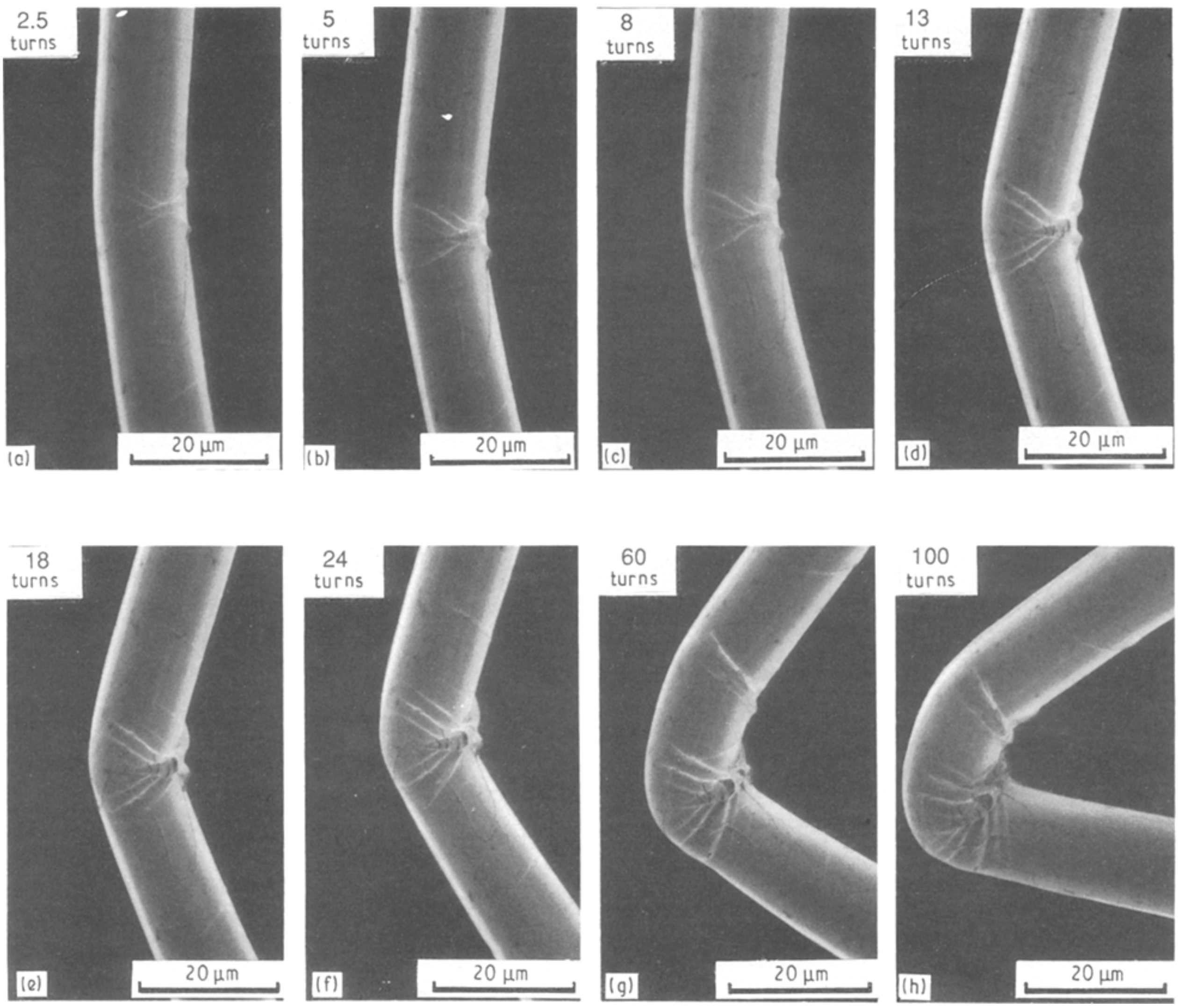

Figure 6 Formation of two major bends on large compression of a PBO fibre showing the localization of kink bands around the bends.

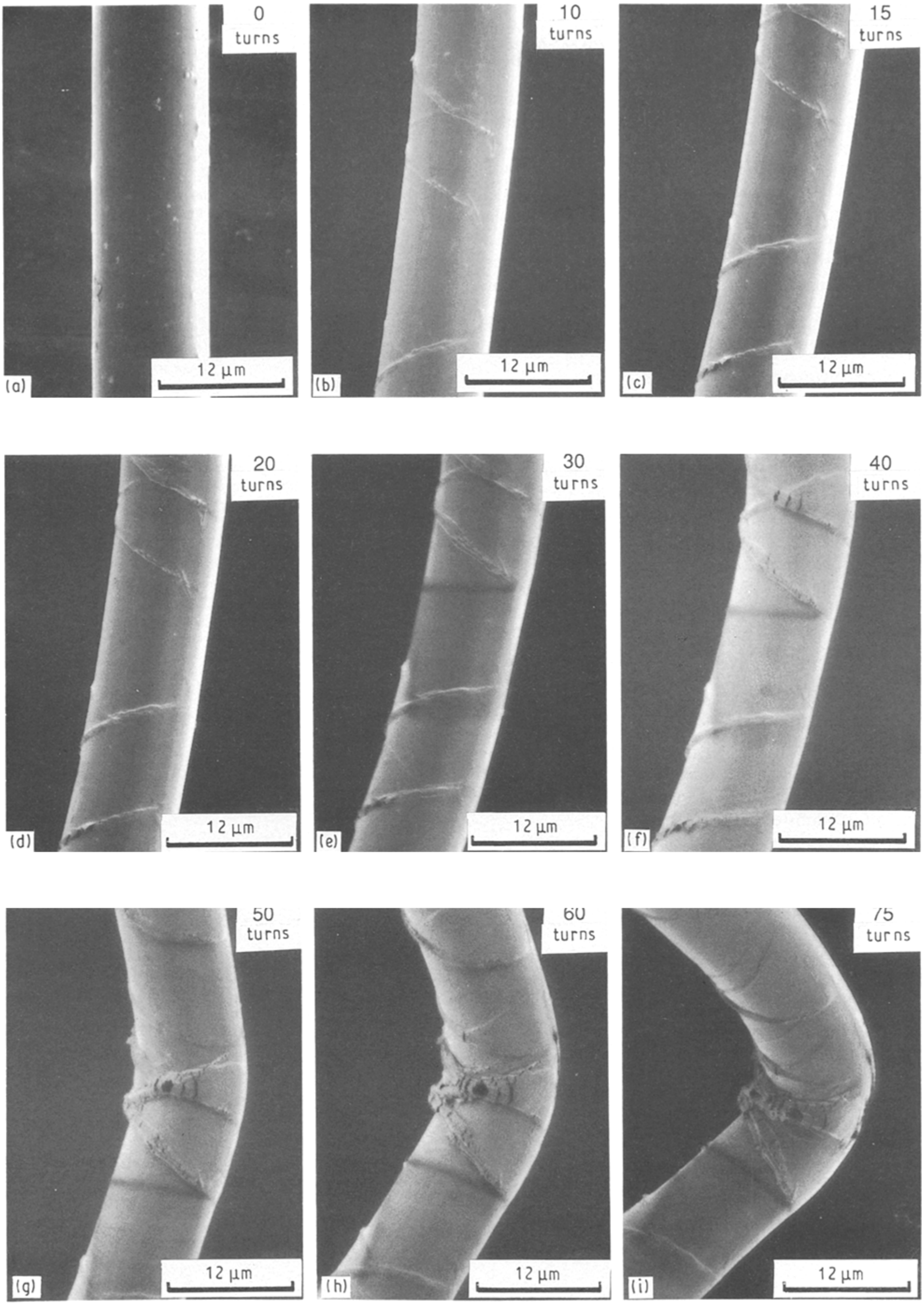

Figure 7 Compressive deformation process in sequence for Kevlar 49 fibre showing the dispersity of kink bands and voiding along the length of fibre on continued compression.

compression, as shown in Fig. 7g, one crack developed into a larger size opening at the intersection without propagating to other portions of the fibre. Other cracks did extend to areas outside the bands with a slight change in size. From 50–60 turns of compression, as shown in Fig. 7h, a few new kink bands are seen to develop in areas away from the bend. Small cracks or openings are seen to develop on the bands. However, no extensive cracking could be observed along the fibre. The whole failure process seems to be kink band dominated.

If kink band formation is considered to be precursory to cracking and buckling, dispersion of kink bands can certainly reduce the strain localization effect so that a more uniform load-bearing capacity could be produced. This conjecture is consistent with the fine shear band behaviour in polystyrene [16, 17], which contributes to the ductile failure mode under compression. Dispersion of kink bands could be a source contributing to the relatively higher compressive strength of Kevlar 49 compared with other organic fibres.

4. Conclusions

1. Direct observations of compressive deformation on PBO fibres showed that kink bands develop at early stages of deformation, followed by cracking or opening formation at higher strains. Localized microscopic buckling seems to accompany kink band formation.

2. Two types of kink band initiation were observed on PBO. Kink bands formed on the surface without pre-existing surface defects, which seemed to initiate somewhere and propagate across a larger part of the fibre diameter with an apparently high speed. Another cause of kinking was surface defects. Kink bands were observed to initiate from pre-existing cracks or irregularities. Interactions between kink bands and cracks seem to exist.

3. Kevlar 49 fibre takes more compressive strains than PBO before the incipient formation of kink bands. Kink bands developed in a dispersed manner along the fibre before cracks or openings developed at the intersections of kink bands.

4. These observations suggest that dispersion of kink bands may be a microstructural guideline for improving compressive strength.

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References

1. S. KUMAR, *SAMPE Q.* 3 (January) (1989).
2. S. J. DeTERESA, Air Force Report, AFWAL-TR-85-4013, March (1985) Ch. III, p. 34.
3. B. W. ROSEN, "Mechanics of Composite Strengthening" (ASM Seminar, Philadelphia, 1964).
4. W. W. ADAMS and R. K. EBY, *MRS Bull.* 16 November/31 December (1987) p. 22.
5. S. J. DeTERESA, S. R. ALLEN, R. J. FARRIS and R. S. PORTER, *J. Mater. Sci.* **19** (1984) 57.
6. M. G. DOBB, D. J. JOHNSON and B. P. SAVILLE, *Polymer* **22** (1981) 960.
7. J. M. GREENWOOD and P. G. ROSE, *J. Mater. Sci.* **9** (1974) 1809.
8. S. R. ALLEN, A. G. FILIPPOV, R. J. FARRIS, E. L. THOMAS, C. P. WONG, G. C. BERRY and E. C. CHENEVEY, *Macromolecules* **14** (1981) 1135.
9. M. G. DOBB, D. J. JOHNSON and B. P. SAVILLE, *Polymer* **22** (1981) 960.
10. V. L. St JEOR, C. C. CHAU and M. H. THOMSEN, in "Proceedings of the 12th International Congress for Electron Microscopy", Vol. 4 (San Francisco Press, San Francisco, CA, 1990) p. 544.
11. S. J. DeTERESA, R. S. PORTER and R. J. FARRIS, *J. Mater. Sci.* **23** (1988) 1886.
12. R. E. ROBERTSON, *J. Polym. Sci. A-2* **7** (1969) 1315.
13. C. C. CHAU and J. C. M. LI, *J. Mater. Sci.* **17** (1982) 652.
14. R. B. POND, in "Proceedings of the ASM Seminar on the Inhomogeneity of Plastic Deformation", 1971 (American Society for Metals, Metals Park, OH, 1973) pp. 1–16.
15. S. KUMAR, *Int. Fibre J.* (February) (1989) 4.
16. J. B. C. WU and J. C. M. LI, *J. Mater. Sci.* **11** (1976) 434.
17. J. C. M. LI and J. B. C. WU, *ibid.* **11** (1976) 445.

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