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Some observations of coarse shear bands in polystyrene

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On the surface parallel to the shear direction, coarse bands appear as ridges, usually of isosceles cross-section with a height-to-base ratio of about 1/10. They split and join with thickness conservation but a corresponding change of cross-section. The shear strain along the same band is almost a constant with a most probable value of 1.8 but a spread between 1 and 3 among different bands. The bands terminate by reducing the shear strain to zero or by converting themselves to a bundle of fine bands at obstacles. Upon annealing the height of the band ridge is reduced until it disappears. When a banded surface is polished, the band pattern reappears upon either annealing or recompression. However, in the former case, the bands are valleys with reverse shear strain but in the latter they are ridges with almost no strain. Similar behaviour occurs on the polished cut surfaces in the middle of the specimen after annealing. On the side surface, the bands are wavy and they are steps rather than ridges. They also split and join with thickness conservation and they terminate freely. Based on these characteristics, the propagation of coarse bands can be viewed as the motion of macroscopic Volterra dislocations.

1. Introduction

As in metals, shear bands appear on the surface of polymers during deformation. These bands have been studied by many people, notably Argon *et al.* [1], Bowden and Raha [2], Brady and Yeh [3, 4] and Kramer [5]. Wu and Li [6] recognized two types of shear bands in atactic polystyrene after compression: coarse and fine shear bands. They compared their morphology, propagation behaviour, strain contribution, ductility characteristics and yielding criteria and concluded that there are two distinct deformation mechanisms. What these mechanisms are exactly remains to be studied. This communication represents a continued effort in the attempt to understand the deformation of atactic polystyrene. This study is concentrated on the coarse bands.

2. Experimental details

2.1. Materials and sample preparation

The atactic polystyrene used in this study was the same as that used in previous experiments [6]. Samples were cut from 0.25 in. thick sheets of commercial products which were compression-moulded in an amorphous state by the Westlake Co. The glass transition temperature was determined to be 101° C.

Materials cut from the sheet were annealed for 20 h at 115° C to eliminate any moulding strains and were furnace cooled slowly to room temperature over a period of 6 h. Samples 2 cm × 2 cm and 2.4 cm × 1 cm were then cut by using a milling machine. A hole of 1.1 mm diameter was drilled at the centre of the square specimens and a notch of 60° was made at one side of the rectangular specimens to serve as stress concentrators from which shear bands would initiate. All specimens were polished to 0.05 μm alumina finish. They were then annealed again at 96° C for 16 h to remove the polishing stresses and cooled slowly to room temperature.

2.2. Mechanical compression test

Specimens were compressed in an Instron machine at different strain-rates to initiate plastic flow. To produce the thick coarse band, the rectangular

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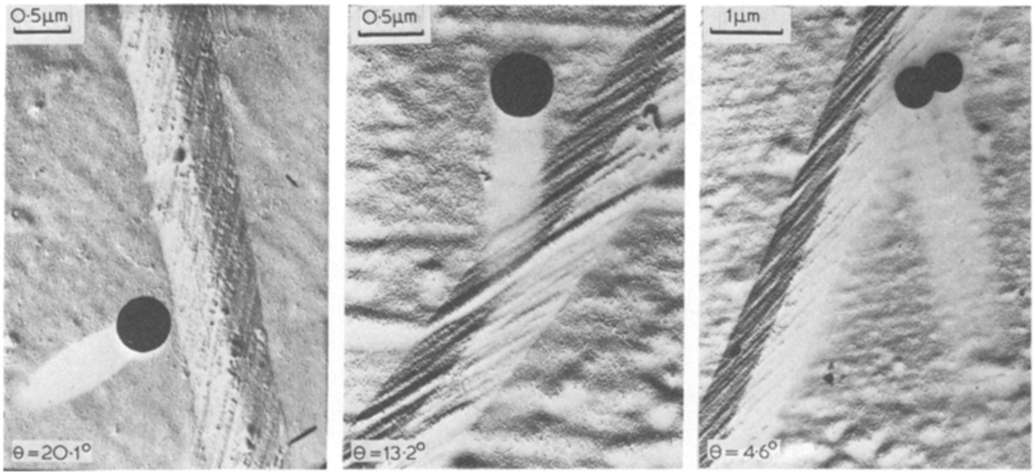

Figure 1 Coarse bands with polystyrene balls at different shadowing angles (E.M.).

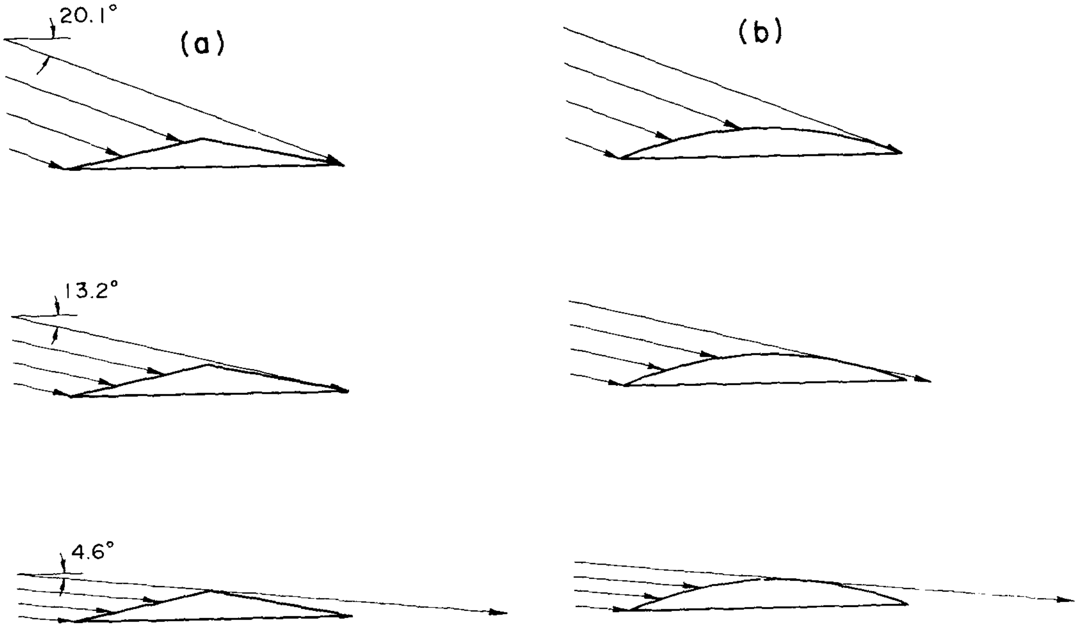

Figure 2 Triangular versus curved cross-sections of the coarse band.

samples were compressed at a high strain-rate of 0.1 $\sec^{-1}$. The thick bands would initiate from the notch and propagate speedily toward the other side of the specimen. Note that the shape of the specimen must be such that the bands can propagate all the way to the free side surface. Otherwise the bands will not be well defined. As soon as the shear bands reached the other side of the specimen, the cross-head was raised at the same rate using cross-head control dials.

2.3. Replicas for electron microscopy

The high-resolution single-stage replicating technique was used. This is the same as the one used previously [6] but with some modifications. It is described below:

(1) Shadow the specimen by evaporating a heated Pt–Pd wire (8 mil* in diameter and 1.5 in. long). In order to determine the surface topography of the coarse bands, drops of aqueous solution containing polystyrene balls, all of 0.481 μm

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*1 mil ≡ 2.5400 × 10^{-5}m.

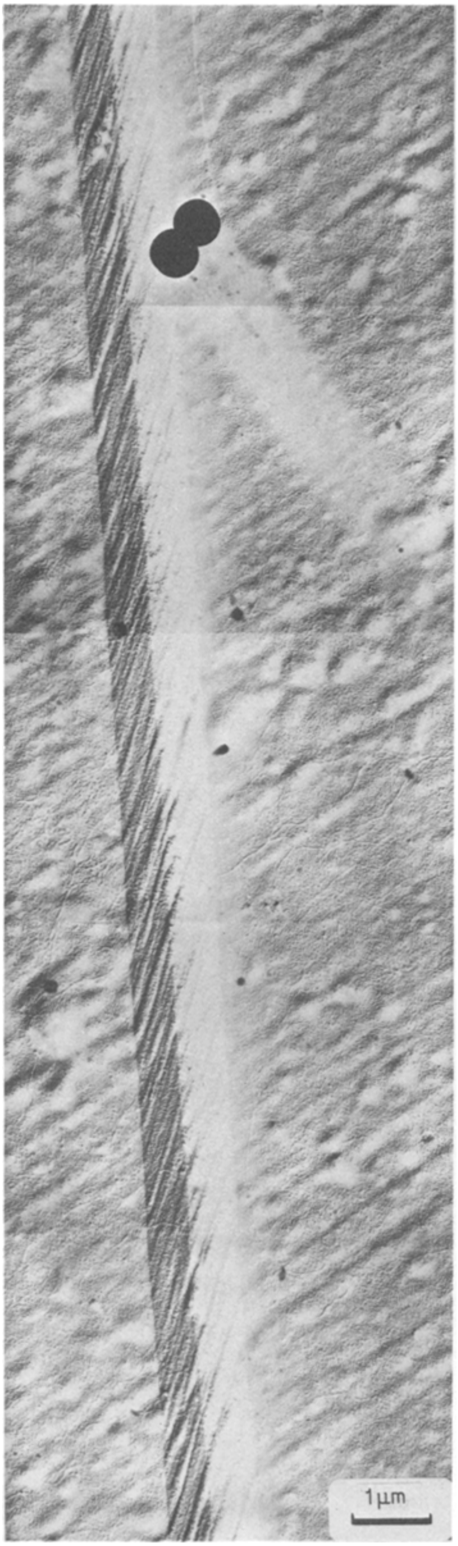


Figure 3 Another view of coarse band with polystyrene balls (E.M.).

diameter, were placed on the specimen surface and dried before shadowing. The shadowing angles ranged from 60° to about 86° measured between the normal of the specimen surface and the straight line connecting the specimen and the evaporation source. Specimens were placed on the platform at a distance of 8 to 10 cm from the source.

(2) Evaporate a layer of carbon on the shadowed specimen surface to increase the strength of the replica.

(3) Place a drop of 50% aqueous solution of polyacrylic acid at the specimen surface and allow to dry for about 10 h.

(4) Peel off the dried polyacrylic acid pieces with tweezers, the shadowed material should adhere tightly to the dried pieces.

(5) Put the polyacrylic acid pieces in water for about 30 min to ensure complete dissolution of the polyacrylic acid in water. The replicas should float to the top of the water.

(6) Carefully lift out the replicas with 150 mesh/in. grids by using tweezers.

Replicas were then examined in the JEM 100B transmission electron microscope. High-contrast images of coarse bands can be obtained. The small polystyrene balls were randomly interspersed between the bands. The polyacrylic acid on the replicas should be dissolved completely so that the replicas become smooth and clean.

3. Results and discussion

3.1. Surface topography of the coarse band

In the previous paper [6], the coarse bands always showed black and white contrasts with a sharp demarcation line in the middle. It was not certain whether the bands are ridges or valleys on the surface. To settle this question and to determine more about the surface topography of the coarse bands, polystyrene balls of 0.481 μm diameter (uniform size) were placed on the surface before replication as described in Section 2.3. As seen in Fig. 1 the shadow of the polystyrene ball always points to the same direction as that of the coarse band indicating that the band is a ridge on the surface.

To gather more information on the cross-section of the coarse band, different shadowing angles were used in the replica preparation. As shown in Fig. 1, the angles were calculated from the diameter of the polystyrene ball and the length of the shadow. Inspection of the photographs reveals that the demarcation line between the black and white

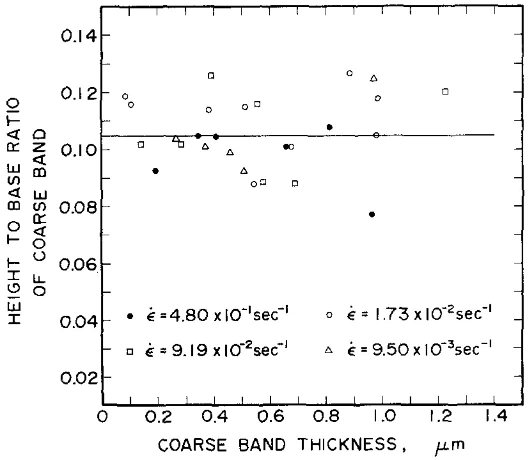


Figure 4 Height-to-base ratio of coarse bands developed at different strain-rates.

regions in the coarse band does not seem to be affected by the shadowing angle. Such an observation seems to suggest that the cross-section of the coarse band is not a curve. As shown in Fig. 2b, a curved surface would change the proportion of the black and white contrasts in the band when the shadowing angle is changed. Furthermore, for a curved surface, the contrast should be a gradual change from black to white with different shades of grey instead of a demarcation line separating the two regions of uniformly black and white contrast. Hence it is more likely that the cross-section of the coarse band is an isosceles triangle as shown in Fig. 2a. Such a cross-section is consistent with the observations. Confirmation of this conjecture will be presented later for thick bands, using a surface profilometer.

As seen in Fig. 1, the shadow of the ridge summit appears when the shadowing angle is (90–4.6) or 85.4°. Another view is shown in Fig. 3. From this picture, the height-to-base ratio of the triangular cross-section is 0.12. Note that, when the angle is as low as 4.6°, the actual shadows of polystyrene balls, which are much clearer when viewed inside the electron microscope, are somewhat longer (about 12 times the diameter of the balls) than those seen in the picture. Many measurements of this ratio were made and the results are shown in Fig. 4. The average ratio is 0.105 ± 0.015 which has no obvious dependence on either the strain-rate of compression or the thickness of the band within the ranges studied. A direct measurement of this ratio will be reported later for thick bands, with a surface profilometer.

3.2. Splitting and joining of coarse bands

A coarse band usually maintains a nearly isosceles cross-section during propagation. It can split into two bands as shown in Fig. 5. Just before splitting, the cross-section may become asymmetrical and the splitting takes place at the broader side of the ridge as shown in Fig. 6. One of the split bands still propagates in the same direction as the main band before splitting, while the other propagates in a different direction. Both split bands seem to acquire isosceles cross-sections afterwards. A schematic illustration of the change in cross-section is shown in Fig. 7. Sometimes a band can split in the middle into two bands of about equal thickness as shown in Fig. 8. These split bands also acquire isosceles cross-sections. A schematic representation of the change in cross-section is shown in Fig. 9.

Inspection of Fig. 5 and 8 gives an impression that the angle between the split bands is related to the asymmetry of the cross-section of the splitting band. However, careful measurements on many cases of splitting failed to confirm such a relationship.

The sum of the thicknesses of the split bands is about 7% larger than the thickness of the splitting band, as shown in Fig. 10.

The coarse bands can not only split but can also join together into one band. Since one set of coarse bands usually starts from the notch and

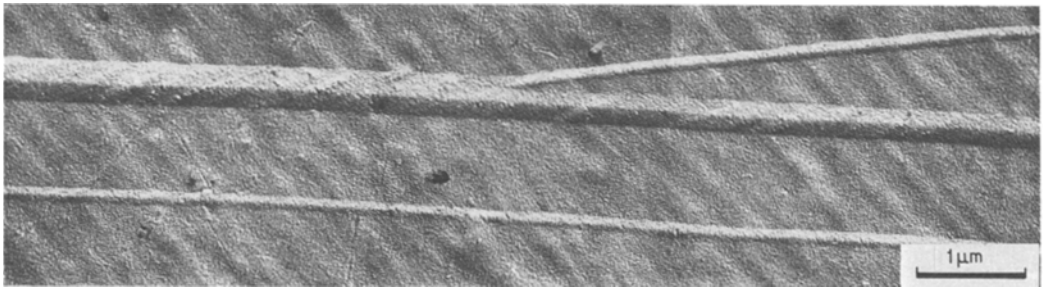


Figure 5 Splitting of a coarse band (E.M.).

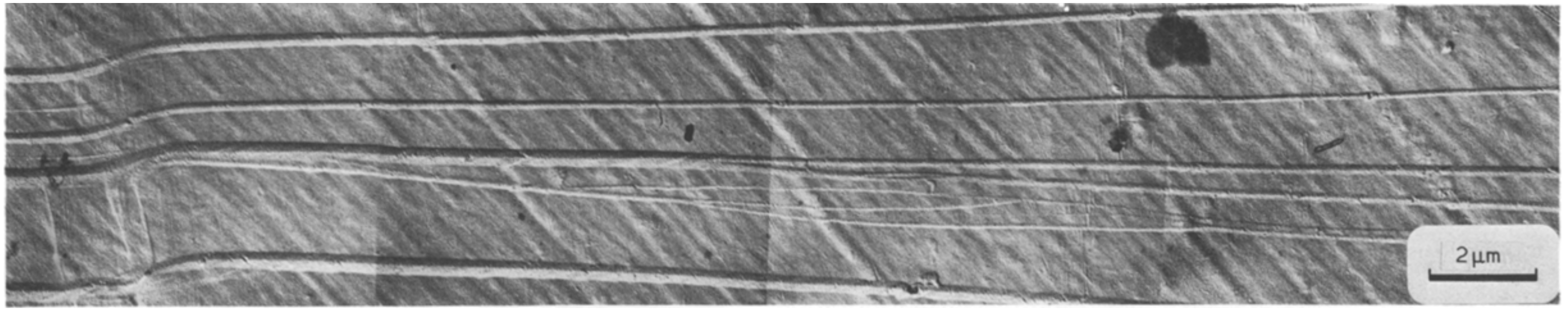


Figure 6 Variation of cross-section of a coarse band before splitting (E.M.).

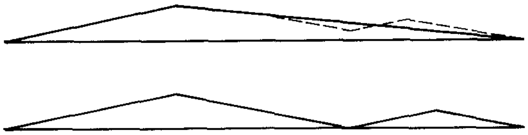
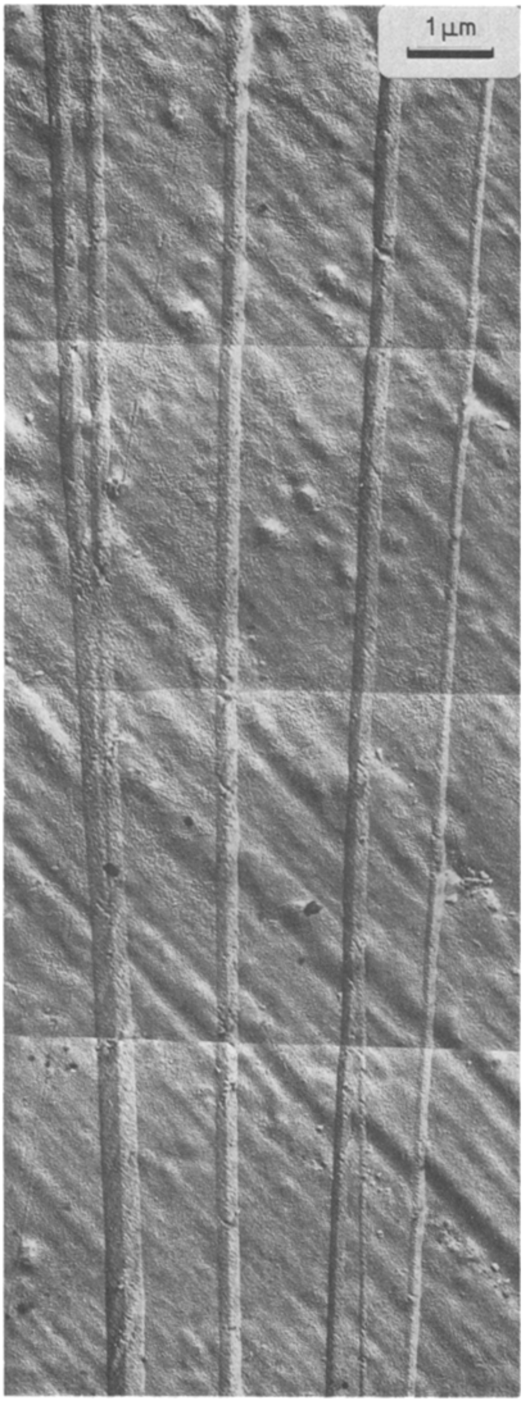


Figure 7 Splitting of coarse band with asymmetric cross-section.



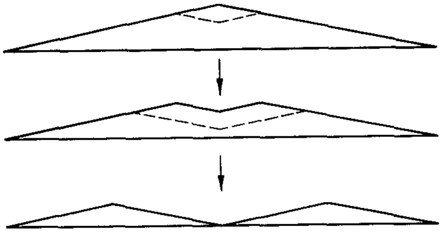


Figure 9 Splitting of a coarse band with symmetric cross-section.

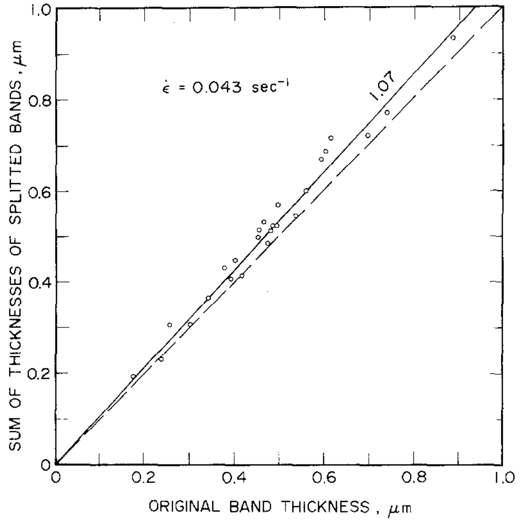


Figure 10 Thickness conservation of the splitting process.

propagates away from it, it is possible to determine the direction of propagation. As shown in Fig. 11, the direction of propagation is indicated by the arrow at the corner of the photograph. It is seen that the coarse bands can join as frequently as they can split. The joining process seems to be the reverse of the splitting process, i.e. the two small isosceles cross-sections join their bases together to become a large asymmetric or isosceles cross-section taking backward steps as suggested in Fig. 7 or Fig. 9.

A careful inspection of Fig. 11 reveals the possibility that the cross-section can change from asymmetric to isosceles during propagation. At the time of joining, the joined bands are asymmetric as shown in Fig. 11. Then after propagating some distance they seem to become isosceles.

Fig. 12 shows an interesting situation in which a band is splitting but has not quite completed the

Figure 8 Splitting of coarse band in the middle into two bands of about equal thickness (E.M.).

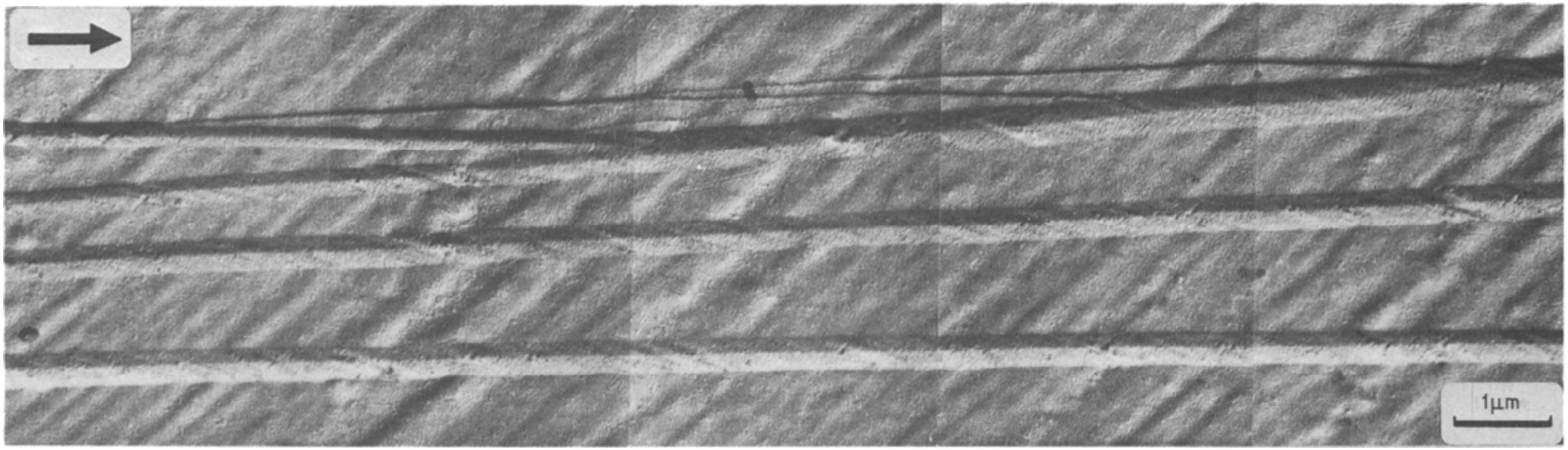


Figure 11 Joining of coarse bands (E.M.).

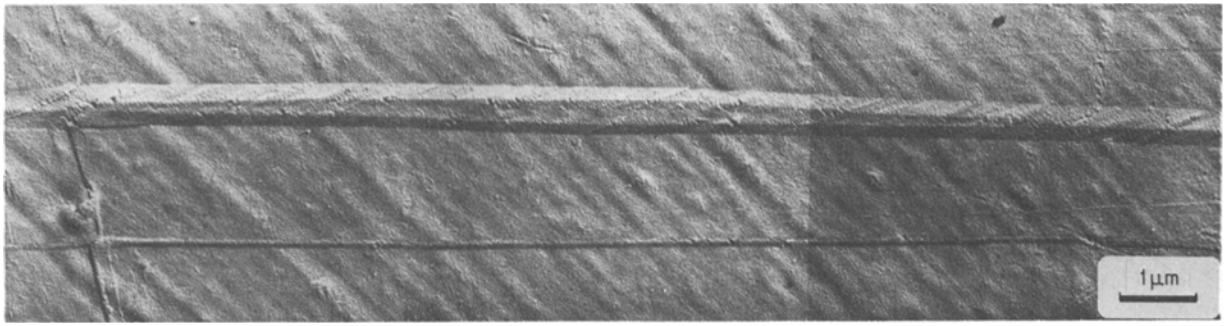


Figure 12 Partial splitting and re-joining of a coarse band (E.M.).

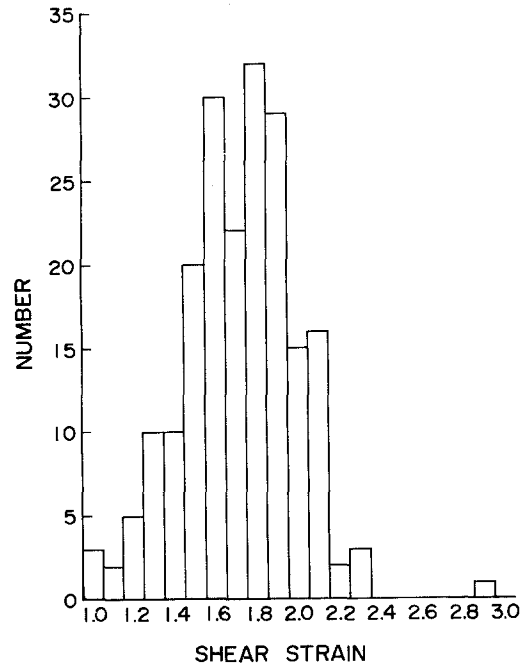


Figure 13 Shear strain distribution of coarse bands in one specimen.

process before the two partially separated bands join together again. It shows that the splitting and joining are two reversible processes during the propagation of coarse bands.

3.3. Shear strain in the coarse band

It was reported [6] that the shear strain in the coarse band falls between 1.2 and 1.6. To see whether this is true in the specimens used in this study, 200 measurements were made. The distribution is shown in Fig. 13. The shear strain is defined as the displacement of a scratch divided by the local thickness of the band. It is seen that the range is larger, between 1 and 2.5 with one as high as 2.9. The most probable shear strain is about 1.8. There appears to be no apparent relationship between the shear strain and the thickness of the band.

3.4. Termination of coarse bands

Coarse bands can terminate before reaching the other side of the specimen surface. They can terminate in an otherwise feature-free area or they can terminate by converting themselves into fine bands. The first type of termination is shown in Fig. 14. Several terminations are seen without any obvious obstacles. The bands simply stop by a fading away of their identity at the ending. The displacements are fairly constant along the band and gradually decrease to zero at the end. Some measurements are shown in Fig. 15 for the thinner band in the photograph. Macroscopically the band end represents a boundary between slipped and unslipped areas and is, therefore, a Volterra [7] type of dislocation. However, since the change of displacement is not abrupt at the end, it is a special kind of Somigliana [8] dislocation.

The other type of termination is shown in Fig. 16. The propagation of a band is blocked by another band near the middle of the photograph and converts itself into a bundle of fine bands. The fine bands always appear as two sets nearly perpendicular to each other. They are shear bands as evidenced by the kinking of other bands in their path. The general direction of spreading of the fine bands is also different from that of coarse bands as reported before [6].

3.5. Annealing of coarse bands

Annealing of coarse bands causes their shear strain to recover and the bands to disappear. The kinetics of shear strain recovery is reported elsewhere [9]. The fact that the band ridges disappear also is demonstrated by the following experiment. The banded surface was well polished before annealing to remove all the ridges. Then the specimen was annealed for 75 min at 105° C. New bands appeared on the surface. A comparison of the new pattern with the old one before polishing shows that the new bands are at the old band locations. Furthermore, the new bands are valleys instead of ridges as shown in Fig. 17, using 0.481 μm polystyrene balls again to cast shadows during replication. It is seen that the directions of the shadows of the polystyrene balls are just the opposite of those of the new bands. The scratches were introduced during polishing and hence were straight to begin with. However, as seen in Fig. 17, the scratches were displaced by the annealed bands as a result of shear strain recovery.

The variation of band cross-section was observed further using a Sloan Dektak surface profilometer on a thick coarse band. A single coarse band of about 280 μm thick was produced on a 1 × 2.4 cm^2 notched specimen by compression. The surface profile is shown in Fig. 18a. It is seen that the base-to-height ratio is about 11, in agreement with the electron microscopy results just reported for thinner coarse bands. Note that the vertical and horizontal magnifications are different. The

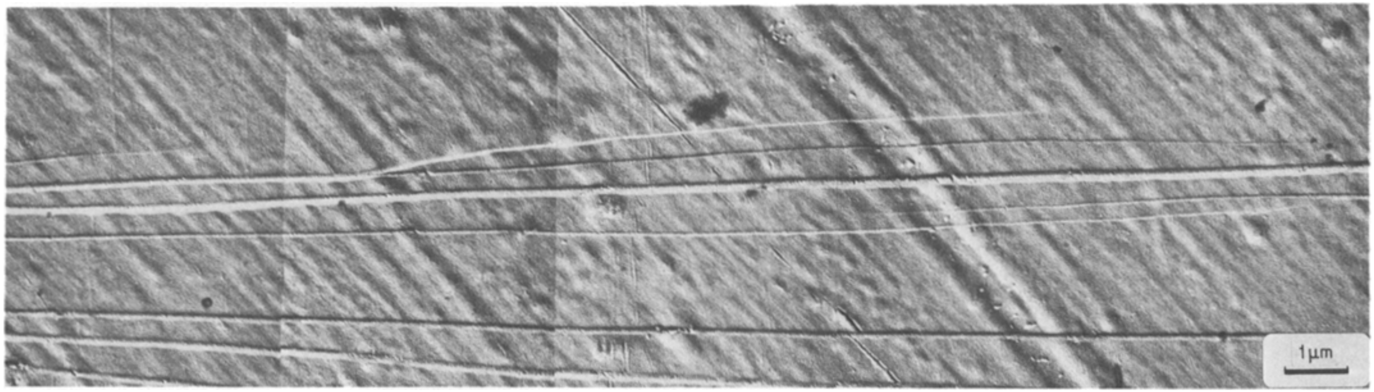


Figure 14 Free termination of coarse bands (E.M.).

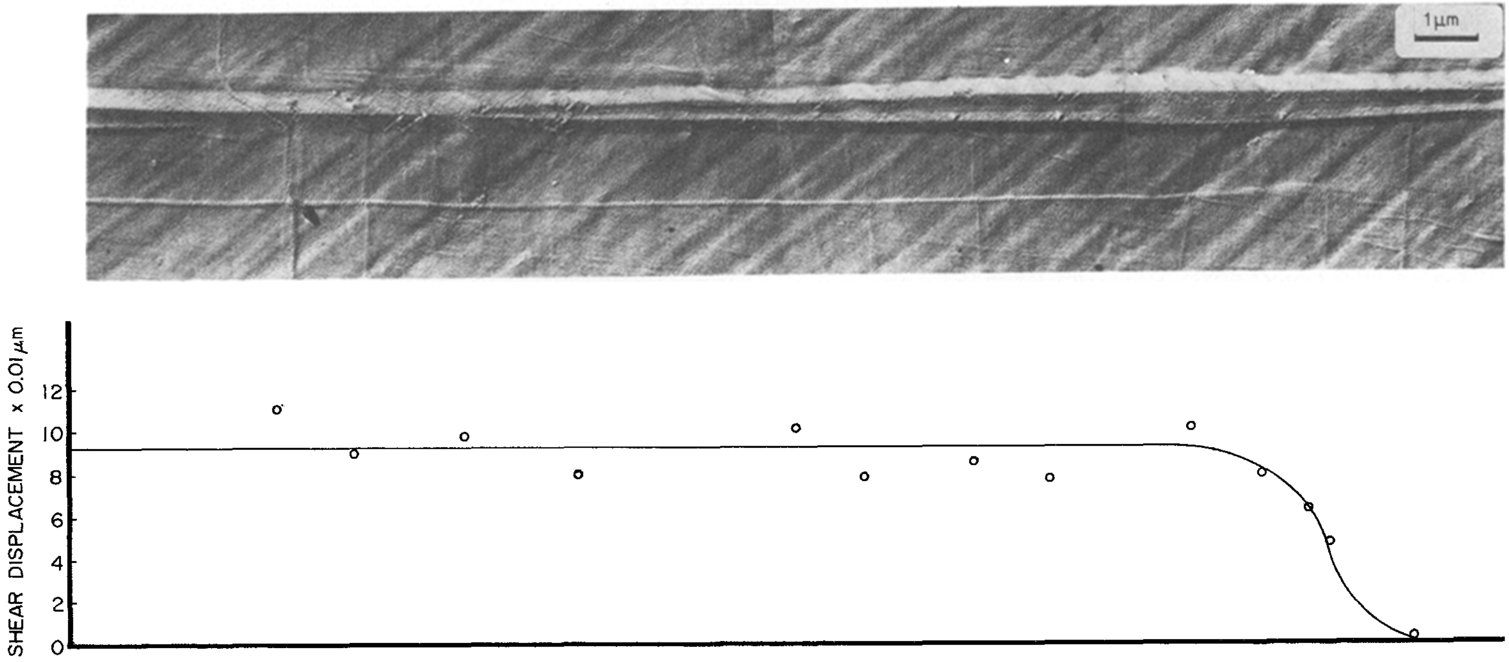


Figure 15 Shear displacement along a coarse band.

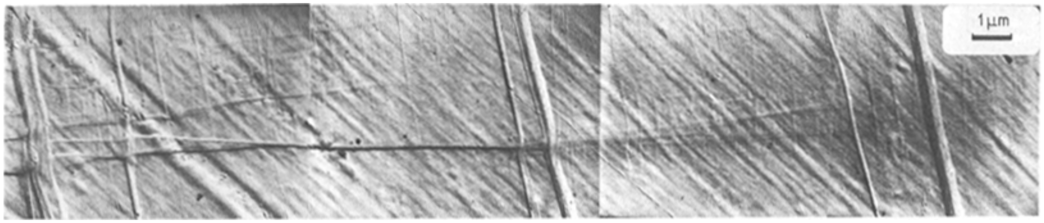


Figure 16 The conversion of a coarse band into fine bands at an obstacle (E.M.).

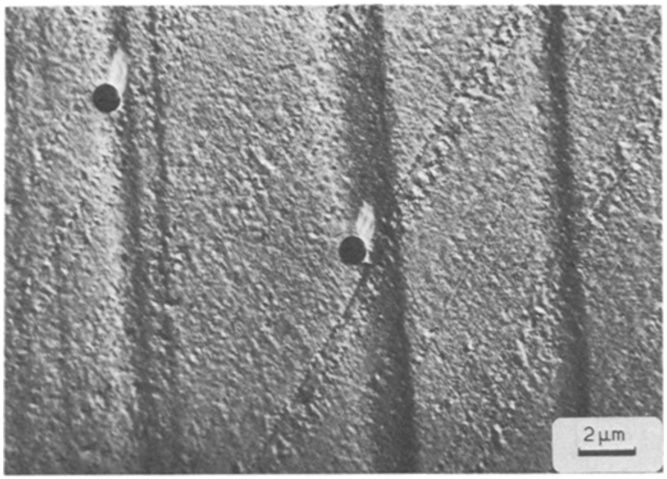


Figure 17 Polished banded surface annealed to develop valleys probably at the original coarse band locations (E.M.).

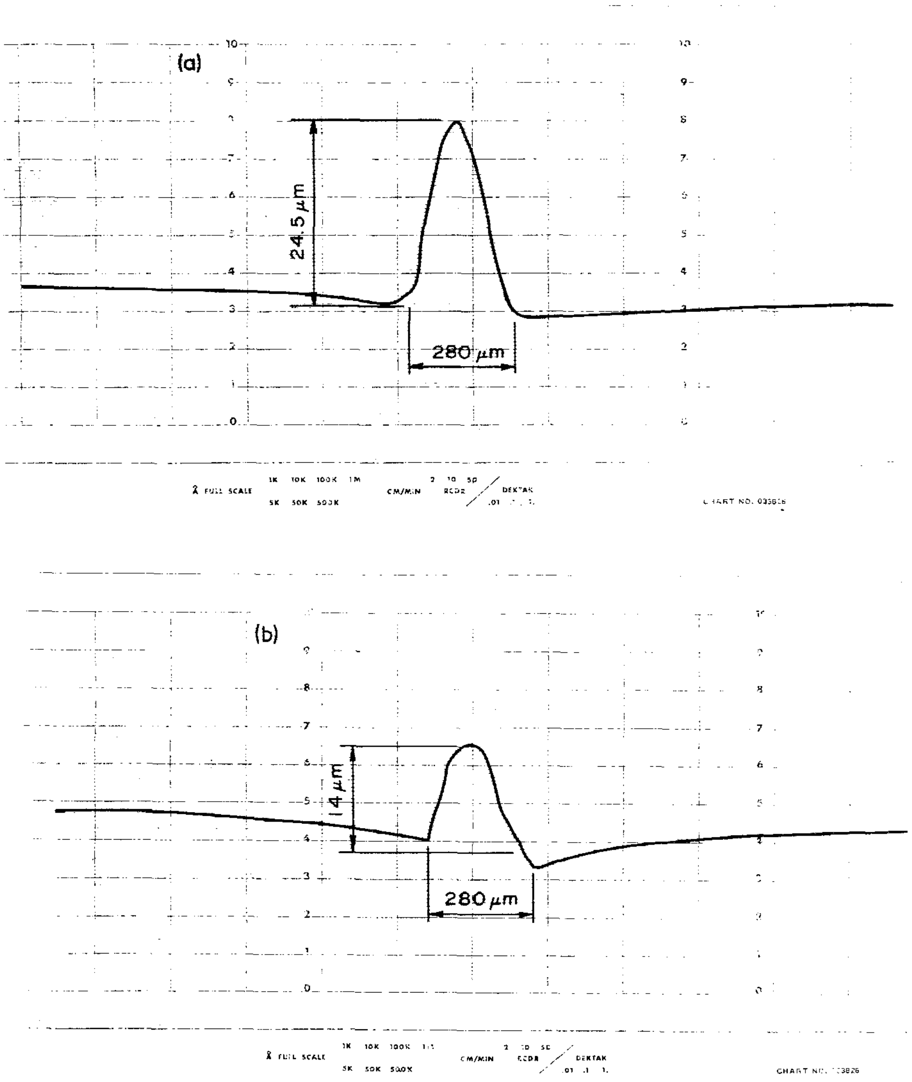


Figure 18 Dektak trace of coarse band profile (a) before annealing, (b) after annealing.

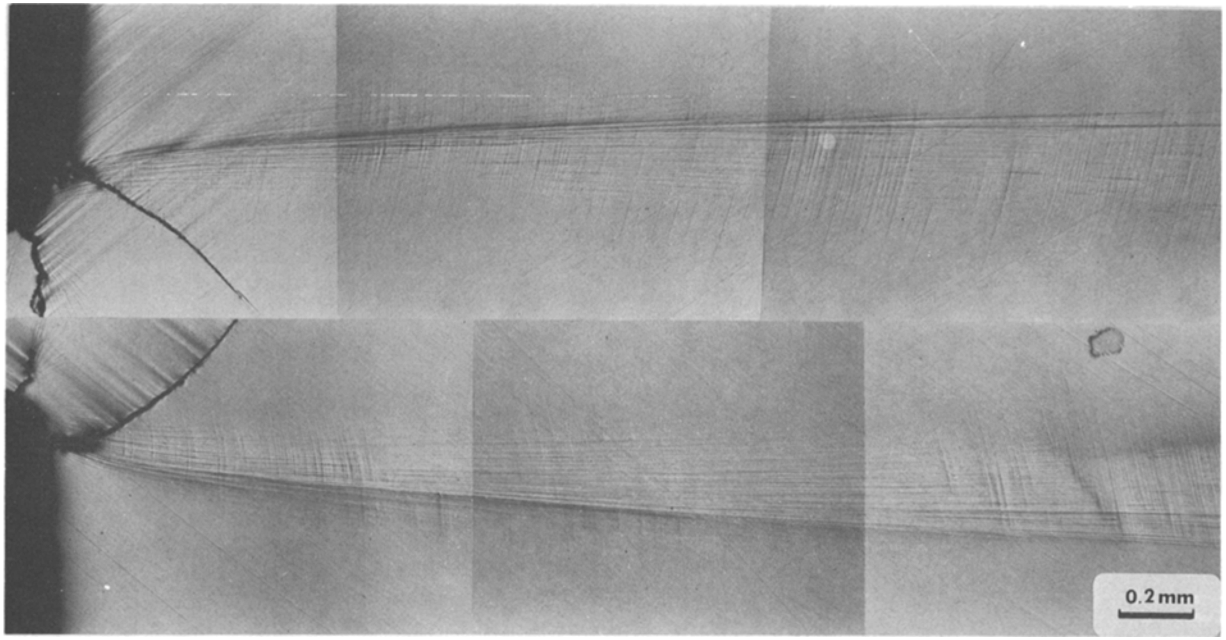


Figure 19 Annealing of polished cut surfaces after deformation (O.M.).

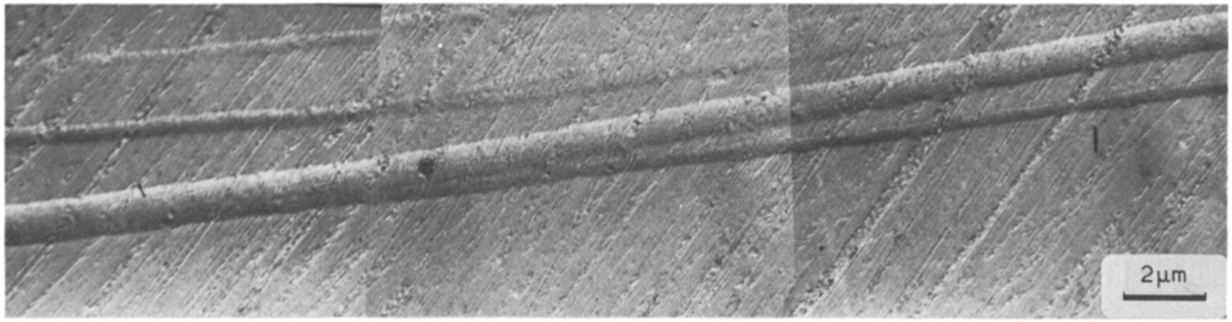


Figure 20 Appearance of bands on a polished banded surface after redeformation (E.M.).

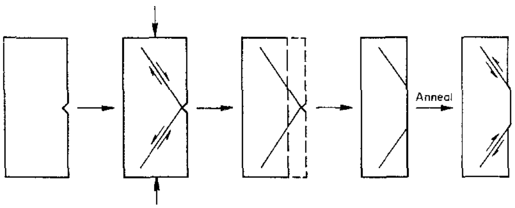


Figure 21 Experimental procedures to reveal slip steps on the side surface.

triangular cross-section also confirms our notion based on electron microscopic observations using different shadowing angles. It seems that the band cross-section is similar for both thin and thick bands. The banded specimen was then annealed at 106° C for 10 min and the band profile traced again by the Dektak. The result is shown in Fig. 18b. It is seen that the ridge is rounded at the top while the base remained the same. Further annealing reduced the height even more and finally the ridge disappeared completely. Such a volume shrinking effect is consistent with the annealing of polished surfaces just described.

The shear strain recovery and the apparent volume shrinkage of the coarse bands are not limited to the surface region. A banded specimen was cut into two halves by sawing along a plane parallel to the shear direction. The cut surfaces were polished and both halves annealed at 105° C for 75 min. Both halves showed bands with matching patterns as shown in Fig. 19. Both were valleys on the surface as revealed by polystyrene balls in the electron microscope. Furthermore, the scratches were displaced (not shown) by the bands after annealing due to shear strain recovery.

3.6. Redeformation of coarse bands

Recompression in the same direction after polishing also makes the old bands reappear as shown in Fig. 20. However, new bands are also produced depending on the extent of recompression. The difference is that the new bands have a regular shear strain (~ 1.8) but the old bands have very little shear strain. Although both are ridges on the surface, the height-to-base ratio is much smaller for the old band. This different behaviour of old and new bands suggests that there is a saturation of shear strain in the coarse bands such that further deformation can strain the band only slightly.

3.7. Morphology of slip steps

So far all the observations have been made on the surface parallel to the direction of shear and, therefore, ridges instead of slip steps were seen. On the side surface which makes an angle (~ 40°) with the shear direction, steps could be observed. However, steps formed on a polished surface after compression were not sharp enough to be photographed. Hence the annealing technique just described was used here as follows. As shown in Fig. 21, a notched specimen was first compressed to form bands and then the specimen was cut parallel to the side surface to remove the notch. The cut surface was polished and the specimen annealed at 104° C for 4 h and furnace cooled. Steps were formed on the polished surface due to strain recovery as shown schematically in Fig. 21.

Fig. 22 shows the pattern of slip steps. Unlike the other surface where the ridges are straight, the slip pattern is wavy on this surface. The fact that these lines are steps is shown in Fig. 23 by using an interferometer. The step heights seem to range from about 500 Å to 12 000 Å. The step direction is indeed what was expected in Fig. 21. To see the steps more closely, replicas were made as usual and the electron micrograph is shown in Fig. 24. The contrast shows that the bands are steps and not ridges. The scratches are not displaced showing the lack of parallel shear component. The bands also split and join with conservation of thickness.

These observations suggest that individual coarse shear bands do not extend throughout the thickness. They start from a source (notch) and propagate in two directions which form the plane of a band packet. One direction, the shear direction, is well defined and the bands propagate in straight lines. The other direction, which is perpendicular to the shear direction, is not well defined and the bands propagate in wavy curves. In addition, the bands split and join in both directions during their propagation.

4. Conclusions

(1) On the specimen surface parallel to the shear direction, the coarse bands appear as ridges, usually of isosceles cross-section with a height-to-base ratio of about 1/10.

(2) The coarse bands split and join with thickness conservation, i.e. the sum of the thicknesses of the split (or joining) bands is about the same as

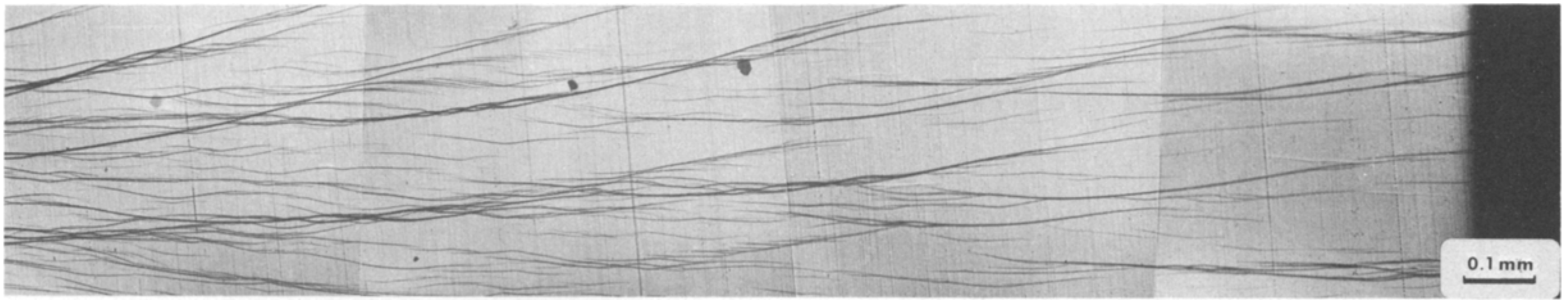


Figure 22 Morphology of slip lines on the side surface (O.M.).

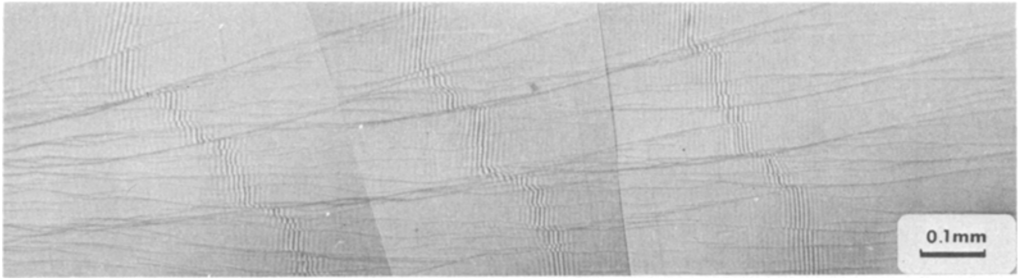


Figure 23 Slip steps as revealed by interferometer fringes (O.M.).

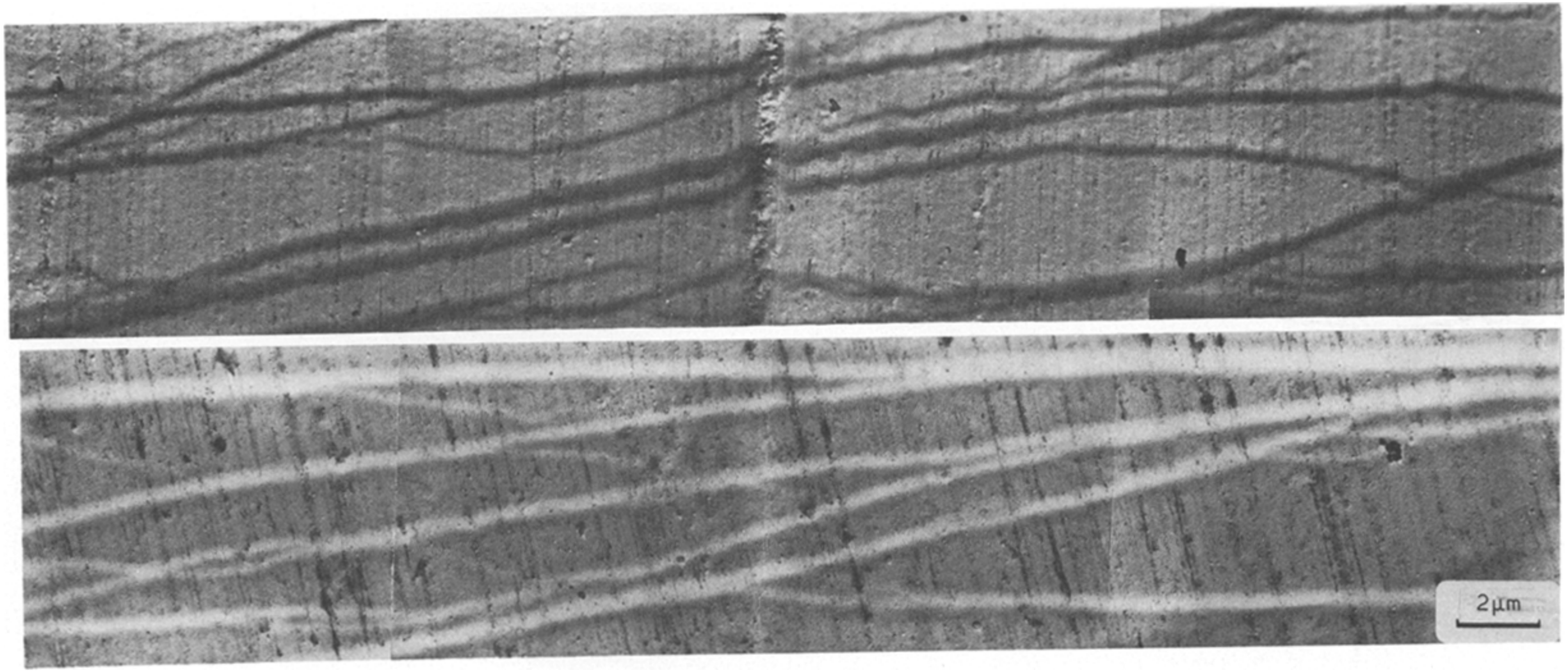


Figure 24 Another view of slip lines on the side surface (E.M.).

the splitting (or joined) band. The triangular cross-section of the splitting band changes as suggested in Figs. 7 and 9 during splitting. The joining process is the reverse of splitting.

(3) From 200 measurements, the distribution of shear strain of the coarse bands was determined to have a range between 1 and 2.9, with a most probable value of 1.8. However, the shear strain along the same band is almost a constant.

(4) The coarse bands can terminate in an otherwise feature-free region by reducing their shear strain to zero. They can also terminate at obstacles by converting to bundles of fine bands.

(5) A polished banded surface develops the original band pattern after annealing except that the bands are valleys rather than ridges. The shear strain in the band recovers upon annealing.

(6) The ridge of the coarse band is reduced in height upon annealing until it disappears. The thickness seems to remain constant during annealing.

(7) Annealing after the polishing of cut surfaces parallel to the shear direction reveals bands of matching patterns. These bands are valleys and displace scratches due to shear strain recovery.

(8) A polished banded surface also develops the original band pattern after further compression, except that the shear strain is very small. The bands are, however, still ridges.

(9) On the side surface which makes an angle to the shear direction, the coarse bands are wavy and are steps rather than ridges. Otherwise they split, join, and terminate freely just as the straight bands on the parallel surface.

(10) Since the coarse bands have very similar features to crystalline slip bands (straight on one surface and wavy on the other, steps, reappearance upon further deformation, free termination, etc.) their propagation can be viewed as the motion of macroscopic Volterra dislocations.

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