

Figure 5 a piece of the lamella two striations wide has been partially removed from the surface and folded back on itself. The overall length is just equal to the lamella thickness. In Figure 6, however, the "fiber" lying parallel to the lamella is more than twice as long as the corresponding lamella thickness. It remained attached to the lamella on the right side, the thicker portion corresponding to the width of the lamella at that point. At the end of the thick region it has split into "fibers." It is believed that these fibers are portions of the sheets, as is the piece in Figure 5. The fact that it is wider than the striations is probably due to its being cylindrically coated with platinum and carbon. The implications of the fact that it is longer than the lamella thickness is discussed in the last section of this paper. Similar fibers have also been observed on other micrographs of these samples to be several times as long as the thickness of the lamella to which they are attached; the most prevalent type of fiber or ribbon, however, is that whose length is equal to the corresponding lamella thickness. The fibers of low contrast on these two, and other figures, were formed when the Pt replica was stripped from the sample.

Many of the thicker type III lamellae have one or more kinks visible on their fracture surface. Usually, as in Figure 6, they extend from one side of the lamella to the other, the striations clearly changing direction at the kink. There is often a related change in the fracture plane. The change in level and slope of the fracture plane at the kinks makes it difficult to determine the angle through which the striations kink. It can be as large as  $90^\circ$  and often appears to be about that angle. Occasionally, instead of extending directly from one surface of the lamella to the other, the kink results in the formation of an isolated patch of displaced material, as to the right and above the partially removed piece of material in Figure 5, or it may reverse directions several times within the lamella. The latter structure may also be due to the presence of several intersecting kinks.

### Electron Diffraction Observations

Geil has shown<sup>7</sup> that it is often possible to remove portions of the polymer from the surface along with the replica by using the technique utilized for these preparations. Electron diffraction patterns from this material have been related to the single-crystal nature of the lamellae in polyethylene<sup>7</sup> and annealed polyoxymethylene.<sup>18</sup> It was also found, as mentioned previously, that considerable quantities of the polymer in samples E and F remained attached to the replicas. Some of the contrast of the striations and the fibers observed on the fracture surfaces are due to this polymer.

An electron diffraction pattern and corresponding bright field micrograph of the material adhering to a fracture surface of the lamella is shown in Figure 7. The diffraction patterns here and from other similar samples are fiber-like (on the original of Figure 7 both  $\{110\}$  and  $\{200\}$  reflections are visible) and of relatively low intensity. The length of the crystals is such that one would expect the  $\{002\}$  reflection to be visible. Even with longer

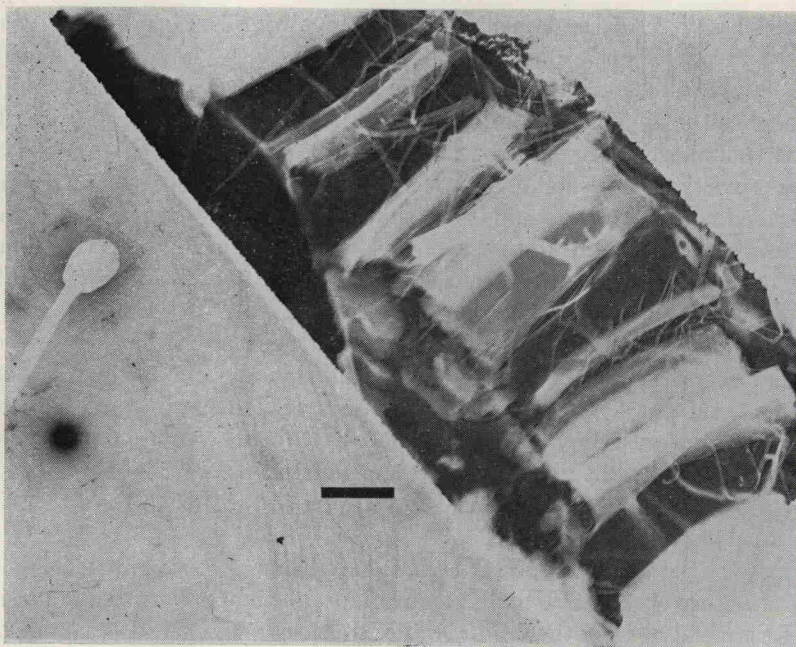


Fig. 7. Selected area electron diffraction patterns from the material adhering to the fracture surface of a type III lamella in sample E. The area giving rise to the patterns was recorded on the same plate as the pattern; the patterns should be rotated counter-clockwise about  $10^\circ$  to compensate for the rotation of the image in the microscope. This figure is printed as a negative.

exposure times and larger areas it was not possible to observe it. This may be due to a tilting of the lamella surface, however, and not to its absence. Likewise, the low intensity of the  $\{110\}$  and  $\{200\}$  reflections is probably due to the fact that only a few of the crystal's planes may be properly oriented to reflect. The alignment of the molecules parallel to the striations has been confirmed by observation of the optical birefringence of lamellae attached to the replica.\*

Good single-crystal diffraction patterns, sometimes with several orders of reflections and resembling those obtained from solution grown polyethylene single crystals, were obtained from regions in which an entire lamella remained attached to the replica (Fig. 8). Tilting of these lamellae with respect to the beam resulted in the type of pattern shown, the center of intensity of the pattern being displaced from the center of the pattern. Comparison of the selected area image that was taken at low intensity in conjunction with Figure 8 and which was unfortunately out of focus, and the subsequent micrograph shown in Figure 8, which was focused using an intensity sufficient to give a visible image on the microscope screen, indicates that some beam damage had occurred. However, the appearance of various regions of nearly uniform thickness is believed to be real.

\* The optical birefringence measurements were made by E. W. Fischer.