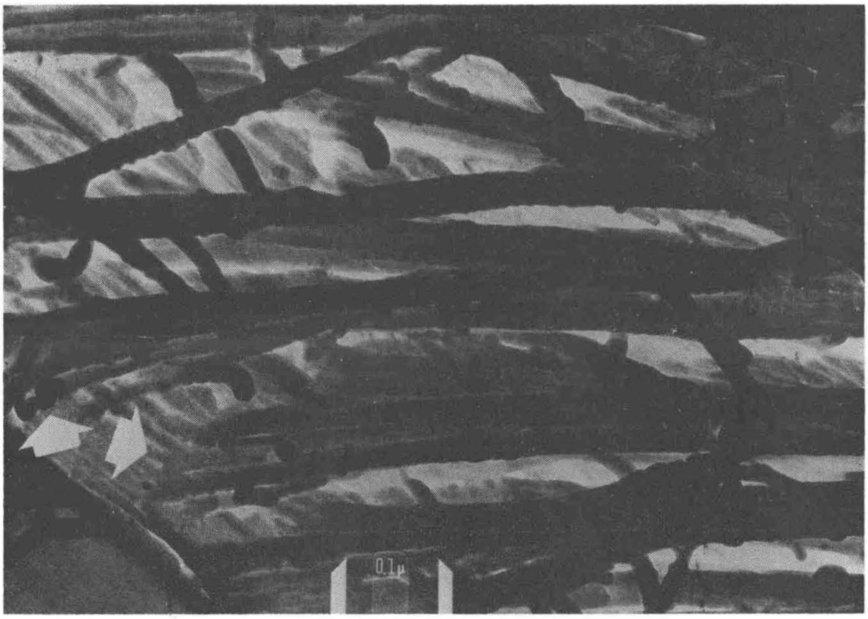

Fig. 7. Carbon replica showing inner core fiber bundles. Electron micrograph.

The ribbonlike fibrous texture of the inner core seen in the SEM photomicrographs was also observed in the shadowed carbon replicas. Electron micrographs showed that the ribbons extended for tens of microns and contained fine fiber bundles oriented parallel to the long axis of the strand. Figure 7 is a replication electron micrograph of these fiber bundles, in which parting of the individual fibers from the bundles during fracture replication is visible (see arrows). Note both the fiber lengths and apparently weak interfiber bonding. Individual fiber bundles in microtomed sections were examined by staining with Br_2 vapor, which preferentially attacks the less ordered regions between the component fibers. Specimens were mounted in an epoxy embedding material (Cargille NYSEM) and sectioned on a Sorvall Porter-Blum MT-2 ultramicrotome by use of a Dupont 43° diamond knife. Sectioning across a strand proved impractical since the fiber bundles readily splayed apart owing to the weak bonding between the fiber bundles. Sectioning parallel to the strand axis proved to be more feasible, but stringy, rather than smooth, sections were obtained with this procedure. Figure 8 is a transmission electron micrograph of microtomed fibers from the strand inner core stained with Br_2. The fibrous, stringy texture was quite visible; however, fiber splaying due to the action of the knife was also obvious. The ultimate subunit of the ribbon structure was found to be a 200–250 Å diameter fiber (see arrows in Fig. 8). Since individual fibers adhered to several of their neighbors after splaying apart, some type of interfiber bonding may have existed within a fiber bundle. A nodular structure superimposed on the ultimate fibers was also observed; however, this was probably an artifact of the Br_2 vapor staining process.

Fig. 8. Microtomed, Br_2-stained section from the inner core. Transmission electron micrograph.

Electron Diffraction

Unstained sectioned fibers and, in a few cases, shadowed fibers adhering to the carbon replicas were examined by electron diffraction using electron microscope stage tilting techniques. Exceptionally well-developed spot patterns were obtained from the strand inner core where the ribbon like structure was observed. Proper stage rotation revealed a highly ordered and oriented crystal structure within the ultimate fibers. Figures 9 and 10 show sharp electron diffraction patterns and the corresponding reciprocal lattice diagrams obtained from the inner core material. Note that the shorter exposure time of Figure 9 permitted observation of lower-order reflections, whereas the longer exposure time of Figure 10 provided the higher-order reflections. In both cases, $(0kl)$ reflections were observed while $(h0l)$ reflections were undetectable with further stage rotation. No explanation was found for the failure to detect the $(h0l)$ reflections, other than thickness effects associated with difficulty in passing electrons through the thick dimension of the ribbon. This would suggest that interfiber bonding in ribbons occurs primarily along $(0k0)$ planes.

The polyethylene crystal structure is based on an orthorhombic unit cell, requiring that the reciprocal and true lattice directions be parallel. Furthermore, polyethylene belongs to the D_{2h}^{16} space group. Such symmetry conditions require that permissible $(0kl)$ reflections satisfy the equation: $k + l = 2n$. Each of the twenty-six distinct reflections observable in