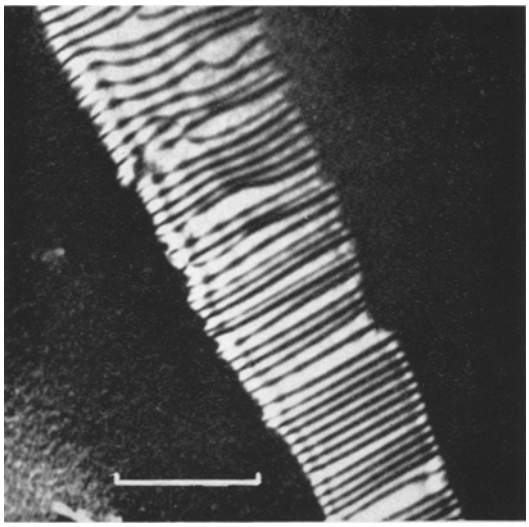

Figure 5 Low-angle boundary in a grain of an olivine rock. Scale line represents 1 μm. Dark-field image.

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Cross-Sectioning Techniques for Scanning Electron Microscopy

The usual methods of examining fibre cross sections with the scanning electron microscope (SEM) are not fully satisfactory. One relatively common cross-sectioning technique involves cutting fibre bundles, gluing them longitudinally to a sample stub, coating with a conductive layer, and tilting the specimen stage to obtain a view of the cut fibre ends. Alternatively, fibre bundles may be pulled through a specially designed stub, the exposed ends cut flush with the stub surface (using a razor blade), and finally, the mounted sample coated with a conductive layer. This technique is an adaptation of the Shirley plate method, commonly used in optical microscopy.

Both methods present imaging problems. Recently an improved technique was published by Anderson and Lipson [1]. These authors described a Hardy microtome method in which the fibres are forced into a slot, sectioned in an epoxy medium, left in the embedding matrix, glued to a sample stub, and coated with a conductive layer.

In our laboratory we have found the most advantageous techniques to be adaptations of the classical paraffin method [2, 3], which is well known to all textile microscopists. Depending on the hardness of the fibre and the cross section thickness desired, one may choose either paraffins of differing melting points and hardnesses, or a mixture of a paraffin and a matrix-hardening additive (i.e. carnauba wax). Our adaptation of the classical paraffin method for preparing SEM cross section samples simply involves embedding the yarns, cutting serial sections, and mounting them onto glass slides with Mayer's albumen. The slides are then washed twice with xylene to remove the embedding matrix. Finally, the slides are washed twice with ethanol (to set the albumen) and allowed to dry. Slides prepared in this manner may then be scanned with a light microscope to select sections for SEM examinations. Those sections to be studied are split from the glass slide with a glass cutter. (While it is not critical, a convenient fragment size is approximately 5×10 mm). The glass segment is mounted onto a sample stub with Duco cement, coated with a conductive layer, and placed in the SEM. This method is handy, since by using multiple embedding fixtures [4], a number of yarns may be embedded, sectioned, and processed simultaneously. This permits the mounting of a number of yarns on the same SEM sample stub. Therefore, direct comparisons between yarn samples are facilitated.

An alternative method we have found to work well, particularly when attempting to match the

embedding matrix hardness to that of the fibre, is the use of methyl methacrylate in combination with various ratios of dibutyl phthalate. Benzoyl peroxide is used to catalyse polymerisation of the methacrylate. If the fibre is soluble in methyl methacrylate monomer. the methyl methacrylate/dibutyl phthalate mixture must be prepolymerised [5] to a rather viscous consistency prior to embedding the fibre and subsequently completing the polymerisation. Gelatin capsules or Beem capsules* may be used as embedding containers. There are many variations of yarn holders which may be used to embed yarns.

After the polymerisation step is completed, sectioning is performed. In this method the sections usually come off one at a time instead of in a "ribbon", as occurs with paraffin. However, the methyl methacrylate/dibutyl phthalate sections can easily be lifted from the microtome knife edge and placed on glass slides coated with Mayer's albumen, and then processed in the same way as the paraffin sections.

We routinely section with an American Optical 820 rotary microtome equipped with steel knives. Obviously, section quality can be improved significantly by using glass knives; or if an ultra-microtome is available, by sectioning with diamond knives. Also, rather than using glass slides, sections could be mounted on 12 mm (or less) diameter cover slips. These could be mounted whole on the sample stubs, eliminating the necessity of splitting fragments from glass slides.

The techniques described above offer several significant advantages. First, they are simple extensions of well established, proven procedures. As such, they require no unusual microtomy skills. Anyone who has mastered the preparation of sections for light microscopy can readily use our adaptations to prepare good sections for SEM examinations. Secondly, these techniques provide the distinct advantage of being able to study both the surface and the internal morphology of the same fibre element from any angle simply by tilting the stage from 0 to 90°, or by rotating through 360°. There is no "dark side" of the fibre, as is true of replicas or fibres directly adhered to SEM stubs.

Mounting on glass slides provides the advantage of examining cross sections by both normal light microscopical methods (polarised light, interference, etc.) and then removing the immersion oil and examining the same sections with the SEM. The combination of physical optical parameters with SEM morphological studies on serial sections provides useful correla-

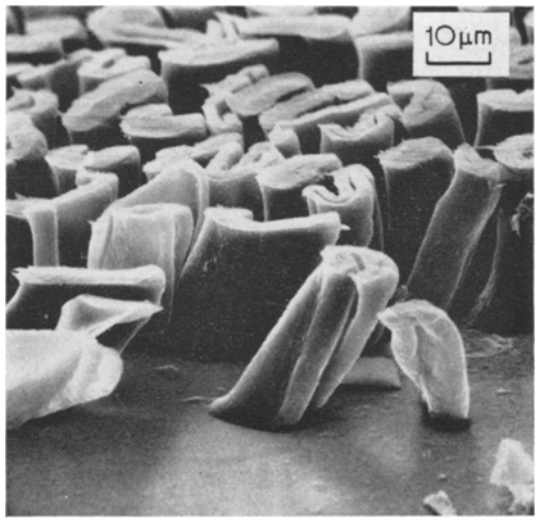

Figure 1 A cross section (25 μm thick) of cotton yarn. A beneficial aspect of the method is that yarn integrity can be maintained in the sectioning method. Utilising the depth of focus of the SEM, yarn twist and its effects on individual filaments can be studied. Observe the varying inclination angles of the filaments reflecting yarn twist and the changes in individual fibre convolutions within the short length of 25 μm.

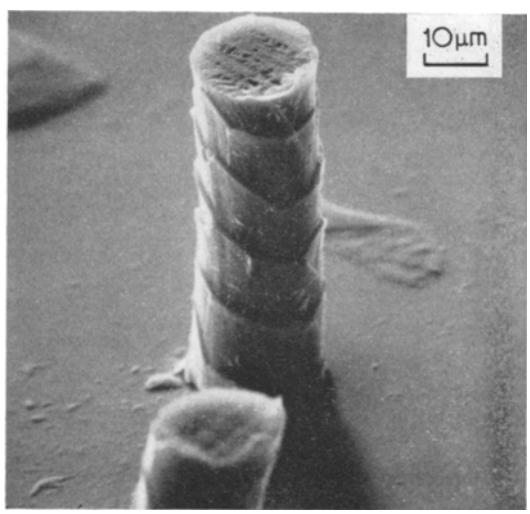

Figure 2 A cross section of wool (40 μm thick). On a thick section such as this, a detailed examination of surface scales and internal structure can be performed at the same time. This points out the advantage of having free-standing sections for SEM viewing.

*May be obtained from Ladd Research Industries, Inc., Burlington, Vermont.

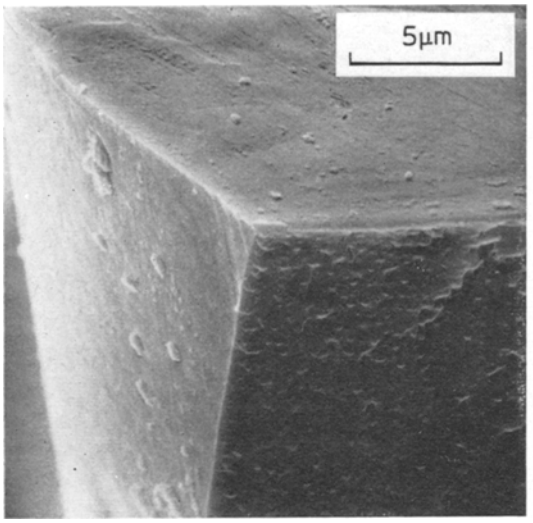

Figure 3 A cross section/longitudinal section of cold-drawn polyester fibre (25 μm thick). This combination of sectioning and SEM is unique in providing the only known method of studying fibre structure by means of simultaneously examining surface, cross-sectional, and longitudinal structures. The technique can be greatly enhanced by ion or chemical etching to reveal structural elements.

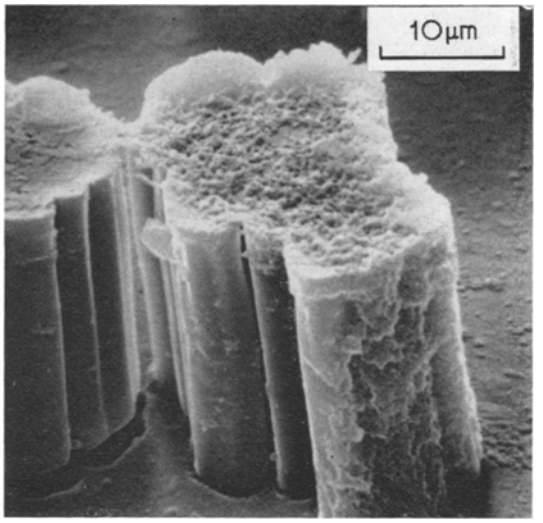

Figure 5 A cross section of CTA fibre (25 μm thick). The cross section was saponified for 30 sec and the saponified layer removed with cupriethylene diamine/water according to a previously described method [7]. This exemplifies how the technique may be used to study chemical effects on fibre morphology, or fibre structure effects on chemical reactivity. The interior is heavily etched, as is one side of the fibre, while the remainder of the surface appears unaffected. Shorter saponification times affect only the interior, and the surface not at all. This supports previous work [7] contending that there is a cuticle layer having properties differing from the internal portion of triacetate fibres.

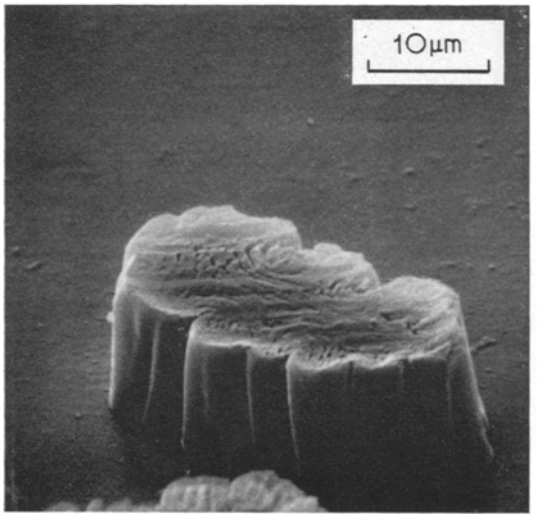

Figure 4 A cross section of cellulose triacetate (CTA) fibre (20 μm thick). The lobular shape, relatively smooth surface texture, and the hint of a skin-core texture are visible. Knife marks can be seen running parallel to the bottom edge of the figure.

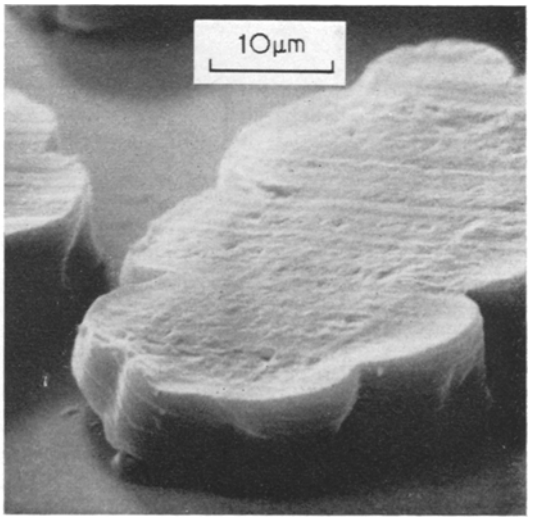

Figure 6 A cross section of CTA fibre (5 μm thick). Here the surface convolutions, surface microwrinkles, and a definite skin-core texture can be seen.

tive data. Examples of areas for investigation are skin-core structures, void textures, radial heterogeneities, variation of structures in length increments through necked regions of thermoplastic fibres, and in general, the effects of chemical and mechanical treatments on fibre morphology.

Stereoscopical investigation is a useful adjunct to the SEM examination of sections. Another dimension is added to the investigation mentioned previously by longitudinally sectioning cross sections [6]. Surface, radial, and tangential sections of the same fibre element can then be studied simultaneously. Additionally, the quality of sections prepared for light microscopy can be studied. It is quickly seen that what generally appear to be excellent light microscopy cross sections in reality suffer from knife marks, irregular thicknesses, and other defects. This observation helps prevent the pitfall of assigning structural interpretations to what are, in reality, cutting artifacts. This is particularly applicable to phase and interference microscopy. Examples of applications are given in the figures below. In all examples, the accelerating potential on our Cambridge Stereoscan SEM was 20 kV, the beam angle was 60°, and the samples were coated with 60% gold/40% palladium alloy.

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