Part I
Introductory review

1

SINGLE FIBRE FAILURE

FIBRES IN FABRICS

The immediate appeal of a textile fabric lies in its comfort, style and warmth, but ranking equally with these as a measure of quality and value is its durability. How soon will the appearance of the material deteriorate with use? — or, in some perverse instances like blue jeans, become more attractive? How long will it be before the fabric becomes so full of holes or so thin that the garment has to be discarded? The consumer is only concerned with a practical reaction to such questions, but the textile technologist can see that these changes result from the breakdown of the fibres in the fabric.

Textiles are not used only in the traditional clothing and household uses: they have been used for thousands of years in some engineering applications such as ropes, sails, containers and covers. Following the Industrial Revolution, there came a new range of products like conveyor belts, drive belts, filter fabrics and tyres. Today, partly due to a new generation of high-performance fibres such as carbon and Kevlar, an even wider range of advanced engineering applications is opening up: composites, artificial arteries and components of space vehicles are just three examples from a long list. In most of these industrial uses, strength is a major design criterion, and it depends on the resistance of fibres to failure under the imposed combinations of stress. Sometimes thermal resistance is needed. After these initial criteria are satisfied, avoidance of structural fatigue leading to premature failure becomes another design necessity.

There are two possible approaches to design for product performance. The first, which has proved practical and successful, is the craft route of a combination of knowledge and experience applied qualitatively to the selection of raw material and fabric construction, followed by trials and revision if necessary. The second way is engineering design, with mathematical calculation of predicted performance. However, the problems are difficult because textile materials are complicated structures. Nevertheless, it is becoming necessary to move to this approach because of the increasing demands on products and the increasing range of choices. Fig. 1.1 shows what has to be done in basic research and application studies before engineering design can be applied to the strength, and, more important, the life of textile products. This book is concerned with one aspect of the basic research: the study of how fibres fail under stress.

Even with the craft approach, the importance of fibre strength was recognized, and measurement of strength is one of the tests always used to provide entries on a fibre data sheet. But until comparatively recently little was known about the way in which fibres break. The introduction of the scanning electron microscope (SEM), which became commercially available in 1965, opened up the subject.

FIBRE FRACTOGRAPHY

In bulk materials like metals and plastics, the form of fracture can be seen with the naked eye on a large test piece, and a great deal of detail can be observed with light microscopes. Such studies became the science of fractography. For these materials, the SEM was a useful new technique for examining the detail, although the study of shadowed replicas in the transmission electron microscope (TEM) already provided some similar information, though less easily.

With fibres, it was different. It was not until the SEM was in use that it became possible for the fibre scientist to be shown the general form of fracture, let alone the detail. The reason is that fibres are only a few micrometres in diameter, so that to the naked eye a broken fibre is nothing more than a line with an end.

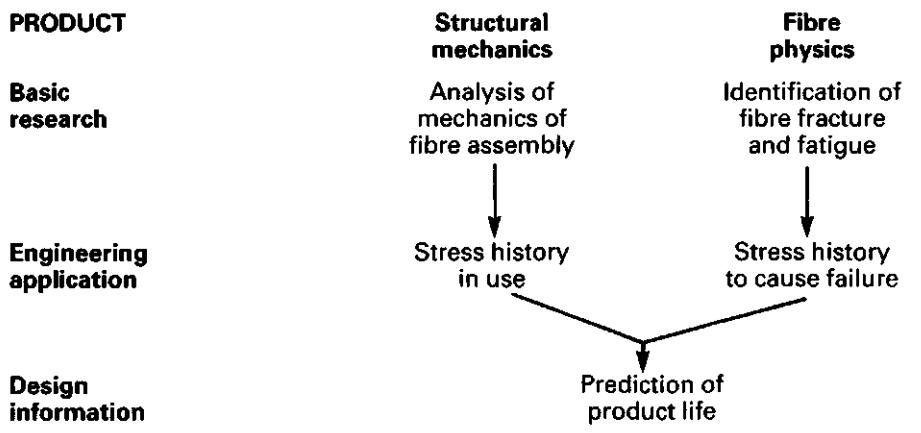


Fig. 1.1 — Past and future design procedures for textile products.

There had been some earlier studies by optical microscopy; but worthwhile results could only be obtained by a dedicated microscopist, such as Gladys Clegg of the Shirley Institute, who devoted long hours of painstaking work to produce beautiful drawings of cotton after swelling, staining and mounting of fibres removed from yarns. There was no way in which a broken fibre could be put directly in the microscope and a picture taken to provide the information needed by an investigator whose principal interest was in the mechanics, and not in the microscopy. The difficulty is not so much the limited resolution of the optical microscope, but is more the lack of depth of focus and the difficulty of obtaining contrast in order to show up clearly the complex shape of a broken fibre end. Furthermore, the problem was not only the great difficulty of establishing reasonable viewing conditions and interpreting what was seen, but that it was easy to be misled.

Transmission electron microscopy was of little help because it was not possible to obtain replicas of complicated fibre breaks with deep re-entrant cavities or multiple splitting. Even the simplest forms of fibre ends would be too three-dimensional to be studied. Surface damage could be examined by replication; and in one study in the 1950s, John Chapman showed frictional wear very clearly by using a conventional electron microscope to observe a fibre directly in the rarely employed reflection mode. However, this is only possible with the electrons directed at a glancing angle, and leads to a strongly foreshortened image.

The available techniques of optical and conventional electron microscopy were thus of very limited value in studying fibre breaks.

The SEM changed the scene, and it became possible for the first time to look at a picture of a broken fibre, in much the same way as one would look at an ordinary photograph of a broken metal bar.

The reasons for this are: (1) the specimens are not transparent to electrons, so that the image formed from the scattered electrons is similar to that seen on looking at a solid opaque object; (2) the great depth of focus means that the whole fibre end is in focus; (3) the usual mode of use of an SEM gives an image which appears to be lit from the side, and this shows up the three-dimensional character very clearly, and only rarely with ambiguity. It is possible to make stereo pairs to give a true three-dimensional image and show depth more clearly within the specimen, but we have not found this to be necessary in our studies of fibre breaks.

Usually, there is no problem in seeing the general form of a break, although sometimes, when there is axial splitting, it may be necessary to take several pictures along the break and join them up to form a montage, in order to give an overall view of the break at a suitable magnification. Finer detail within a break can be seen at high magnification, up to the practical limit of resolution. Instrumentally, the limit is given by the electron beam spot size, and manufacturers now claim 3.5 nm or less; but, in practice, resolution with organic materials is limited by the extent of spreading of the beam as it penetrates into the specimen, and for routine examinations in our SEM is generally about 15 nm. Most fibres are better examined at relatively low beam voltages (between 5 and 10 kV) in order to reduce the penetration and spreading of the electrons, whereas microscopists working with metals, which give a stronger resistance to penetration because of their greater density and atomic number, usually choose a higher voltage (20 kV or more) in order to reduce spot size. The use of a lower voltage also limits loss of surface detail in the image resulting from excessive depth of penetration within the sample.

Most fibres are electrical insulators and therefore charge up in the electron beam. The problems of charging are usually overcome by coating the specimen with metal, though care must be taken that too thick a coating does not mask or distort features of the fracture. Again, the problems are reduced when a lower voltage is used.

TECHNIQUES OF EXAMINATION

The SEM used at UMIST at the time of publication is shown in Fig. 1.2. Electrons are generated by the electron gun at the top of the column, and form a beam, which passes down the column through electromagnetic lenses that control the size of the beam. The final lens focuses the beam on to the specimen surface. The focused spot of electrons is not stationary, since the final lens includes scanning coils, which deflect the beam in a square raster, across a selected area of the surface of the specimen. Electrons have very short wavelengths and are easily deflected and absorbed by other materials and gases, and therefore the gun, column and specimen chamber have to be under vacuum, when the SEM is operating.

The specimens are mounted on special holders, which are fitted on to the stage of the microscope within the specimen chamber. The type of holder depends on the make of the SEM. In our SEM solid aluminium stubs of the type shown later in Fig. 2.3(a,b) are standard; they are 15 mm and 32 mm in diameter. However, the shape and size can vary between makes of SEM, and it should be noted that much larger holders, up to 150 mm in diameter, and maybe larger, can be fitted into the specimen chamber. The choice of holder depends entirely on the type of sample to be examined and on the specimen size limitation of the particular SEM used.

The image formation results from the collection of electrons emitted from the specimen by an electron collector situated to the side of the stage. The collected electrons provide a signal which is amplified and presented on a CRT (cathode ray tube) similar to a TV monitor. The CRT screen is scanned in synchronism with the electron beam scanning over the specimen surface, and the magnification is given by the ratio of screen area to the area of the surface scan. The view of the specimen appears as if the user was looking along the same line as the electron beam, but with the illumination offset in the direction of the electron collector. The particular SEM design and choice of operating conditions influence the image seen.

In order to examine broken fibre ends properly, they must be able to be viewed from all angles, by using the tilt and rotation facilities on the stage of the SEM. This means that single fibres must be held upright, projecting from the stub. A convenient way to do this is to sandwich the fibres between two layers of adhesive copper tape with the broken ends protruding, and then to grip the sandwich in a specially designed split stub. Fig. 1.3 shows a photograph of such an arrangement. If the fibres are fine and straight, about ten fibres (five paired ends) can be mounted on a 15-mm diameter stub, leaving sufficient space between each fibre for clear viewing of the fracture surface; but with crimped, coiled or very thick fibres, the number may be fewer. Coarse monofils can project several millimetres from the edge of the tape, but with fine fibres the distance must be minimized in order to avoid the fibres being bent over or caused to move by the electron beam. However, it is useful to be able to see the fibre surface away from the fracture region, since this can be a source of information on the form of

Fig. 1.2 — A scanning electron microscope.

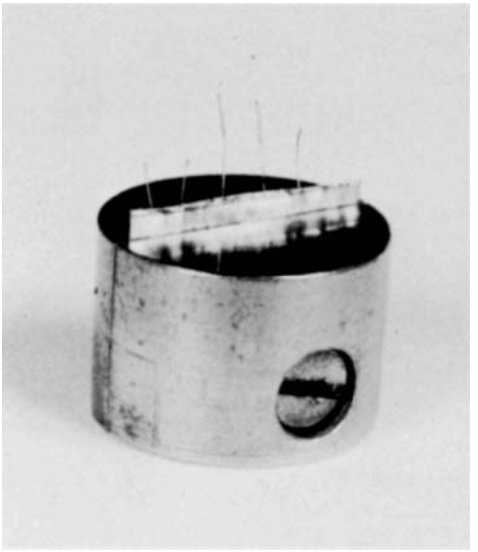


Fig. 1.3 — Fibres mounted on a stub for examination in SEM.

damage. Furthermore, it is fairly common for the effects of fracture to spread some distance along a fibre, either as axial splits, multiple transverse cracks or a generally ragged break. Compromise, and skill on the part of the microscopist, are thus needed.

Most textile fibres are organic polymers and are poor conductors of electricity. Even moisture-absorbing cellulose fibres, such as cotton and rayon, which are moderate conductors in an ordinary atmosphere, become insulators when dried out in the vacuum chamber of the SEM. They are therefore prone to charging by the electron beam. This phenomenon is caused by the fact that electrons do not have a direct path to earth, and, as a result, they remain on the specimen surface, building up electron charge as the electron beam continuously scans the specimen. Charging causes excessive contrast, flaring, banding, streaking and sometimes even image distortion. Charging phenomena are complex and not fully understood, but some materials are worse than others: resin-treated cotton, viscose rayon and wool are particularly bad.

In order to overcome charging problems, the fibres are usually coated with a thin conducting layer after being mounted on the stub. It is necessary to have a coating over all parts of what may be a complicated broken surface and to have a continuous conducting path from the fibre end to the copper tape, and so to earth through the stub. Carbon or various metals can be used for coating, but we find gold to be most suitable for our needs. At first, evaporation of metal in vacuum coating equipment was employed; but now it is more common to use the method of sputter-coating, in which gold atoms are liberated by bombardment of the target by ionized molecules of an inert gas, usually argon. The gold atoms are scattered by the gas molecules, with many eventually settling on the specimen. This scattering of gold atoms ensures a good coverage of the specimen surface. A typical set-up is shown in Fig. 1.4.

The main function of the gold coating is to make the surface of the specimen electrically conducting, in order to prevent charging. The good electron emission property of gold increases the number of electrons emerging from the surface of the specimen, and its density limits to some extent electron penetration and spreading of the spot, thus enhancing the image quality. These advantages must be balanced against the danger of obscuring or falsifying the appearance of the fibre surface details by too thick a coating.

Even in a thinly coated specimen, charging, beam damage and penetration can be prevented or reduced by careful choice of SEM operating conditions. The best conditions must be found by experiment with the particular microscope being used. For our particular requirements, we have found values from 5 kV up to 11 kV to be satisfactory.

Provided care is taken, the above procedures give a good image of textile fibres, without information being seriously lost or changed by artefacts of electron/specimen interaction, the presence of a coating or drying out of the specimen. However, there may be some circumstances, such as the examination of the development of cracks in a fibre being strained in the microscope, where coating is undesirable, or drying changes the situation. Charging may be reduced in these circumstances by treating the fibres with antistatic agents, or by employing special environmental mounts which release water vapour in the immediate vicinity of the specimen. Artefacts due to drying can be reduced by the special environmental mounts or by cryogenic stages, which enable the specimen to be examined in a frozen state.

There are other means of reducing charging effects and of examining uncoated specimens. The normal practice in collecting electrons from the surface of the specimen is to use a secondary electron detector, which collects electrons of various energy levels, but the bulk of the signal is composed of low-energy secondary electrons. It is these low-energy electrons

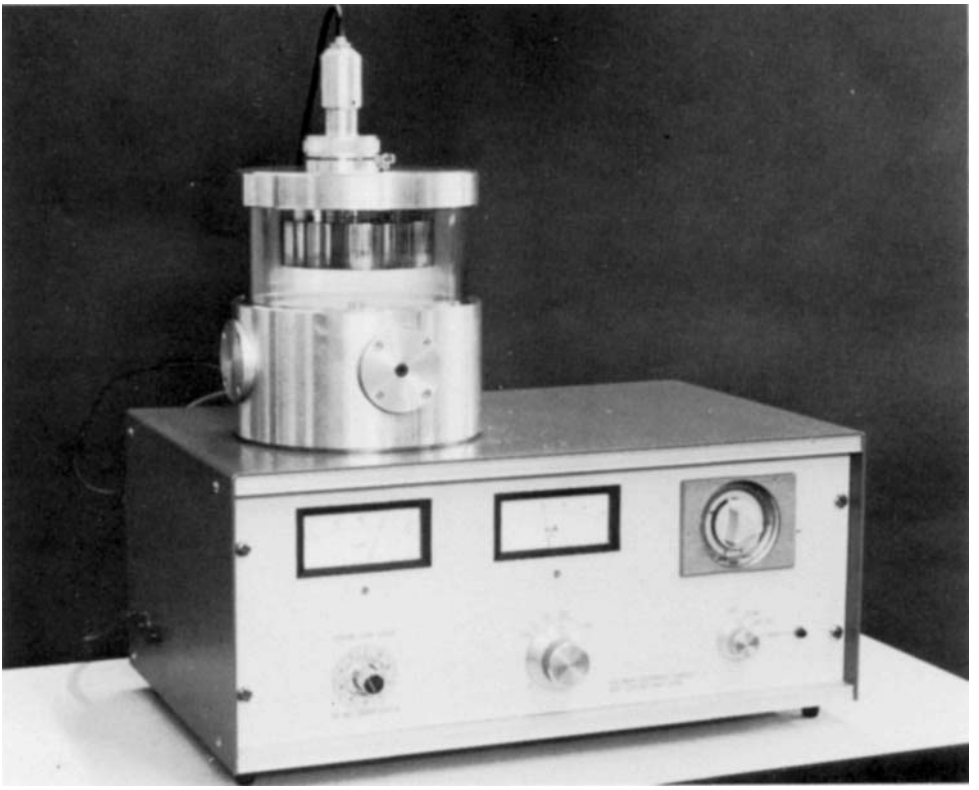


Fig. 1.4 — Sputter-coating equipment.

which are affected by charging phenomena. High-energy electrons, normally termed backscattered electrons, are far less influenced by charging problems, and there are now commercially available backscattered detectors of two types, namely the scintillator and the solid-state backscattered detectors. These detectors are very useful in examining poorly conducting or uncoated specimens, which may otherwise have charging problems. They often give a better topographical image, and also show atomic number contrast. The latter is useful in the examination of metals and other situations in which atomic number difference is seen.

If a backscattered detector is not available and metallic coating of the specimen is undesirable, then the specimen should be examined at low accelerating potentials, say 5 kV and lower, in order to reduce specimen charging effects and beam damage. Due to the potentially poor signal-to-noise ratio of the image at low voltages, slow scan rates are usually used, and this in turn may exacerbate problems of charging and beam damage.

The latest development in overcoming these problems is the use of digital image/frame store systems. This technique yields clear images from normal output signals from the SEM. Images can be collected rapidly at scan rates from below 5 MHz up to 10 MHz (television rates), and then stored and processed as required. Consecutive frame scans can be continuously integrated, and this gives a dramatic reduction in noise level in the final displayed image. One or more processed images can be stored and recorded. The processed image can be further enhanced by the sharpening of vertical edges, or by expansion of selected parts of the image grey-scales. It can be displayed in monochrome or pseudo-colour.

Digital image processing has two main advantages over the conventional system when examining uncoated, insulator-type samples: (a) the specimen can be scanned at fast rates, including TV rates; (b) whilst the image is being processed, the electron beam can be switched off so that the specimen is no longer exposed to electrons. Both of these features reduce the possibility of specimen charging and beam damage. Digital image processors and frame store systems are offered as add-on accessories for retro-fitting to SEMs. They are also beginning to be supplied as standard items with new SEMs, and can be used with video-printers that produce high-resolution, hard-copy prints of the processed image.

It is not possible, or appropriate, to provide a complete course of instruction in the scanning electron microscopy of fibres in this book. Further information can be found in the references listed in the bibliography at the end of the book, or, better still, through training in a good laboratory experienced in dealing with fibres, or from specialist training courses.

CLASSIFICATION OF BREAKS

Lord Rutherford once said that science was either 'stamp-collecting or mathematics'. The early years of fibre fracture studies have been 'stamp-collecting': observing the different forms

of failure and classifying them into categories. We have now identified 18 distinctly different categories of break and other fibre ends, illustrated in Fig. 1.5 and discussed in greater detail in later chapters of this book. This classification is based on a pragmatic combination of visual form of the break, macroscopic cause and structural mechanism.

Types 1–6 were found in laboratory tensile tests on different sorts of fibre. But our concurrent studies on worn textile materials rarely showed similar breaks. This is not surprising because textiles in use do not usually fail through the application of a single excessive load: they break down after repeated small or moderate loading over a long period of time.

We were therefore led to the study of fatigue testing in the laboratory, with the development of new instruments and test methods. Types 8–12 were distinct forms found with different ways in which repeated stresses can be applied to fibres. The principal methods used have been: (1) tensile fatigue, namely application of cyclic axial stresses on a fibre; (2) flex fatigue by pulling a fibre backwards and forwards over a pin; (3) biaxial rotation fatigue by rotating a bent fibre over a pin so that the material alternates between tension and compression; and (4) surface abrasion. The differences in the way in which the breaks occur illustrate clearly the need to be specific in characterizing fibre fatigue, and to make comparisons between fibres only on the basis of a well-defined test method.

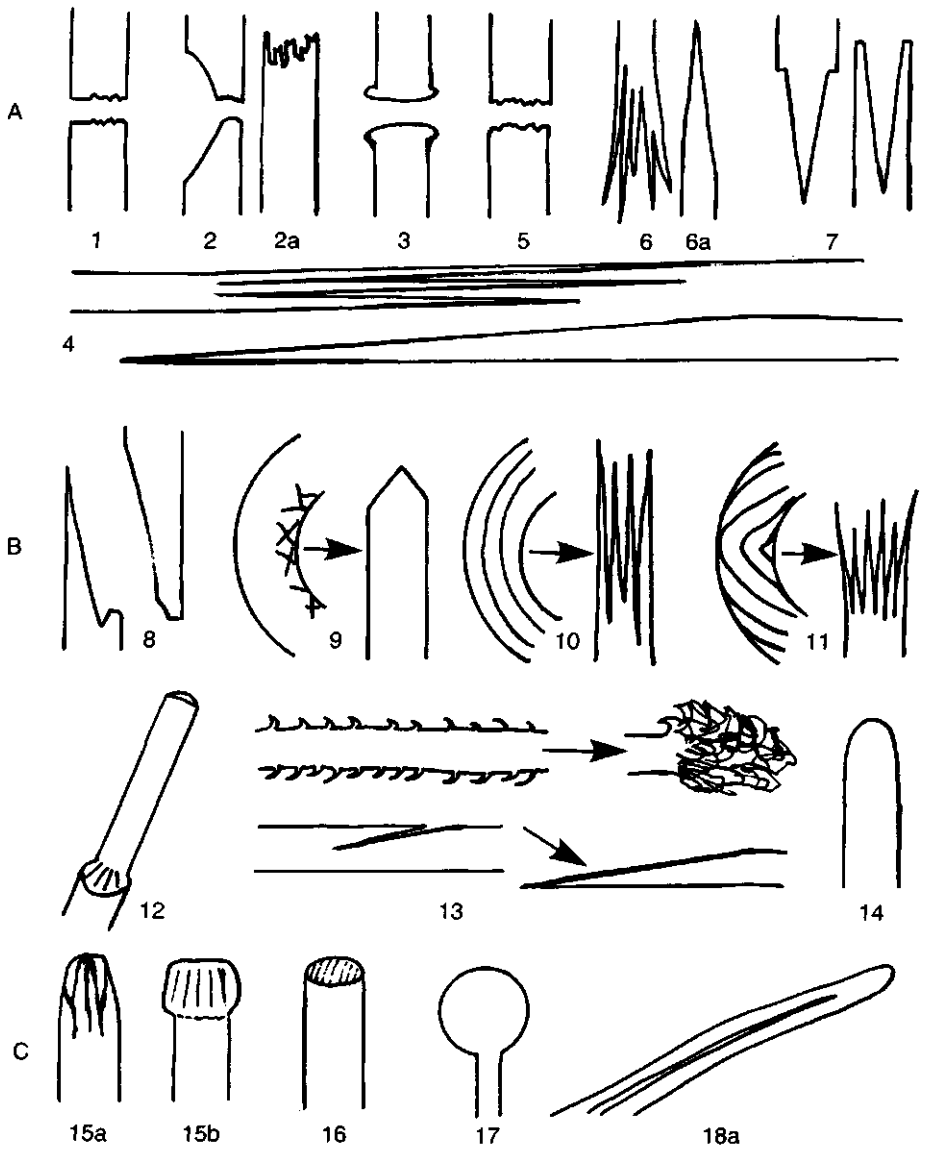


Fig. 1.5 — Forms of fibre break and other fibre ends. A — Tensile failures: (1) brittle fracture; (2) ductile fracture, (2a) variant, light-degraded nylon; (3) high-speed, melt-spun fibre; (4) axial splits; (5) granular; (6) independent fibrillar, (6a) collapsed; (7) stake and socket. B — Fatigue: (8) tensile; (9) flex kinkband; (10) flex split; (11) biaxial rotation, bend and twist; (12) surface wear; (13) peeling and splitting, alternative forms; (14) rounding. C — Other forms: (15) transverse pressure, (15a) mangled, (15b) localized; (16) sharp cut; (17) melted; (18) natural fibre ends, e.g. (18a) tip of cotton fibre.

Type 13 includes several forms of break associated with splitting and peeling due to cyclic shear stresses: this category may need to be subdivided, but the forms are not yet clearly differentiated, and, in some instances, have been studied more in relation to failures in use than to laboratory tests. Type 14 is a rounding of the end of a fibre, which developed after further wear of a fibre which had broken in use.

Type 7 is a tensile failure which has only been found after chemical attack on a fibre. Type 15 results from severe lateral pressure by crushing or blunt cutting, whereas type 16 is from a sharp cut. Type 17 is a form of melting. Finally there are natural fibre ends, type 18.

FRACTURE MECHANICS AND POLYMER PHYSICS

Fracture mechanics is the 'mathematics' of Lord Rutherford's statement. The foundations were laid in 1921 with the classic work of A. A. Griffith on brittle fracture, when he investigated the association of fracture with flaws, either on the surface or internal, which led to stress concentrations. The mechanics can be analysed in two different ways.

In what may seem to be the most direct approach, stress analysis is used to find the stress concentration which is then compared with a material property, namely its inherent strength. The difficulties with this approach are that the stress analysis is complicated and the inherent strength is difficult to measure or calculate. Griffith proposed an energy criterion, with the condition for crack propagation leading to fracture being $dE_m > dS_c$, where dE_m is the elastic energy released in the material when the crack advances, and dS_c is the surface energy of the newly formed crack surfaces. For a crack of unit width advancing a distance dx, the criterion can be given in terms of the elastic energy per unit width E and the surface energy per unit area S:

$$\frac{dE}{dx} > S \tag{1.1}$$

The simple Griffith theory applies only to purely elastic brittle materials, like glass, and is not valid when there is plastic deformation which also absorbs energy and blunts the crack. However, in metals and other materials in which the plastic deformation is limited to a small zone close to the crack tip, equation (1.1) can be modified, by redefining S to include the energy of plastic flow at the crack tip, as well as the surface energy of crack formation.

The zone of plastic deformation will be so small that it has a negligible effect on the elastic energy which is associated with the main bulk of the test specimen. On this basis, fracture mechanics has developed considerable mathematical complexity and power as a means of predicting the conditions for failure of metal structures. These treatments also usually assume small strains and isotropic and ideal elastic–plastic mechanical behaviour. A recent account of the subject is the treatise by Atkins and Mai (1985). The application of fracture mechanics to polymers has been covered by Williams (1983).

For most fibres the situation is more complicated because their structure is highly anisotropic, the stress–strain curves have a more complex non-linearity, deformation is viscoelastic and viscoplastic, and strains are large. Furthermore, the zones of plastic deformation are not restricted to small regions near the crack tip, but often extend over distances greater than the crack depth and may include the whole specimen. These are conditions which have not yet been properly analysed in studies of fracture mechanics.

Most of the explanations of fibre fracture which have been given up to now and are included in this book have been purely qualitative accounts of the sequence of events, with some indications of what is happening to the material structure. However, this is a necessary prerequisite for more advanced work. The observation and classification of the forms of failure provide a challenge for research by theoreticians into fibre fracture mechanics, which combines interesting problems in the applied mechanics of stress and strain with the polymer physics of the material response.

PRACTICAL APPLICATIONS

The development of engineering design procedures, incorporating fibre failure, is a long-term development, perhaps aimed more at the twenty-first century than the twentieth. But there are immediate practical applications of the study of fibre failure.

Even a qualitative understanding of the way in which fibres are breaking down in particular applications can be a great help to the thinking of the engineer, whether concerned with improving fibre properties or with assembling them in ways which will minimize the stresses which cause damage.

More directly, SEM studies of fibre breaks are a tool in the pathology of product failure. For example, the pictures shown in **34G** provided an important clue to the discovery of a particular source of environmental damage to work clothing. Needs also arise in connection with consumer complaints and, even more important, with product liability litigation. For instance, after an accident, it may be necessary to establish whether a component has broken due to a design fault by the manufacturer, or due to misuse by the consumer, or indeed whether it has been cut after the accident. In a very specific area, such as the work on

automobile seat belts reported in Chapter 37, comparative studies can be made on the particular product; but, for more general application, there is a need to know how individual fibres fail under different conditions. This can only be based on laboratory testing and the examination of single fibre breaks, which constitute Parts II, III and IV of this book. These individual failures can then be related to the study of wear and damage in products by the techniques which are given in the next chapter and provide the information in Parts V, VI and VII.

ADVANCES IN TECHNIQUES

The SEM techniques for examining the forms of damage and failures in fibres and textiles, as described in the first two chapters of this book, remain essentially unchanged. The most notable advance in SEM technology is a by-product of the digital information revolution. Instead of recording images on photographic film, they can now be digitally stored in a PC with adequate memory. The size of each picture file occupies most of a floppy disk, but easily fits on to a hard disk. Off-line storage of pictures and ancillary information is on CD-ROM. Prints that are adequate for most purposes can be made on an ink-jet or laser printer, as used for printing text. Special laser printers and paper are used for higher quality prints. These changes greatly improve the speed and convenience of obtaining and keeping information.

Other advances are related to more specialised investigations and not just the simple and clear observation of the essential morphology of failure. Environmental scanning electron microscopes allow specimens to be observed in conditions other than a room temperature vacuum. This means that a humid environment is possible, which avoids any changes due to drying of fibres and eliminates the need for coating by providing electrical conductivity in the sample. One application is to observe specimens of fibre or textile as they are being deformed in the SEM. This is illustrated in Chapter 23 by the work of Johnson and Gharehaghaji on the development of damage in wool fibres as they are pulled against wire or pins of the type used in opening machinery.

Coating can also be avoided by the use of newer scanning electron microscopes with field emission electron guns, such as the Hitachi model S-4100 used for **1B(2),(3),(5),(6)**, which are discussed later in comparison with AFM observations. The high power of a modern FESEM gives a high current with small spot size at low beam voltage, so that uncoated samples can be studied at 1 kV with a resolution up to 8 nm, Phillips *et al* (1995). Other examples of the use of an advanced SEM are shown in **8G**. Since these were studies of carbon fibres, electrostatic charging was not a problem and the ultra high resolution available at a higher beam voltage could be used. The many ways in which such a powerful instrument, with a spot size down to less than 0.5 nm at 30 kV, can be used for imaging and chemical microanalysis are described by Boyes (1994).

Transmission electron microscopy (TEM) can be used to study the finer details of crack development as a prelude to failure or other damaging features. Studies reported by Davis (1989), who discusses the problems of difficulty of sectioning fibres without damage and of their low electron density contrast, are shown in **1A(1)–(3)**. An internal crack in a polyester fibre, due to some unknown cause is seen in **1A(1)**. This picture was taken using a negative staining technique, which supports the fibre material during sectioning and increases contrast; the method was developed 20 years earlier, Billica *et al* (1970). Extensive delamination in a stretched polyester film is shown in **1A(2)**. The cracks are crossed by fibrils. Macro- and micro-fibrils are also seen in the replica of the internal splitting which occurs when a strip is peeled off a polyester fibre, **1A(3)**. Such observations are useful in increasing understanding of fracture mechanics and its relation to fibre structure. These three examples all suggest that cracks develop along internal structural boundaries.

Hagege, as reported by Oudet *et al* (1984), has obtained additional information by the use of TEM and electron diffraction in the examination of polyester fibres subject to tensile fatigue, which gives failure of the type shown in **11C**. In this type of fatigue, an initial transverse crack turns and runs down the fibre almost parallel to the fibre axis. A TEM picture of an oblique longitudinal section through such a crack is shown in **1A(4)**. Valuable additional information on the way the crack grows is obtained from a higher magnification view of the crack tip, which is found to be preceded by micropores, **1A(5)**. An oblique transverse section containing a crack tip is shown in **1A(6)**. Away from the crack, the electron diffraction images show patterns which are characteristic of a semi-crystalline material, but in the images near to the crack tip the crystalline arcs are missing, which suggests that the material is amorphous. The reduction in crystallinity was confirmed by infra-red spectroscopy and X-ray diffraction.

Atomic force microscopy (AFM) is a technique which has become available since the first edition of this book was published. A fine probe is scanned over the surface and topographical, mechanical or other information is recorded to produce an image of the material surface with an optimum resolution of 1 nm or less. Phillips *et al* (1995) describe the use of AFM to examine the surfaces of wool fibres and AFM pictures of scales are shown in **1B(1a)** and in a 3D view in **1B(2)**. A picture, **1B(3)**, of the same area as the AFM view, **1B(1a)**, shows what can be achieved in an uncoated sample by using an advanced SEM with a field emission gun.

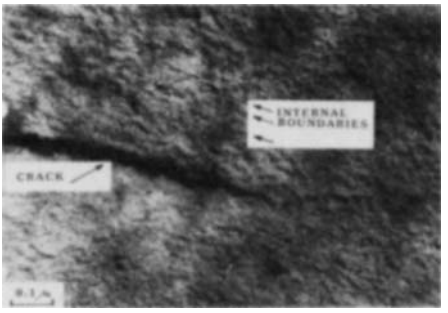
However, as shown in **1B(3)**, better resolution is obtained with coated fibres. The similarity of features, shown in AFM and FESEM, can be seen by comparing **1B(1a)** with **1B(1b),(3)** and, at higher magnification, **1B(2)** with the FESEM image of the same scale edge, **1B(4)**. Another FESEM picture by Phillips *et al*, **1B(5)**, shows a crack opening between the scales of a wool fibre.

An AFM picture of the internal cross-section of a wool fibre, which records the differences in mechanical resistance to probe penetration in different parts of the material, is shown in **1B(6)**. So far, atomic force microscopy has hardly been used to study aspects of fibre fractures, but it clearly has great potential for such studies. Cracks and other deformations of the internal structure of fibres could be examined. Jones (1995) has used AFM to show one form of damage to fibre surfaces, the effect of exposure to ultra-violet light. The difference in surface texture is shown in **1C(1),(2)**.

In some circumstances, at lower magnification, there are benefits in using optical microscopy rather than SEM. Examples of features of damage in ropes are shown in **39M–39P**, both for the larger-scale forms of whole fibres bent at kinks and, in polarised light, for the observation of internal kink-bands, which are not visible, except as surface projections, in SEM views. Another use of optical microscopy, preferably combined with computer-assisted image analysis, is in quantitative studies intended to determine the frequency of various types of damage. Once the SEM has been used to identify different forms clearly, they can be picked out in optical microscopy examination, which is easier and quicker. Studies of patterns of wear in carpet fibres have utilised this procedure, as described in Chapter 33.

The new technique of confocal light microscopy enables detailed views of the internal parts of fibres to be examined. In a confocal laser scanning microscope, a small spot of light is scanned through the specimen and the resultant reflected or fluorescent light picked up by a detector. An image can then be seen on a monitor, or digitally recorded, in the same way as for a scanning electron microscope. Burling-Claridge (1997) at WRONZ has used the technique to follow changes within a wool fibre, as it is deformed by bending. Hamad (1995) has used fluorescence confocal microscopy to study microstructural degradation and fatigue failure mechanisms in wood pulp fibres, as used in paper-making. The cumulative development of cracks is seen in the series in **1C(3)**. The cross-sections in **1C(4)** are from a series taken 30 μm apart, obtained without the disturbance of cutting sections. As the techniques are improved with experience, confocal light microscopy should make it possible to follow the development of cracks and other damage within fibres.

The developments in image analysis in recent years make quantitative analysis of features in images, which may be obtained from many forms of interaction with a specimen, much easier and more powerful. An example comes from UMIST studies on archaeological textiles. By image analysis, the scales on a wool fibre, **1C(5)**, can be reduced to a pattern of sharp lines, **1C(6)**, on which measurements can be made rapidly and accurately. In this example, the object of the investigation was to identify different species by the scale patterns of their hairs. Another example of image analysis on yarn structure is shown in **42D(1)–(3)**.



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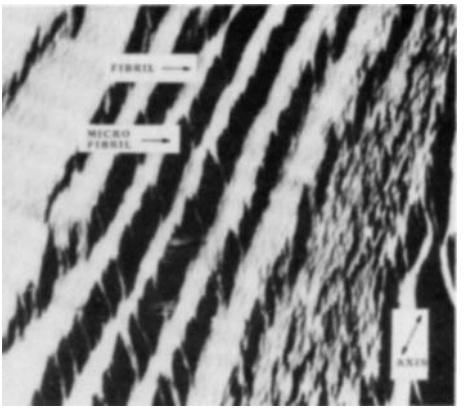

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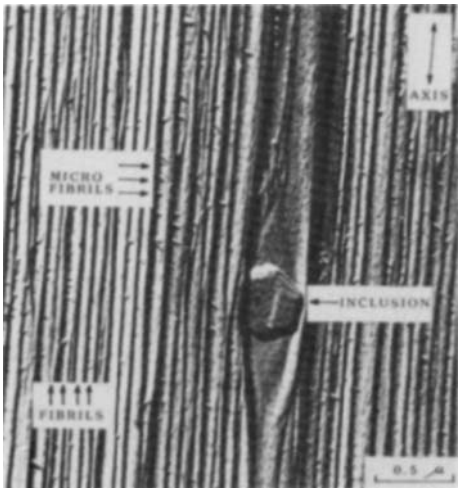

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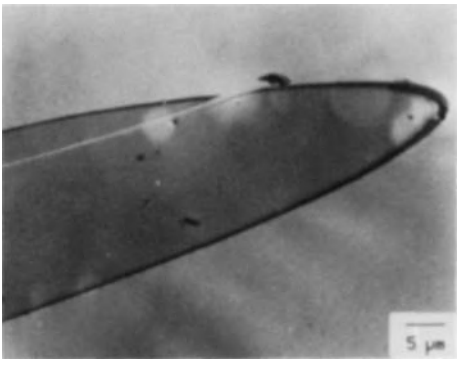

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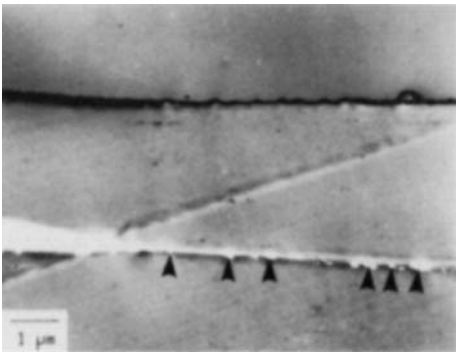

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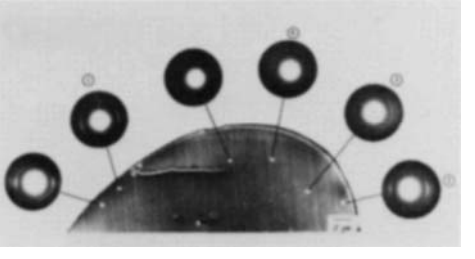

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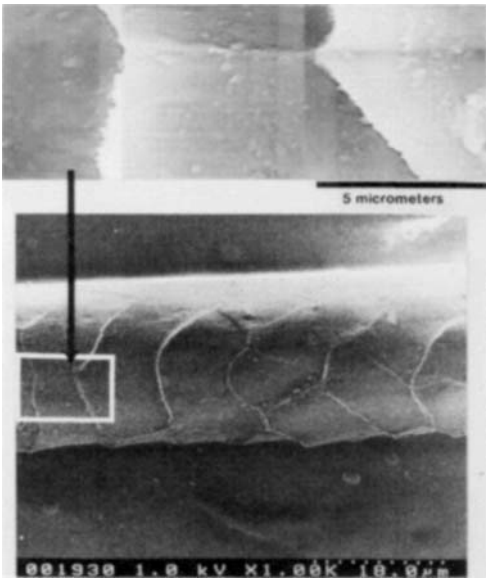
Plate 1A — Transmission electron microscope observations, Davis (1989).
(1) An internal crack of unknown origin in a polyester fibre. (2) Delamination of uniaxially oriented polyester film. (3) Platinum replica of a peeled polyester fibre surface.
TEM of tensile fatigue of polyester fibres, Oudet et al (1984).
(4) Oblique longitudinal section with crack. (5) Tip of crack, preceded by micropores. (6) Oblique transverse section with electron diffraction images.



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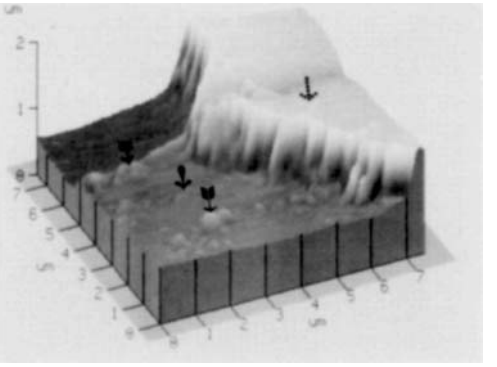

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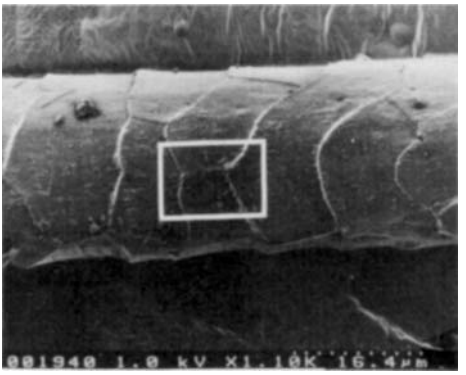

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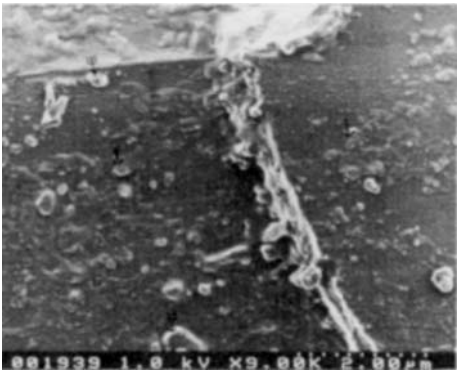

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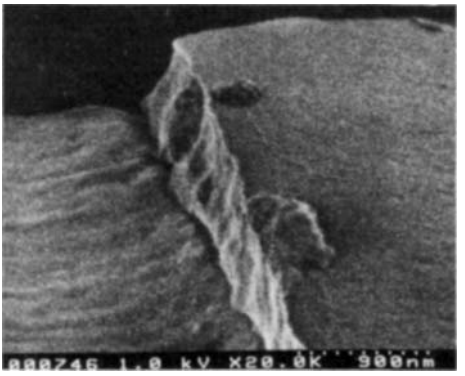

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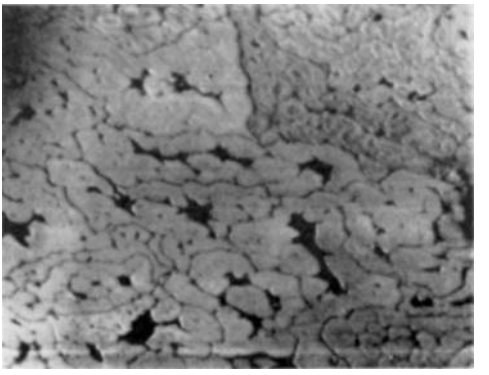

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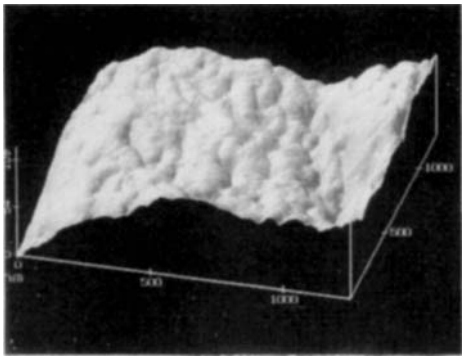
Plate 1B — AFM and FESEM pictures of wool fibre surfaces, Phillips et al (1995).
(1) (a) Upper: AFM mosaic of the surface of a merino wool fibre. (b) Lower: uncoated FESEM image, including the same area of the fibre surface and showing the same shape of the scale edges and other features. (2) Three-dimensional AFM image of one of the scale edges in (1) at high magnification. (3) FESEM image of the same area of the chromium coated fibre with higher resolution. (4) FESEM image of the same scale edge, chromium coated, at high magnification, showing same features. (5) FESEM high magnification micrograph showing a gap between the two scales.
AFM picture of internal structure of wool, courtesy of M. Huson, CSIRO
(6) Showing separate cells in cross-section.



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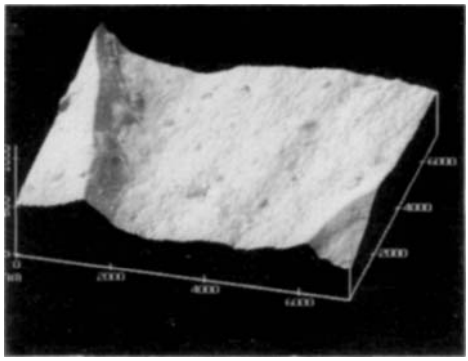

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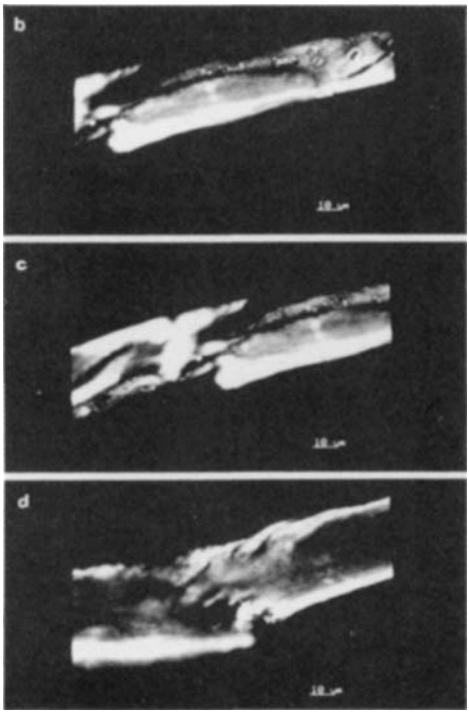

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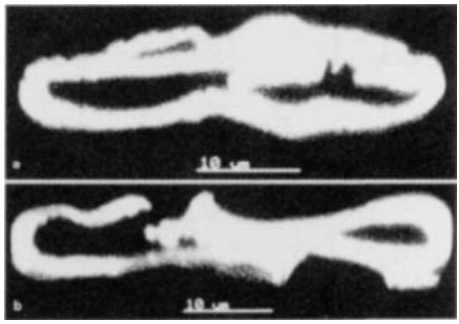

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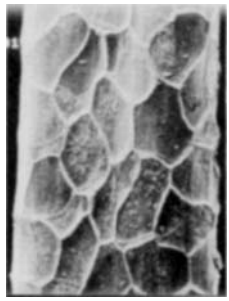

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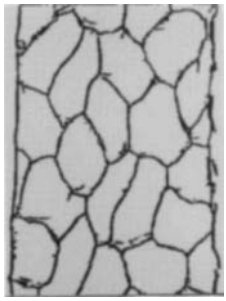

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Plate 1C — AFM images of wool fibre surfaces, Jones (1995).
(1) Unexposed wool. (2) Wool exposed to UV. Scales are in nm.
Confocal images of wood-pulp fibres, jack pine RMP, refined at 6.5 GJ/t and cycled in shear, Hamad (1995).
(3) Series along a fibre. (4) Two cross-sectional views separated by 10 μm.
Image analysis of surface of wool fibre
(5) Image as seen. (6) Pattern after image analysis.

2

EXAMINATION OF WEAR IN TEXTILES

INTRODUCTION

When a single fibre has been broken in a laboratory test, the task of examination is a straightforward one of mounting the fibre and viewing it in the SEM. The examination of wear in a textile product is a much more formidable undertaking. The investigator may be presented, for example, with a pair of trousers, a square yard of carpet, or a six-foot length of rope. Each specimen will contain many millions of fibres. Some parts of the material may be almost undamaged; some will show a few broken fibres, which affect appearance but have a negligible weakening effect; some will show severe damage; and finally, in some places, there may be complete failure, in the form of a hole, of pile worn away leaving only the carpet backing, or of a break or tear.

Where should the examination start? And how should it proceed? A careful plan of work is necessary if meaningful results are to be obtained and if the magnitude of the work is not to become vastly time-consuming. In the end, pictures of two or three fibres may be crucial in providing understanding of what is happening, but only if they can be properly placed in the context of the total sample.

What follows in this chapter is an account of the procedures which we have found useful in a large number of studies over a period of over fifteen years at UMIST, with some earlier experience at the Shirley Institute under the guidance of the late S. C. Simmens.

THE INVESTIGATIVE PROCEDURE

Examination of deterioration in any textile product requires some form of magnifying aid to see fibre damage, and the success of the investigation will depend partly on the equipment available within the investigatory laboratory and partly on the expertise of the laboratory staff.

It is not possible to lay down set rules for examination of worn materials because of the diversity of background of laboratories, but some guidelines can be given based on our experiences in examining a wide range of 'textile materials'. Many 'lay' people equate materials with fabrics, but the term textile materials encompasses many types of products, including, to name but a few: garments of many sorts; some footwear; household materials such as towels, curtains, table linen and bed linen; upholstery and carpeting; workwear and environmental protection garments; ropes; conveyor belts, hosepipes, webbings and many other industrial applications.

In the microscopy laboratory in the Department of Textiles at UMIST, there is a range of equipment from general light microscopes to the more sophisticated and expensive scanning electron microscope (SEM) plus other equipment of use in the work.

The SEM is the ISI model 100A, which is near the top of the range as an imaging instrument, but it does not include analytical facilities, which are available elsewhere in UMIST. For more routine studies, one of the simplest SEM models would be suitable. A list of equipment needed in a laboratory which aims to be well set up for the study of worn or damaged textiles is given in Table 2.1.

The range of textile materials that 'wear out' or 'break down' is considerable and various examples of wear have been investigated at UMIST. These have ranged from worn clothing to worn carpeting, and from ship's hawsers to gas meter gaskets. Not every item can be treated in the same way, but in all investigations the same basic rules for examination apply.

The first step in any investigation is to find out as much about the history of the sample as possible. Always endeavour not to work blind. If the required information is not available or,

Table 2.1 — Laboratory equipment for examining worn textiles

1. *Well-lit table for viewing samples.
2. Macrophotography set-up.
3. *Stereomicroscope preferably with zoom magnification control.
4. *Polarizing microscope for general light microscopy, measurement of some optical properties of fibres and for fibre identification. Plus provision for taking photomicrographs.
5. *Sectioning equipment, fibre and yarn cross-sectioning by either the plate method, hand microtome or precision microtome. Cross-sectioning of fibre, yarn and fabrics by low-speed saw or grinding techniques.
6. Hot-stage for microscope — for investigation of thermal behaviour of fibres and for fibre identification.
7. Interference microscopy for measurement of refractive indices of fibres.
8. *Scanning electron microscope plus sputter-coating equipment.
9. *Provision for developing and printing photographic films.

*Necessary Equipment.

for reasons of security, not released, then this should be noted in the final report, and as much information as possible about the sample gleaned during the course of investigation.

Direct, visual assessment must be made of the sample or samples before any specimens are extracted for detailed examination. The sample should be viewed under good lighting conditions and all aspects noted such as appearance of damage, location of damage, details about wear, discoloration, soiling, colour fading or loss, contamination and any other visible features. At this stage it is advisable to make a record of all observed information together with diagrams or sketches showing the location of damage or other features, as indicated in Fig. 2.1. Photographs of the sample are especially helpful: examples are shown in some of the plates in the case studies, such as **33A**, **35A** and **39A**, **39B**. One photograph of good quality is worth many words of verbal description, but the accent must be on high quality. Any good camera can be used to take a general picture of the garment, often most conveniently while being worn by a model; but, in order to show up damage, a camera with either a macro lens, close-up lenses or extension rings is required. Lighting conditions must be good and carefully noted, to avoid ambiguity between samples and to avoid confusion if more photographs are required at a later date. Remember that a microscopical examination requires removal of specimens which can destroy the history of the sample.

It is advisable at this early stage to check fibre content, yarn and weave structure, seam construction, any other detail that may have some bearing on the examination. Fibre identification is most easily carried out by examining the fibres in a polarizing microscope, but solubility tests and melting-point determinations can be used as back-up tests. Staining tests are often rendered useless when the sample is already dyed or is contaminated. Full details of methods of fibre identification can be found in the book edited by Farnfield and Perry (1975).

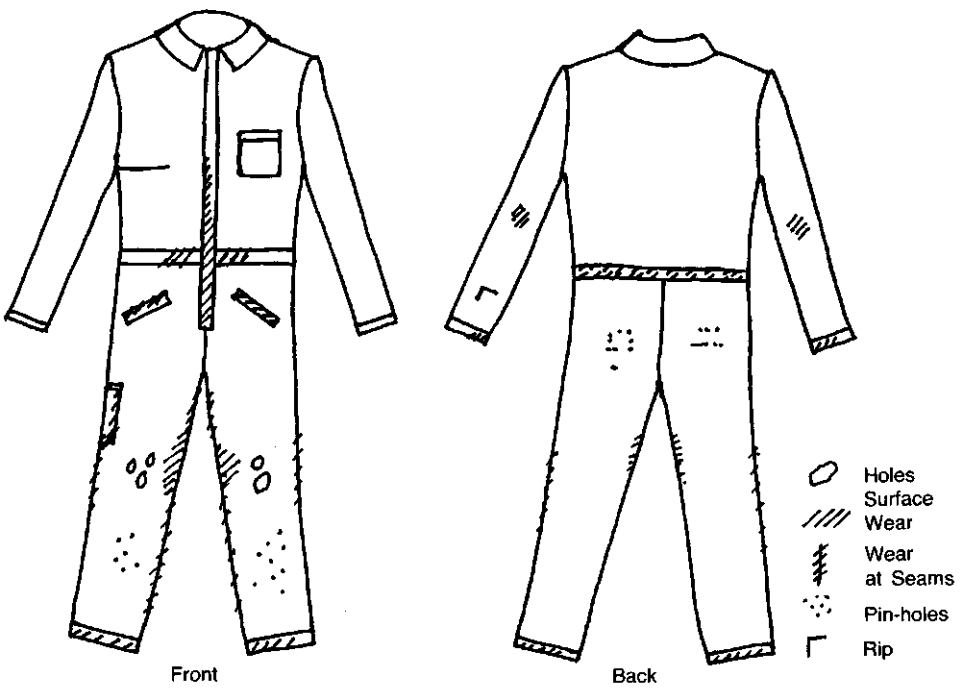

Fig. 2.1 — Sketches illustrating location and type of damage in a worn coverall.

No matter how good one's eyesight, the size and fineness of textile fibres requires magnification for fibres to become clearly visible. An extremely good, first-stage microscope is a stereo microscope, particularly one with a zoom magnification facility. This microscope works in the lower magnification range and gives a three-dimensional image of the sample surface for those people with normal binocular vision. The long working distance between sample and objective lens and the fact that the image is not inverted and reversed as in normal bench microscopes makes examination of damage and checks on fabric structure easy to perform. It also allows easy access, so that the whole product can often be examined, without having to cut out small pieces. If necessary, yarns or fibres can be extracted for detailed study. Again it is advisable to make notes of such features as: damage appearance; location of areas of damage, contamination, discoloration or fading; whether yarn crowns have been flattened; whether the surface is hairy or rubbed-up; and any other interesting detail observed through the stereo microscope. Photography of interesting areas either through the microscope or on a separate macrophotography set-up is an added advantage.

The stereo zoom microscope gives a clear view of the external features of the material, as seen in reflected light, but its use is limited to low magnification. Fibres can be distinguished and the location of broken ends observed, but no detail of damage within the fibre can be resolved.

In order to observe the detailed form of fibre damage it is necessary to go to the higher magnification and resolution of a more powerful light microscope or an SEM. If both are available, it is necessary to decide at this stage which will be of more use, or whether both are needed in the investigation. Each instrument has its advantages and disadvantages and these are summarized in relation to studies of wear of textiles in Tables 2.2 and 2.3. Our experience is that the SEM is usually the most useful tool, and is employed in a simple imaging mode to view the specimen from various directions at appropriate magnifications. In some studies this is usefully assisted by optical microscopy or by X-ray energy analysis in the SEM to make chemical identifications.

Table 2.2 — Advantages and disadvantages of light microscopy

Plus	Minus
1. Internal detail	1. Small depth of focus at high magnifications
2. Surface detail	2. Short working distance at high magnifications
3. Polarised light microscopy (fibre structure)	3. Lower resolving power limit
4. Interference microscopy (fibre structure)	4. Lower magnification limit
5. Colour perception	5. Small samples, mounted in liquid between a cover glass and a microscope slide, and viewed from one direction.
6. Phase contrast and other techniques to introduce image contrast	6. May be confusion between internal and surface features.

Table 2.3 — Advantages and disadvantages of scanning electron microscopy

Plus	Minus
1. Longer working distance	1. Surface detail only
2. Larger depth of focus	2. No colour perception (electron image)
3. Larger specimens	3. Specimens charge up (sputter coating)
4. Higher magnification	4. Possibility of beam damage (sputter coating)
5. Greater resolution	5. Specimen in vacuum chamber
6. Very clear view of surface features	6. Sometimes inability to distinguish between components in a flat (polished) surface
7. Better viewing and handling facilities, e.g. tilt rotation	7. Sometimes inability to differentiate between different types of worn fibres, e.g. between worn wool and nylon fibres.
8. Image processing	
9. Chemical analysis possible by X-ray analysis	
10. Greater surface topography with backscattered detector.	
11. Atomic number contrast with backscattered detector	

SEM STUDY

The range of textile materials is vast, and microscopical techniques of examination vary, depending on the characteristics of the sample. Therefore, for simplicity, let us consider the examination of a worn shirt using the SEM as the main diagnostic tool. The mode of examination can be applied to other garments, and with modifications to thicker larger structures such as ropes and carpets.

The operation of the SEM was described in Chapter 1. It is most useful for examination of the physical effects of damage to a surface. The higher magnifications obtainable, greater depth of field and focus, and large specimen size allow damage to be viewed *in situ*, without disturbing fibre or yarn position. When taking specimens for examination always choose a range of different pieces to cover the full spectrum of damage; whenever it is possible, select from undamaged fabric, then through slight and moderate damage, to severe damage. There are two reasons for this procedure. Firstly, it is necessary to be sure which features are a result of the damage and which were in the original material. Secondly, and most importantly, the region of actual break is usually highly confused, with vast numbers of broken fibres most of which will have failed after the break has started, and may well have been disturbed after the break is completed. The less damaged regions are much more instructive in showing up the sequence of damage and giving clues to its cause. It may be difficult to find undamaged material because, even after only a few wear/wash cycles, the shirt fabric suffers some surface damage which gets progressively worse as the shirt is worn. However, undamaged or relatively undamaged fabric can be found under the collar, in pockets (if any) and in front facings. Slight damage caused mainly by the physical effects of laundering is usually found in the centre back region, and more severe wear is found down the fronts and in elbow regions. The most severely worn parts of the shirt occur along the collar fold, at collar points and along the edges of the cuffs. Rips and tears can occur in various places and are usually accidental or stem from failure of weak places in the shirt.

Care must be taken to avoid bias when taking specimens particularly if a comparison has to be made between several worn garments or items. The way garments wear out depends on working/wearing conditions and also on the individual wearer. Experience has shown that there are variations in wear patterns between users, so that taking specimens at the same places in each garment may solve the problem of personal bias in specimen selection but not cover all the main areas of damage. The positions from which the specimens were taken should be noted either by written notes, or by marking on a sketch of the garment, Fig. 2.2, or on a photograph. The original diagram or photograph of sample damage can be used to mark specimen positions, provided the diagram or photograph does not become too confusing.

The size of the specimen taken will depend on the SEM facilities. We use 15-mm and 32-mm diameter stubs, Fig. 2.3(c,b), in our ISI 100A SEM. The larger stubs will take quite large pieces of material. Alternatively, flat sample holders can be used, Fig. 2.3(a). Specimens are stuck to copper adhesive tape secured to the stub by double-sided adhesive tape. Copper

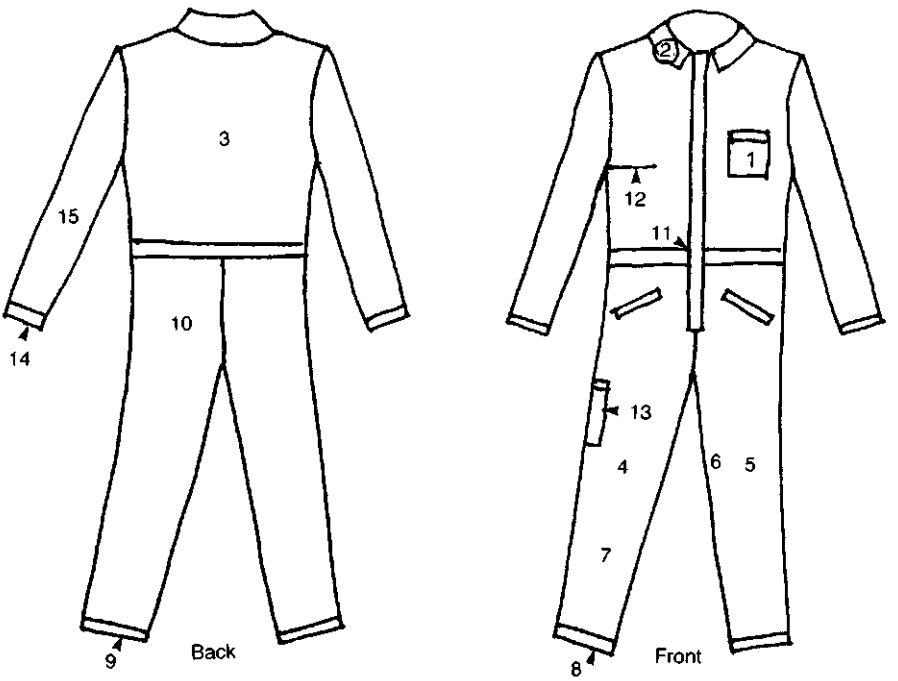

Fig. 2.2 — Sketches showing location of specimens taken for examination. The samples with least wear are (1) inside pocket and (2) under collar.

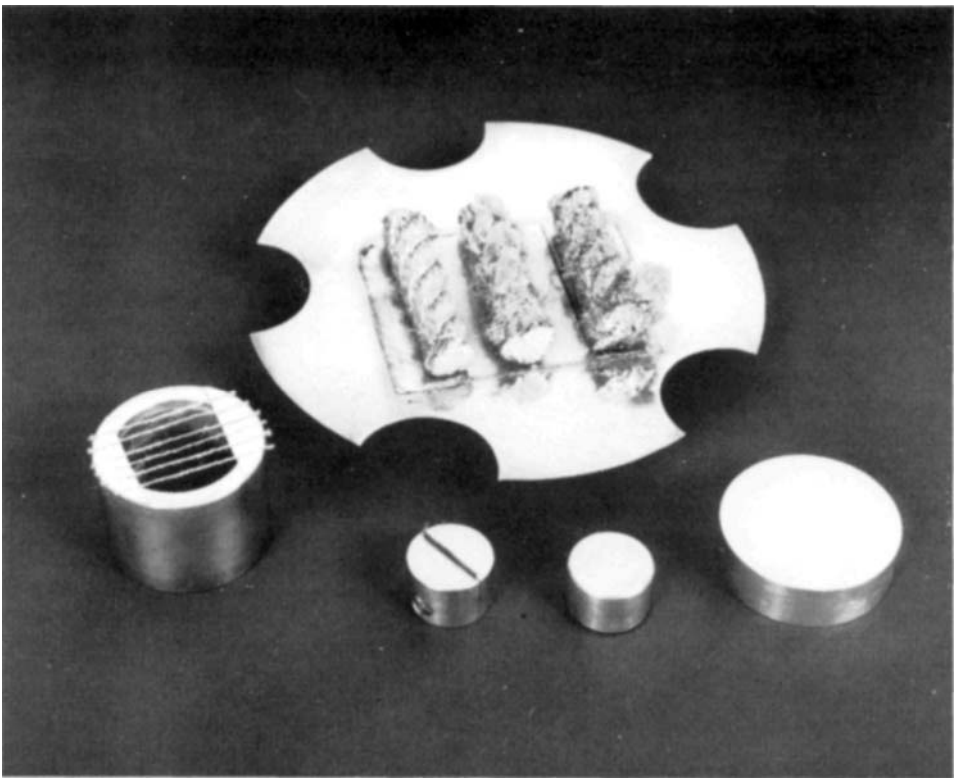

Fig. 2.3 — Stubs for use in SEM. Clockwise from top: (a) large flat specimen holder; (b) large stub, 32 mm diameter; (c) small stub, 15 mm diameter; (d) split stub for mounting fibres, shown in more detail in Fig. 1.3; (e) hollow stub for mounting yarns.

tape is a good conductor to earth for the sample and the sticky layer does not seep up through the sample, as can occur with wet adhesives through a wicking action by the fibres. This is important if worn or frayed edges such as collar folds or cuffs are being examined. Further precautions can be taken to ensure good conduction to earth, particularly for bulky specimens, by painting round the edges of the specimen with silver conducting paint, but, of course, excluding the areas for examination.

Yarns and fibres can be secured to stubs in a similar manner, but to get a clearer view, without the adhesive layer as a background, yarns are often secured across a hollow stub, Fig. 2.3(e), with a carbon base at least 20 mm from the yarns. This means that no signal is received from the base of the stub; and yarns and filaments are viewed against a black background uncluttered with superfluous details. Single broken fibres are examined in split stubs, Fig. 2.3(d) and Fig. 1.3, as described in the previous chapter for fibres broken in laboratory tests.

Personal preference when starting SEM examination is to view the original material to get some idea of what the fabric surface is like before any damage occurs and record this appearance. The direction of warp and weft yarns should be marked on the stub in such a way that it can be clearly seen and understood when the specimen is in the SEM. All stubs must be carefully tabulated to prevent confusion of specimens at a later date. It is important to examine the whole of the specimen surface before choosing areas for photographic record, and always to be wary of making biassed judgements. Choose areas for photographic record which illustrate the type of fibre damage seen, and record some estimate of the amount, severity and extent of damage in notes made at the time of examination: do not rely on memory recall.

Fabric weave controls the height of warp and weft yarns within the fabric; usually one yarn direction receives most damage, as in twill weaves and poplin weaves. Shirts are usually made from poplin fabrics where the warp yarns are damaged first, and it is only after removal of the warp face that the deeper-seated weft yarns are damaged. It is therefore important to know the direction of warp when examining a worn shirt, and the way the various garment pieces are cut from the cloth. Care has to be taken if blended yarns are used in a fabric, because wear of fibres can make their identification suspect in the SEM. Shirts are often made of polyester/cotton blended yarn fabrics, and the main effect of wear here is the loss of cotton fibres, leaving mainly polyester fibres in the worn areas. However, in lightly worn areas difficulty may be experienced in distinguishing cotton from polyester fibres if the cotton component has been mercerized and is of similar diameter to the polyester fibres. Similar difficulty is encountered in wool/nylon blends used in carpets. Undamaged wool fibres can be recognized by their scales, but attrition of the surface removes the scales, and gives the wool fibres a smooth round appearance similar to that of the nylon fibres.

Once SEM examination has been completed, then evaluation is made of the photographic record together with all notes from both the SEM examination and all previous examinations. After evaluation, a report of the results of the investigation can be written. It may be found that further work is needed to fill gaps in information already obtained. This may mean more SEM work, or that some other technique such as light microscopy is required before the investigation of the worn shirt is complete.

LIGHT MICROSCOPY

Light microscopy has several modes of operation not possible on the SEM. One main one is the observation of colour such as effects of dyeing or discoloration of textile materials. It is possible to see colour variations between fibres and depth of dye penetration within a yarn from examination of yarn cross-sections. Fibre identification is possible with the light microscope from a knowledge of fibre appearance both longitudinally and in cross-section, and from the behaviour of fibres when examined in a polarizing microscope. Two or more different fibre components can be identified in a blended yarn and their relative positions within the yarn checked by examining yarn cross-sections.

Important internal features of a fibre can be viewed in light microscopy, such as voids, cracks or slips in molecular alignment (kinkbands). The content and dispersion of titanium dioxide (a fibre delustrant) can be checked for differences between fibres. Variations in birefringence and other defects are seen in a polarizing microscope, and the refractive indices of fibres measured in an interference microscope. Variations in optical properties between fibres of the same type may suggest that the fibres have different histories or have been through different processing conditions. None of these changes in properties can be detected in the SEM, unless the change has physically altered the external appearance of the fibres.

When looking for internal detail it is advisable to match the refractive index of the mountant to the refractive index of the fibre perpendicular to its axis, known as $n_{\perp}$. Then if the fibre is aligned with its axis (lengthways) perpendicular to the vibration direction of the polarizer, the surface of the fibre will be invisible and only internal detail observed, such as delustrant particles or voids. Liquids used as fibre mountants must be chemically inert to the fibres. It is most convenient to make a series of mountants, each of known refractive index, by mixing together two chemically inert but miscible liquids in set proportions to give the range of refractive indices required. Two suitable liquids are liquid paraffin and α-bromonaphthalene.

External damage is seen most clearly when yarns or fibres are mounted in a liquid which is of a much higher refractive index than that of the fibres, e.g. diiodomethane. The contrast in refractive indices clearly reveals damage detail on the surface of fibres.

Staining techniques may be used to reveal damage on fibres. The Congo Red test reveals physical damage, localized chemical and heat tendering of cotton fibres. Chemical damage to wool is revealed by tests such as Pauly reagent for alkali damage and Kiton Red G test for chlorinated wool. Other chemical tests are used on wool to reveal alkali damage (Allworden reaction) and acid damage (Kris–Viertel reaction). The results of all these tests are examined in the light microscope.

The selective dyeing of damaged regions in a fibre is a particularly useful way of estimating the extent of damage in a specimen. The piece of fabric is dyed, and then, when examined in the microscope, the damaged zones on the fibre show up and can be counted. A technique of this type was used by W. D. Cooke to elucidate how pills (small tangled balls of fibre) develop on knitted fabrics by alternating damage at the anchor point and roll-up into the pill.

Cellulose fibres such as cotton and viscose rayon are attacked by micro-organisms if the conditions for growth are present. The growth may be visible in the SEM, but its nature is confirmed by optical examination of stained specimens. Mildew can be detected by the Safrannin–Piero Aniline Blue test, and bacteria are stained by Loeffler's Methylene Blue method. The damage caused by these organisms is distinguished in cotton fibres by using the Congo Red test.

As with SEM work, it is always advisable to make a written record of what has been seen in the light microscope, augmented with diagrams, sketches or better still by photomicrography. Once the examination is complete a report of the finding is made. This may be based solely on light microscopy or may be a combination of both light and SEM work, depending on laboratory facilities. Much of our work is a combination of both.

EXAMINATION OF OTHER PRODUCTS

The handling of samples will depend on the nature of the product. Small pieces of carpet can be mounted in the SEM, but the following points should be noted. The pile of a carpet and its backing trap air, which leads to long pump-down times when sputter-coating. When examining a carpet pile in the SEM, only the top of the pile is seen, and therefore either pile yarns have to be examined separately in the SEM or examined in the light microscope to see how far damage has progressed down into the pile. In carpets with blended piles, e.g. wool/nylon or acrylic/nylon piles, wear removes surface features from wool and acrylic fibres, making differentiation from nylon fibres very difficult; however, in the polarizing microscope the fibres are easily distinguished.

Ropes pose problems with regard to size. The smaller ropes can be mounted whole, and samples larger than a 32-mm diameter stub can still be accommodated in the SEM chamber. With our ISI 100A SEM, specimens up to about 100-mm across can be examined. If the sample has to be divided into smaller pieces, careful examination and recording of detail, preferably by photography, is essential. It should also be remembered that it is not just the outside of a rope which gets damaged; but that damage occurs between strands and also between yarns in the strands, and even between filaments within a yarn. Even small-diameter ropes have to be separated down into smaller units. Careful observations and notes of sample appearance before examination are essential.

Samples which have been plastic coated or have received some surface covering finish or are contaminated cause problems. If damage can be seen through gaps or breaks in the surface covering, examine the damage *in situ* before attempting to remove the coating or contaminant by chemical means, since this causes disturbance of damaged yarns and removal of loose material such as broken fibres in the damage sites.

CONCLUSION

The brief account given in this chapter should help to lead investigators to productive studies of damaged products, and this will be further helped by examining the case studies in Parts VI and VII. However, the only real training consists of experience on the job. The guidelines which have been given can be applied to any textile product or sample, provided its size is manageable — and, indeed, even the largest items such as a massive hawser laid out in a rope-maker's yard can be sampled.

The amount of time and effort needed to complete an investigation depends on the nature of the material and the purpose of the investigation. Sometimes it may only be necessary to identify the presence of features which have been extensively studied in earlier investigations, and thus serve to establish the cause of damage. This type of investigation will be quite short. At the other extreme, the first basic studies of particular forms of damage can be extended almost limitlessly in time as more and more detail is shown up, and so it is necessary, implicitly or explicitly, to balance the cost of the investigation and other working constraints against the value of the information obtained.

Sometimes it is not possible to reach a firm and valid conclusion. In these cases one can only report what has been observed, and perhaps speculate on the possible causes of damage.