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Sectioning Techniques for Elastomer Blend Preparation by Ultramicrotomy for Transmission Electron Microscopy

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The preparation of samples for transmission electron microscopy has often been referred to as more of an art form than a science. This definition has arisen because of difficulties, on the part of microscopists, in defining rules by which the necessary ultra-thin sections can be prepared. Increasingly sophisticated apparatus has alleviated some of the problems related to reproducibility but it has been observed that different operators still continue successfully to use different conditions to achieve the same results. Elastomers are considered by many to be one of the most difficult groups of materials to prepare for electron microscopy. This paper describes some of the techniques involved in sectioning such materials and some of the special conditions that can be applied to improve the chances of success. Many working in this field will have come from a biological background and so consideration is made of the differences in preparation and examination between biological materials and elastomers. Examples of micrographs taken of sectioned elastomer blends are given.

Numerous light microscopical techniques exist for the examination of blend morphology[1,2,3]. However, all of these suffer from the same drawback; the maximum attainable resolution available using a visible light source is physically limited to approximately 0.25 μm by the wavelength of visible light. Consequently in some materials it becomes difficult to observe blend morphology and it is generally impossible to observe any micro-structure within a phase (micro domains, filler location *etc.*). This is not to suggest that there is no place for light microscopy (LM) since clearly that is not so but, with the improvements made in blend technology and the ever-increasing demand for information, many laboratories have discovered that high resolution imaging of blends is no longer an expensive luxury but a necessary requirement. For the laboratory that finds itself in this predicament, only three options really exist:

- Convert an existing scanning electron microscope (SEM) to transmission imaging (the author has dealt with this possibility elsewhere[4]). This provides a dramatic improvement in resolution over light microscopy but does not compete with a transmission electron microscope (TEM) in high resolution terms.

- Set up a fully equipped transmission electron microscope suite.

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- Find a consultancy such as *Rubber Consultants* that offers these facilities and has experience with them.

It should be stated at the outset that good TEM imaging depends upon good specimen preparation. It therefore follows that poor quality specimens inevitably lead to poor TEM images. Unfortunately, there are no short cuts and if the facilities and manpower are not available to do the job to a sufficient standard then the laboratory in question would be well advised to send the necessary work to a third party. If the correct preparative instrumentation is not available, it is highly unlikely that reasonable specimens can be prepared.

The aim of this paper is to describe in some detail the preparation of elastomers for imaging by transmission electron microscopy and to give some examples of the kind of images that can be obtained. Comparisons will be drawn between the information available by LM and that available from identical samples by TEM.

SPECIMEN PREPARATION

The aim of this section is to provide some detail about the preparation of good quality thin and ultra-thin sections. The most easily interpreted images of phase morphology are usually obtained by transmission imaging of materials in cross-section. (Solvent casting of thin films of unvulcanised material is also possible but this topic is not considered here). The preparation of thin and ultra-thin sections of elastomers and elastomer blends is without doubt the most time-consuming aspect of microscopical examination. It must be reiterated that good micrographs can only be obtained from good sections and these can only be obtained by patience and taking the time to develop the necessary skills. In fact the actual taking of the micrograph often accounts for only a fraction of the time involved in the overall analysis. It should also be noted that the right equipment is essential, thus it can be argued that it is highly unlikely that reproducibly preparing ultra-thin sections for transmission electron microscopy using a base-sledge microtome with a steel blade would be possible. The laboratory at MRPRA is equipped with an RMC MT7000 Ultramicrotome with a CR-21 Cryo Prep. Unit. This instrumentation can maintain cryogenic temperatures to ±0.1°C and without this kind of thermal stability the preparation of ultra-thin sections would be more a matter of luck than any skill on the part of the operator.

At this point it is necessary to discuss how thin a usable ultra-thin section should be. In terms of obtaining an image it is unlikely that an operator with a 100kV TEM will be able to obtain meaningful images from sections that are thicker than 200 nm in a simple gum blend. However the usual statement that 'the thinner the better', meaning that if a section is greater than 50 nm thick it is of no use, is patently untrue. It would certainly be true to say that the thinner the section the higher the resolution image that can be obtained from that section, but very few examinations in this context require 5 nm resolution! It is therefore necessary to apply some common sense and an awareness of economics to the subject. Anyone with any understanding of the nature of the problems involved with sectioning elastomers will be aware that a general rule of thumb is that the thinner the section, the longer it will take to obtain the conditions required to obtain that section (and consequently the more it will cost in real terms to produce that section). Cosslett, however, added the rule that the maximum resolution obtainable was approximately one tenth of the thickness of

the section[5,6]. Modern instruments have various techniques for correcting for chromatic blurring but Cosslett's figure still serves as a useful guide. This could be restated that the section could be up to ten times as thick as the resolution required, provided instrumental limitations are taken into account. Therefore, if the operator plans to use TEM for imaging at reasonably low magnification and requires a resolution of merely 20 nm then, in theory, the sections used could be up to 200 nm thick. Had the operator spent extra time trying to obtain sections that were 50 nm thick then it could be argued that the time could have been used more effectively. Common sense also dictates that if the morphological or structural features of interest are likely to be much smaller than the anticipated section thickness then there is a danger of overlapping features and confusion of detail. However, this may not necessarily be a problem provided that the operator is willing to spend some time interpreting the image. For example, the TEM micrograph of the NR/EPDM blend considered below is thick enough to reveal a great deal about the structure of the EPDM phase within the NR matrix although there is some confusion of detail regarding the micro domains. What is required therefore is a little thought as to what information is really required before preparation begins.

It should also be added that, contrary to much popular opinion amongst biologists, it is better to operate the TEM at high accelerating voltages to aid resolution, improve the brightness of the image and minimise beam damage. Traditionally it has been held that low accelerating voltages will improve contrast and give better images but this is not the case with elastomers. Most of the contrast in images of elastomers is artificially produced by chemical staining and further contrast can be added if necessary at the photographic printing stage. In fact, after micrographing a chemically stained blend it is often necessary to remove contrast at the printing stage because a photographic print does not have the contrast range that a TEM negative has. Failure to do so can result in areas on the print being totally white or black and therefore containing no information. The use of low accelerating voltages can also increase the rate at which polymers succumb to damage by the electron beam since the lower energy beam actually heats the specimen more than a higher energy beam to which the specimen is more transparent.

SECTIONING TECHNIQUES

The Knife

For ultramicrotomy there are really only two choices, a glass knife or a diamond knife. (There was a short-lived attempt at introducing sapphire knives several years ago but these do not appear to have attracted sufficient long-term interest to survive). The advantage of a diamond knife is that it is intrinsically sharper than a glass knife and the edge remains sharp for far longer. However, as might be expected, diamond knives are extremely expensive to buy and they have to be kept and used very carefully since they are easily damaged. The author has tried a number of diamond knives on materials filled with either zinc oxide or silica and in each case the knife edge was damaged quite quickly, presumably by individual particles within the matrix. It is not known whether all diamond knives will behave in this manner with filled elastomers but the knives tested were rejected on the grounds of lack of longevity. On the positive side, the sections which were cut were thinner and less prone to most artefacts than those cut using a

glass knife. However knife marks were apparent and, as discussed below, knife marks are usually a clear sign of damage to the knife edge.

In contrast, glass knives are cheap to make and are disposable after use. It is true that it is unlikely that an operator will be able to produce a glass knife that is as sharp as a diamond knife, but from an economic point-of-view many laboratories cannot justify the costs of purchase and regular resharpening (usually about half the cost of the original knife) of a diamond knife. Glass knives are usually produced in-house by a dedicated knife making apparatus. A knife maker is a mechanical device for reproducibly scoring and breaking glass into knives. It should be stressed that making a glass knife can be a time-consuming process and that the best knives (with the fewest stresses in the edge) are made by a slow break usually taking in excess of fifteen minutes. Knives can, of course, be broken more quickly for LM where ultra-thin sections are not required. When broken, the knife edge has the appearance illustrated in *Figure 1*. A rule of thumb is that the slower the break, the fainter the stress mark. With practice it is possible to break knives in which the mark is almost invisible.

There are essentially two regions on the knife edge. The right-hand two thirds of the knife are generally unsuitable for ultramicrotomy due to increasing roughness. This part of the knife is best used for trimming the block face prior to sectioning. The left-hand third of the edge is far more regular and seems to become sharper towards the left. There is a tendency for the edge to become rough where the stress mark joins it but once again a slow break reduces this problem. Indeed there are a number of blends that can only ever be sectioned at the far left-hand side of the knife.

Sectioning Temperature

To consider sectioning temperature effectively, it is first necessary to consider the nature of the 'cut' itself. A simple attempt at cutting a vulcanisate with a razor blade illustrates the difficulties involved with cutting rubber. In order to section rubber, it is necessary to reduce its temperature to below its glass transition temperature (T_g). However, if natural rubber with a glass transition temperature of −72°C is considered, it is unlikely to be sufficient simply to reduce the temperature to −80°C. This is because when one is sectioning below the T_g of a material one is not actually cutting the specimen but fracturing it in a controlled manner. The process of fracturing the sample liberates energy in the form of heat which can raise the localised temperature substantially at the tip of the knife. The rise in temperature is likely to be dictated to some degree by other sectioning conditions including the sectioning speed, which is the speed at which the sample is passing over the knife. However, the important point to note is that it is quite possible to raise the temperature at the fracture initiation point to above the T_g. The effect of this would be that the knife may then have a tendency to stick into the sample causing a number of artefacts including tearing. Some operators have suggested that a localised temperature rise of 50°C is possible[7], but experience suggests that, provided the operator is not trying to section using 'impossible' conditions, it is necessary to be merely 20–30°C below the T_g. If the material under consideration is a blend it is usually important to judge the sectioning temperature from the lowest T_g.

Size and Shape of the Block Face

The size and shape of the block face are far more important than one might imagine. With

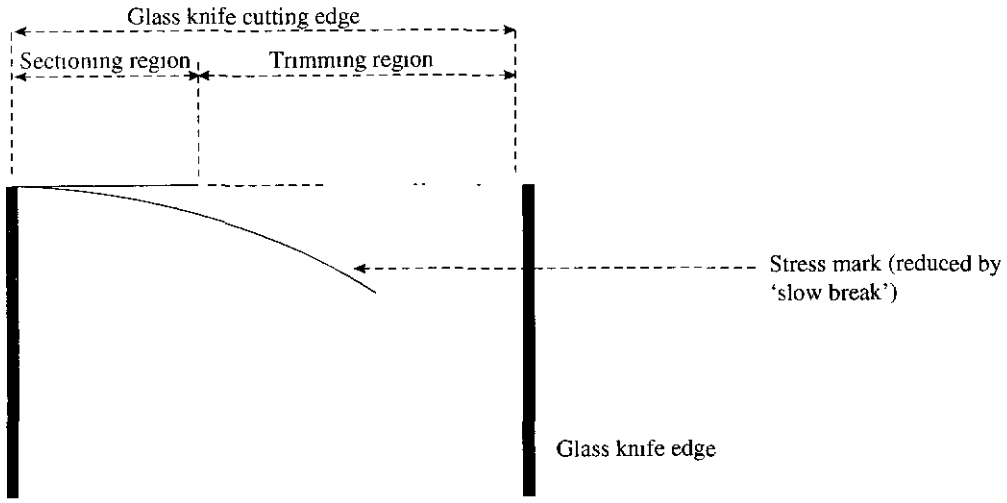

Figure 1. Diagramatic representation of a glass knife edge viewed from above

respect to the size of the block face the general rule is 'the smaller the better' and it is suggested that the best results are usually obtained from block faces less than 0.5 mm wide with the longest face oriented vertically. In terms of shape, a large number of books have been written about microscopy and microtomy concentrating on biological specimens and much of the advice given does not apply to elastomers to the point that application of some of the ideas can be distinctly unhelpful. One such idea concerns the shape of the block face. Biologists generally examine materials which have been embedded in a hard polymer and can be sectioned at room temperature. Consequently they are able to section into a trough filled with water and can produce long ribbons of sections that float. Their preferred block face shape is illustrated in *Figure 2* which maximises the chance of ribbon forming. When one is sectioning at cryogenic temperatures this is not possible and neither is the trapezoid shape desirable with its broad leading edge often blunting the edge of the knife. Instead two other shapes are proposed; the first of these seems to be fairly common practice in polymer circles which is to use a triangular shaped block with the sharp edge pointed towards the knife. This is a useful shape but has as its greatest pitfall the problem of the section not detaching from the block at the end of the cutting cycle. A little consideration of the problem leads to the simple modification to the shape as shown in *Figure 2* which reduces the likelihood of the section not detaching. As with much of sectioning this is a simple change but it makes a big difference to the ease with which sections can be prepared. It is likely that the operator will experience problems in trimming the block to such a precise shape. The suggested technique is to trim the block face to a triangle using a razor blade prior to mounting it in the microtome. It should be cooled to the required temperature at which it is then possible to trim the block using a small pre-chilled scalpel.

Knife Angle, Clearance Angle and Sectioning Speed

These could be described as general operating conditions. It is the interplay between

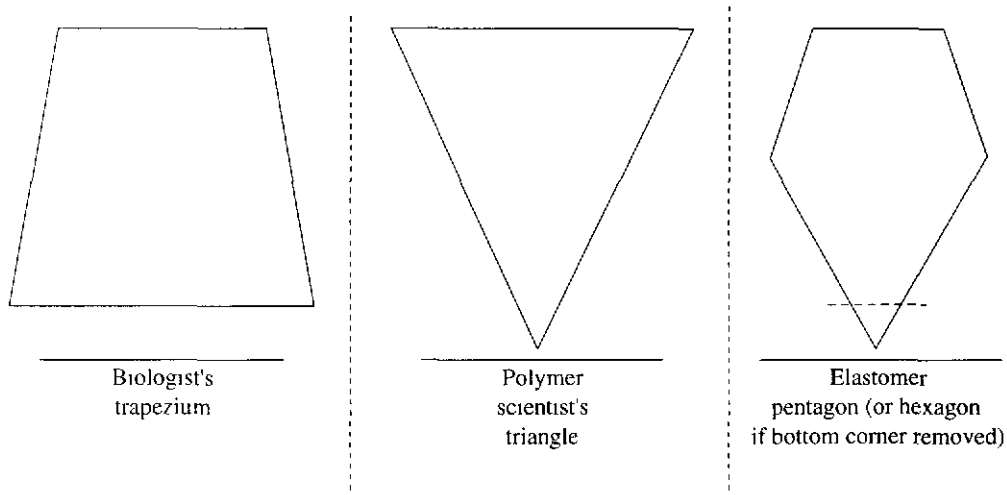

Figure 2. Different block face shapes.

these three sets of conditions that usually determine whether sectioning will be successful or not, provided that the correct temperature has been established. The distinction between knife angle and clearance angle should first be drawn. These are illustrated below in *Figure 3*.

When working with glass knives there is always a competition between their sharpness and longevity. Experience suggests that knives made with small knife angles, *e.g.* 35°, tend to be sharper than knives made with large knife angles, *e.g.* 60°. The choice of the knife angle is often reliant on the particular operator and the way in which he or she sections. Some dependence also exists on the type of specimen under consideration. If the sample is very hard then using a larger angled knife may be necessary. However, it is likely that this will be at the cost of sharpness and consequently the sections cut may not be of a desirable thickness. For most elastomer applications it has been found that a 45° knife seems to provide the optimum conditions. It should be noted that the actual knife angle may differ from the angle set on the knife maker since the fracture will tend to curl away towards the knife edge at the end of the score mark.

The clearance angle is determined by the hardness of the specimen at the temperature used in that the harder the block is, the larger the clearance angle will need to be in order for sections to be cut. This angle can be varied between 1° and 10° on most ultramicrotomes. However, it should also be noted that the steeper the angle, the more prone the sections will be to compression because the knife will have a tendency to 'stick in' to the block face. There is also a tendency for the knife to scrape across the surface of hard block faces if the clearance angle is too shallow. If this happens it will cause a rapid blunting of the knife edge, and this will be further degraded when the knife finally catches because the section will be far thicker than originally intended. It is therefore up to the operator to judge the specimen and set the clearance angle accordingly.

Sectioning speed is dictated by both the sample and the operator. No clear guidelines

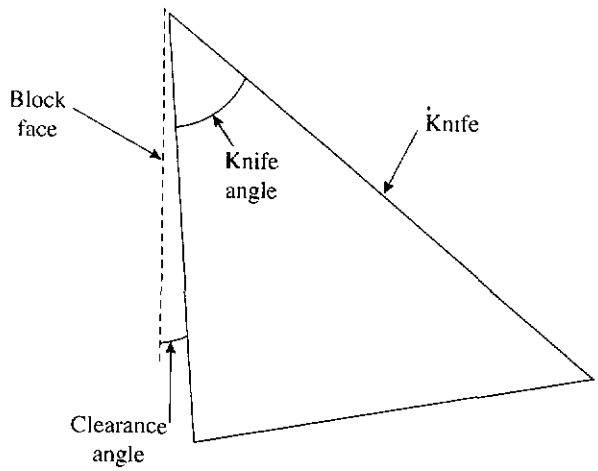

Figure 3 The knife angle and clearance angle on a glass knife.

can be set but it is often that sectioning speed dictates, more than any of the other variables with the possible exception of temperature, whether sectioning will be successful or not. Usually it is better to start at a slower speed and increase if necessary. With practice it becomes more easy to section manually since the operator maintains more control than if motor control is selected. The speed at which one chooses to section is very much a part of the 'art' of sectioning.

Sectioning Using a Trough Liquid

Again this is a point at which skills and practices developed for biological specimens are largely inapplicable to elastomers. As stated above, biologists usually work at room temperature with embedded samples. With such samples it is possible to section into a knife trough filled with water on to which the sections will float and from which they can be easily collected. However, when one is working at cryogenic temperatures, water is not a viable option as a trough liquid (except in the special case described below). There are some trough liquids available that do not freeze at the cryogenic temperatures described here, although sections do not float on these in the same way that they do on water at room temperature. One such example is n-propanol which is effective down to about −125°C. The best results can be obtained from having a small amount of n-propanol in the trough that can be swept carefully up the knife to the edge using a single hair brush. Care must be taken not to add too much since its relatively high viscosity leads it to being easily dragged over the edge of the knife and on to the sample, at which point it usually becomes necessary to stop and clean the block. Careful sectioning will then lead to sections sliding down the n-propanol into the reservoir. When sufficient sections have been taken, more n-propanol is added to the trough to raise the level and sections are retrieved using a TEM grid. These sections are quickly but gently laid on to the surface of a water-filled petri dish. The interaction between the n-propanol and the water usually leads to the sections being floated off on to the water

and flattened out by the action of surface tension. The required sections can then be chosen and removed on a fresh TEM examination grid.

Sectioning without a Trough Liquid

In numerous cases it is found that sectioning with n-propanol is not appropriate. A simple alternative is to section without the use of a trough liquid. The advantage of this is that it is generally easier to judge sections *in situ* and reject those that are unsuitable. Sections are collected on the knife itself and are then carefully positioned on a TEM grid by holding the grid against the knife (away from the knife edge) and manipulating the sections with a single-hair brush. This can be less time consuming than other techniques since it is possible to remove only as many sections as are needed, rather than to continue sectioning until plenty have been removed in the hope that at least some of them will be suitable. The main disadvantage with sectioning dry is that a buildup of static electricity in the chamber, which is a common problem, can lead to sections being difficult to handle and prone either to sticking to the sample or the knife, or flying around the cryo-chamber when attempts are made to move them. Anti-static guns are available but they are expensive and careless use can damage the sensitive electronics of a cryo-unit. There is also no flattening action caused by the surface tension of the water, as in the above case.

It should also be added, at this point, that if the operator is working with unvulcanised materials then this is the technique of choice since interactions between ultra-thin sections and some trough liquids (including water) have been observed. This could lead to distortions in the sections, artefacts and, consequently, misleading information.

Sectioning on to Ice

This is a recently developed technique[8] and is ideal for sectioning materials that have a tendency to curl (*e.g.* NR/BR blends). It is probably the most time consuming technique and can only be used if the cryo-ultramicrotome has an inbuilt defrost unit that automatically raises the temperature to just above ambient. High temperature bake-out units should be avoided for this application unless the bake-out temperature can be set to 35°C–40°C. The RMC CR-21 unit is ideal since it raises the temperature of the apparatus to 35°C. The apparatus is prepared by placing a knife with a trough filled with distilled water in the knife holder. The apparatus is cooled in the usual way and the water in the knife trough will freeze. Sections are cut and arranged on the ice surface so that they are touching without overlapping. After sufficient sections have been taken, the apparatus is warmed until the ice melts. As it turns from ice to slush the sudden increase in surface tension flattens out the sections. (Prior to melting some manipulation of the sections is possible although care must be taken above the T_g not to distort them). Once all the ice has melted, the sections can be retrieved on a TEM grid. This is a time-consuming technique since the apparatus has to be repeatedly warmed and cooled between samples. It does however give good results with difficult specimens.

COMMON PROBLEMS

Curling

Curling during sectioning of vulcanised material usually suggests that the section is too thick. This is solved 'simply' by cutting thinner sections. The most irritating type of curling occurs when seemingly flat sections curl as

they are brought up from cryogenic temperatures to room temperature. It seems likely that much of this is due to inbuilt stresses in the material from moulding. While the material is a coherent whole it retains its shape but in some cases, when a section is removed from the bulk cryogenically and allowed to warm to room temperature, the stresses caused the section to curl. This is a particularly common problem in blends containing elastomers with very different T_gs. One good example of this phenomenon is NR/BR blends. As the section is warmed, BR will return to being elastomeric near −110°C (dependent on the type of BR) whereas NR will remain a glass until its temperature is raised above −70°C. The consequence of this is that the BR phase will start to relax out the strains while the NR phase in still a glass. This often leads to the section curling.

There are two possible techniques for overcoming the problem of curling. One of these is the ice-sectioning method described above. The other is to use a triangular block face because this will have a tendency to curl from all three edges resulting in the curl effectively bracing itself and thus leaving a flat region at the centre of the section. This second method is a little less predictable as it depends on the section being the same thickness throughout. However, practice and perseverance can bring some good results.

Knife Marks

These are an inevitable consequence of sectioning an elastomer which contains particulate matter such as zinc oxide, silica, carbon black, *etc.* As the knife cuts through such a specimen and strikes a hard particle the knife may be slightly damaged at that point. From then on, the damage to the knife edge will be translated on to any section and block face, as it passes over that point on the knife, as a long line in the direction of sectioning. Obviously the more filler present in a material the more likely the knife is to be damaged. If a diamond knife is used then the edge will probably not be damaged as quickly, but any damage will be transmitted to any sections taken with that part of the knife until it is resharpened.

Knife marks do, however, have an important use. They can reveal the direction of sectioning which may be important when trying to decide whether the shape of a structure has been influenced by compression (see below). Where the knife marking is severe a computer imaging macro, such as the one written at MRPRA, can be used to improve the visual appearance of an image that has been digitally collected.

Compression

When a section is removed from a block, it often appears to be shorter than the vertical face of the block from which it was removed. This is known as compression. In more severe cases wrinkles appear at right angles to the sectioning direction. These regions should be avoided when taking micrographs. Usually the effect can be reduced by changing the clearance angle and/or the sectioning speed. Bad compression seems to be a result of too high a sectioning speed and/or too steep a clearance angle.

Chatter

Chatter occurs when a high frequency vibration is set up between the specimen block and the knife and leads to regular variations in the thickness of the section. This is observed as parallel lines at right angles to the sectioning

direction. As with compression its cause is usually a combination of wrong clearance angle and sectioning speeds.

Inconsistent Sections

Once again in biological circles it is expected that the correct sectioning conditions will lead to a ribbon of ultra-thin sections floating on the water in the trough. It is also expected that once the conditions are correctly set sections will be cut serially, *i.e.* on every cutting stroke. Experience indicates that this is not the case with elastomers. With most technological materials it is unlikely that the operator will be able to stay with any single region of the knife for a prolonged period of time before it becomes blunted. The time taken for this to occur depends largely on the material and on the conditions that the operator is attempting to use. It is quite conceivable that the knife will become blunt after only five or ten cutting cycles and the operator will need to move to the next piece of knife edge. Clearly with the usable knife edge being quite short it is necessary to obtain the correct conditions quickly. With practice and experience a good operator should be able to assess from the type of material what kind of conditions should be set up initially. However, even then it is highly unlikely that serial sections will be cut, or if they are it is unlikely that they will be cut for more than a few sections. Generally speaking the harder the block the steeper the clearance angle that will be required. Sectioning speed is more difficult to assess and seems to depend on too many factors (including operator preference) to be able to give complete guidelines. Generally speaking it is better to start at slow speeds and shallow clearance angles because these will do less damage to the knife edge if they are incorrect.

EXAMPLES

NR/EPDM

This particular blend is an excellent example of how LM can appear to supply all the necessary information. However, once the material has been examined by TEM it becomes clear that there is far more information that can be obtained. The sample in question was prepared at −100°C with a dry glass knife for TEM and using a trough filled with n-propanol for LM. Sectioning was carried out manually and sections were mounted in polybutene for phase contrast LM using a Leitz Ortholux II light microscope or stained in osmium tetroxide for one hour prior to examination using a Phillips EM300 transmission electron microscope. As mentioned above, it has been found that better results are obtained from an instrument running at a higher accelerating voltage and so examinations were carried out at 100kV. Contrast was maximised by using a small objective aperture, in this case 30 μm which is the optimum size for the EM300[9].

Light Microscopy

Figure 4 shows a phase contrast light micrograph that was originally taken at the maximum usable magnification for the technique. The EPDM phase is identified as the lighter phase. The micrograph appears to give a reasonable amount of information regarding blend morphology and a reasonable mean estimate of cross-sectional phase dimensions could be determined. For some applications this limited amount of information would be sufficient.

Transmission Electron Microscopy

Figure 5 shows the same material taken at approximately thirteen times the magnification

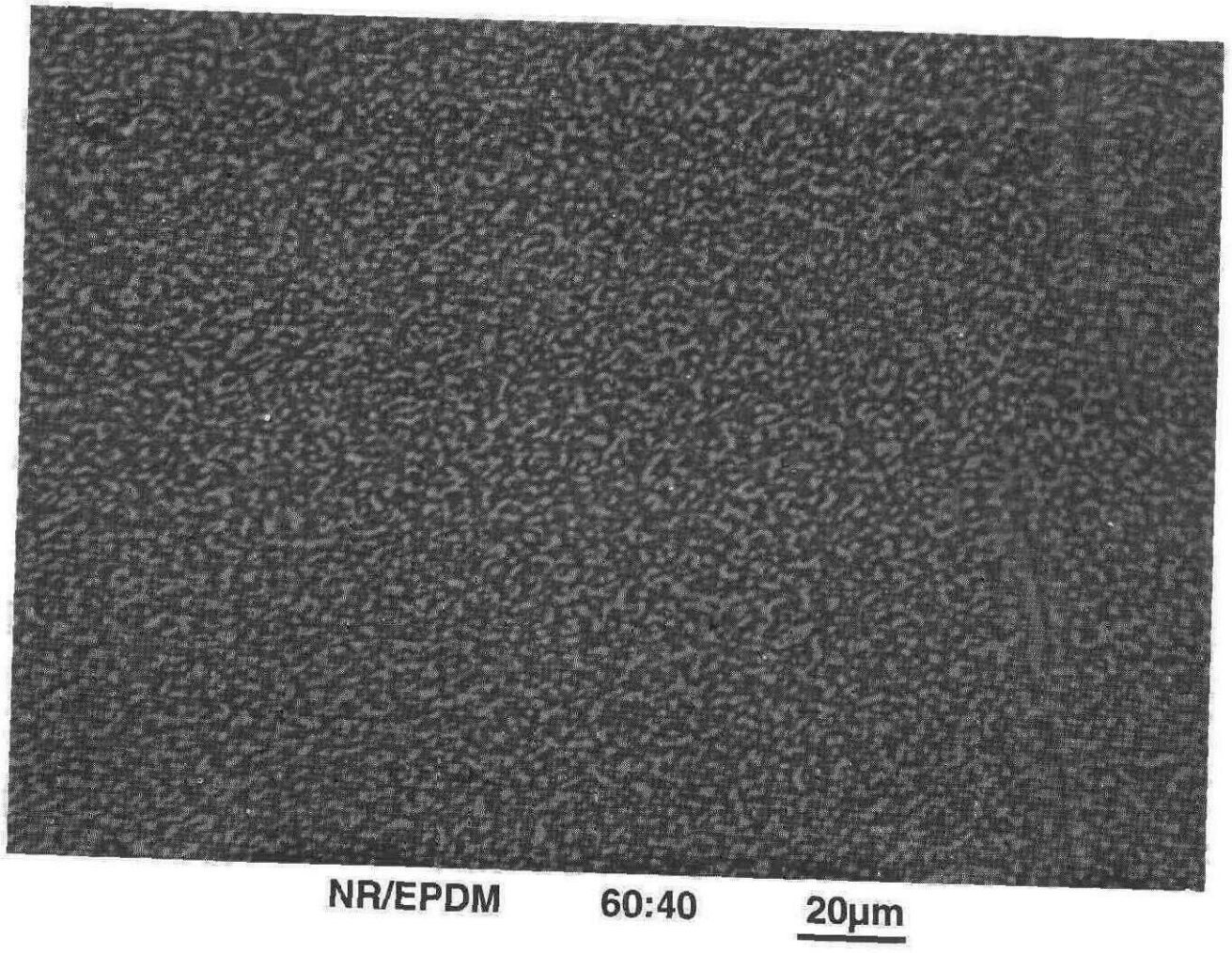

Figure 4. Phase contrast light micrograph of NR/EPDM 60:40.

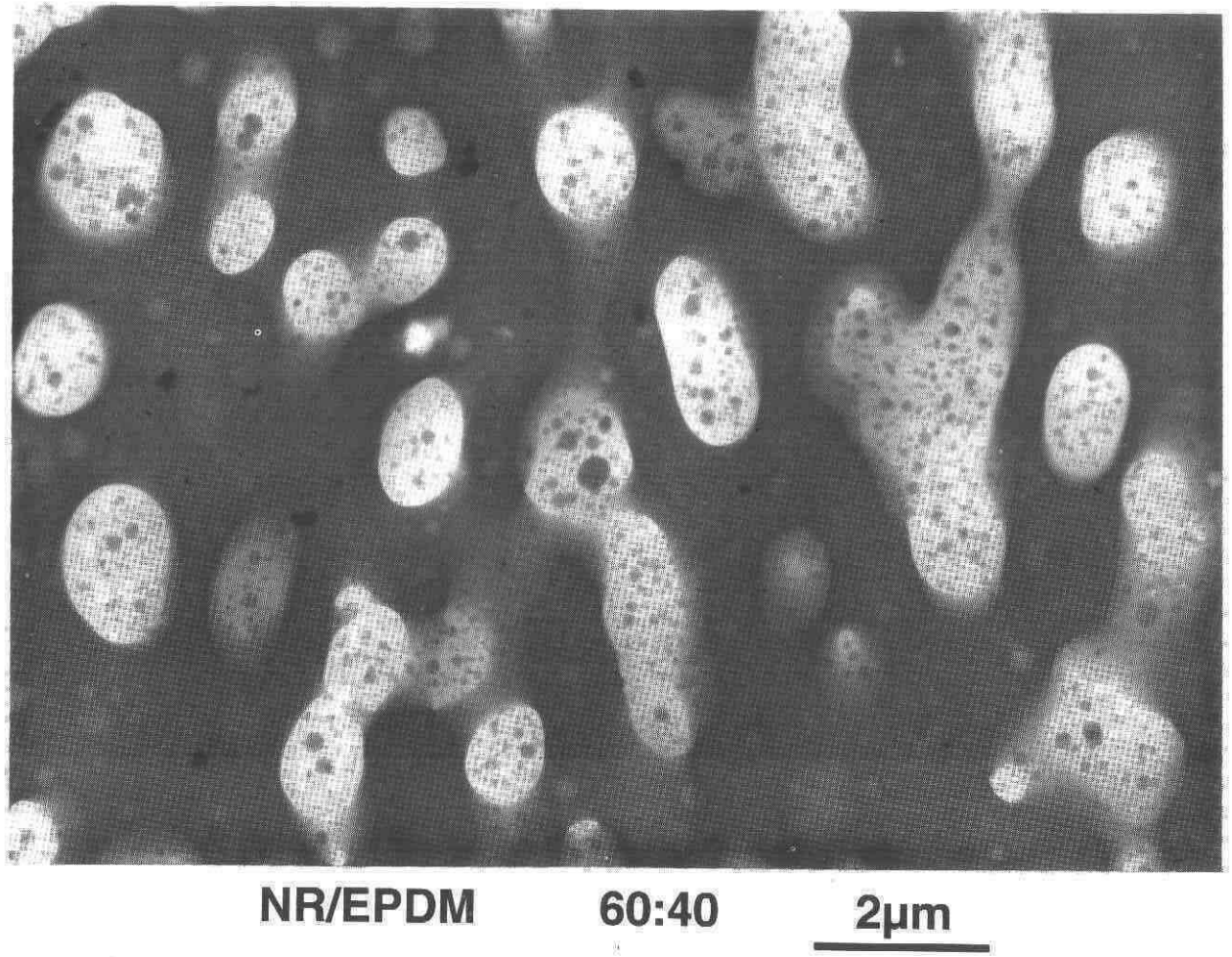

Figure 5. TEM micrograph of NR/EPDM 60:40.

used on the light microscope. The EPDM has not been stained by the osmium tetroxide and is the lighter phase. This micrograph is a good illustration of the point made at the beginning of this paper regarding thickness wherein the section is sufficiently thick for the three dimensional nature of the EPDM phase to be observed. Using one's imagination it is possible to mentally extrapolate to produce an image of the NR matrix with the EPDM phase tunnelling through it. A phase microstructure of small micro domains of stained NR sited within the unstained EPDM phase and *vice versa* can also be observed.

NR/CR/NBR

Rubber technologists will be aware of the problems involved in blending NR and NBR. A number of solutions have been successfully devised at MRPRA. One of these is the use of a compatibiliser, in this case chloroprene rubber (CR). The sample was prepared in the same way as the above example. Instrumental conditions were as above.

Light Microscopy

Figure 6 shows a phase contrast light micrograph of this material. A blend morphology can be observed but it is impossible to determine which phase is which since three phases are present but only two can clearly be seen. No conclusions can be drawn regarding the blend morphology or, more importantly, the phase structure of this material.

Transmission Electron Microscopy

Figures 7 and *8* show the same material at magnifications that are thirteen and forty times that of *Figure 6*. The morphology and structure of the blend are far more clear than in the LM micrographs and it can be seen that the CR resides at the interface between the lighter NR phase and the darker NBR phase. The CR can be identified as the darkest of the three phases. Its position is consistent with the expected position of a successful compatibiliser *i.e.* as the 'glue' between two largely incompatible materials. These results therefore confirm that the compatibiliser is working as intended. It would not be possible to obtain this information by light microscopy.

CONCLUSION

The above examples illustrate the increase in the level of information available by TEM by comparison to LM and how important good sectioning technique and an understanding of the processes involved is to successfully obtaining good images. Sectioning elastomers is a complex and time consuming process and despite the advances in preparative equipment there are still reasonable grounds for referring to this as being as much an art as a science. However, any intelligent operator with a reasonable level of dexterity, patience and understanding should be able to develop the necessary skills.

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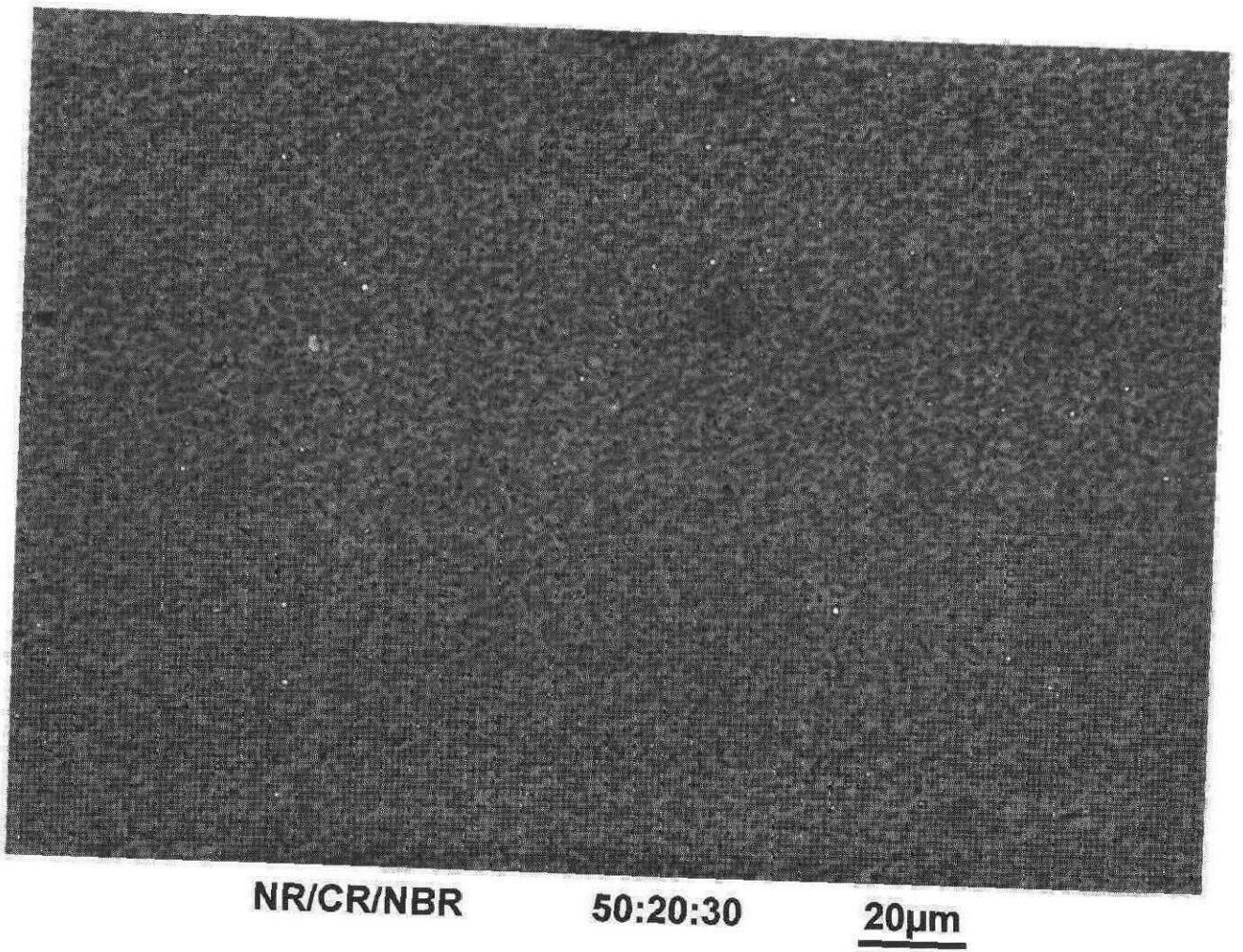

Figure 6. Phase contrast light micrograph of NR/CR/NBR 50:20:30.

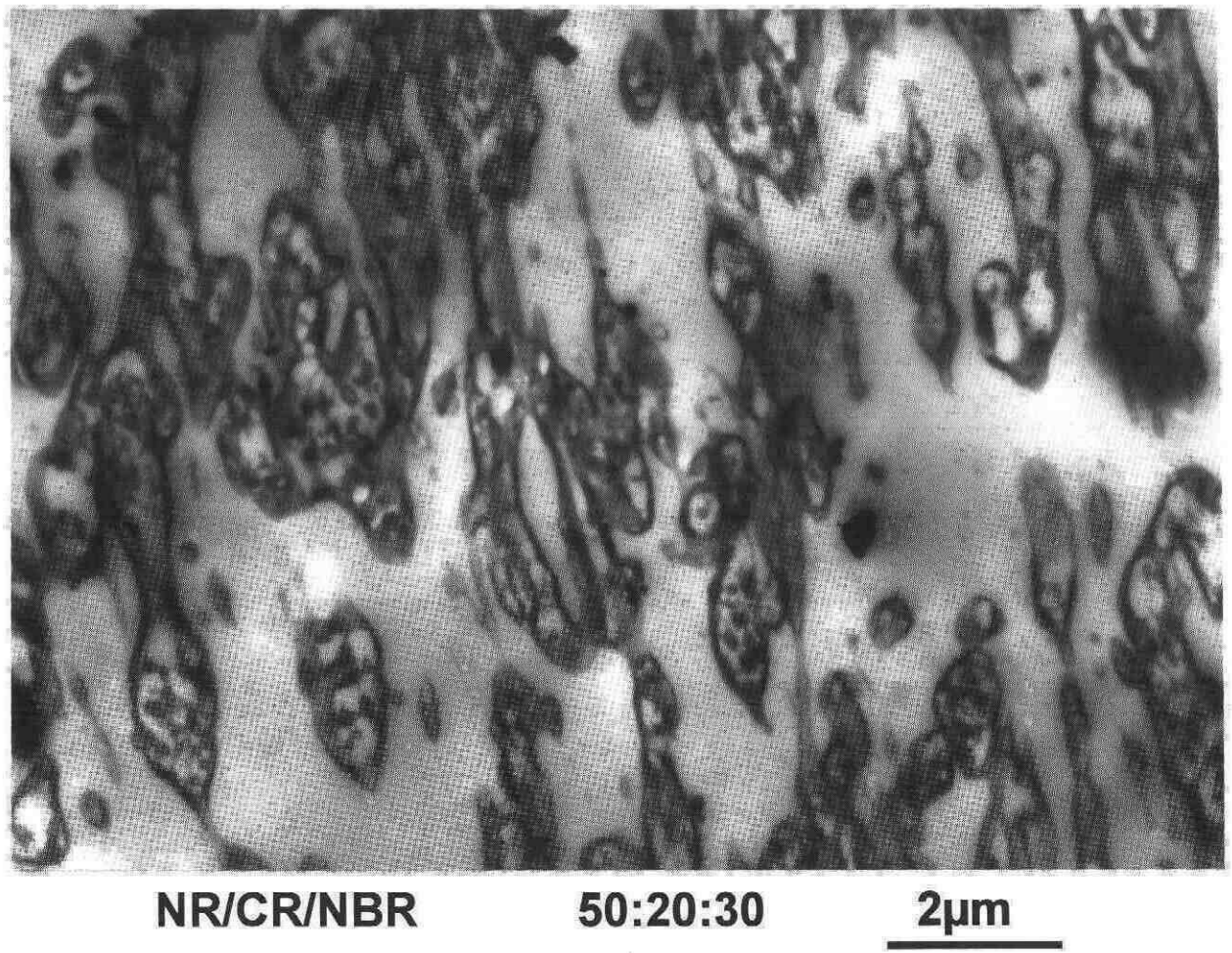

Figure 7. TEM micrograph of NR/CR/NBR 50:20:30.

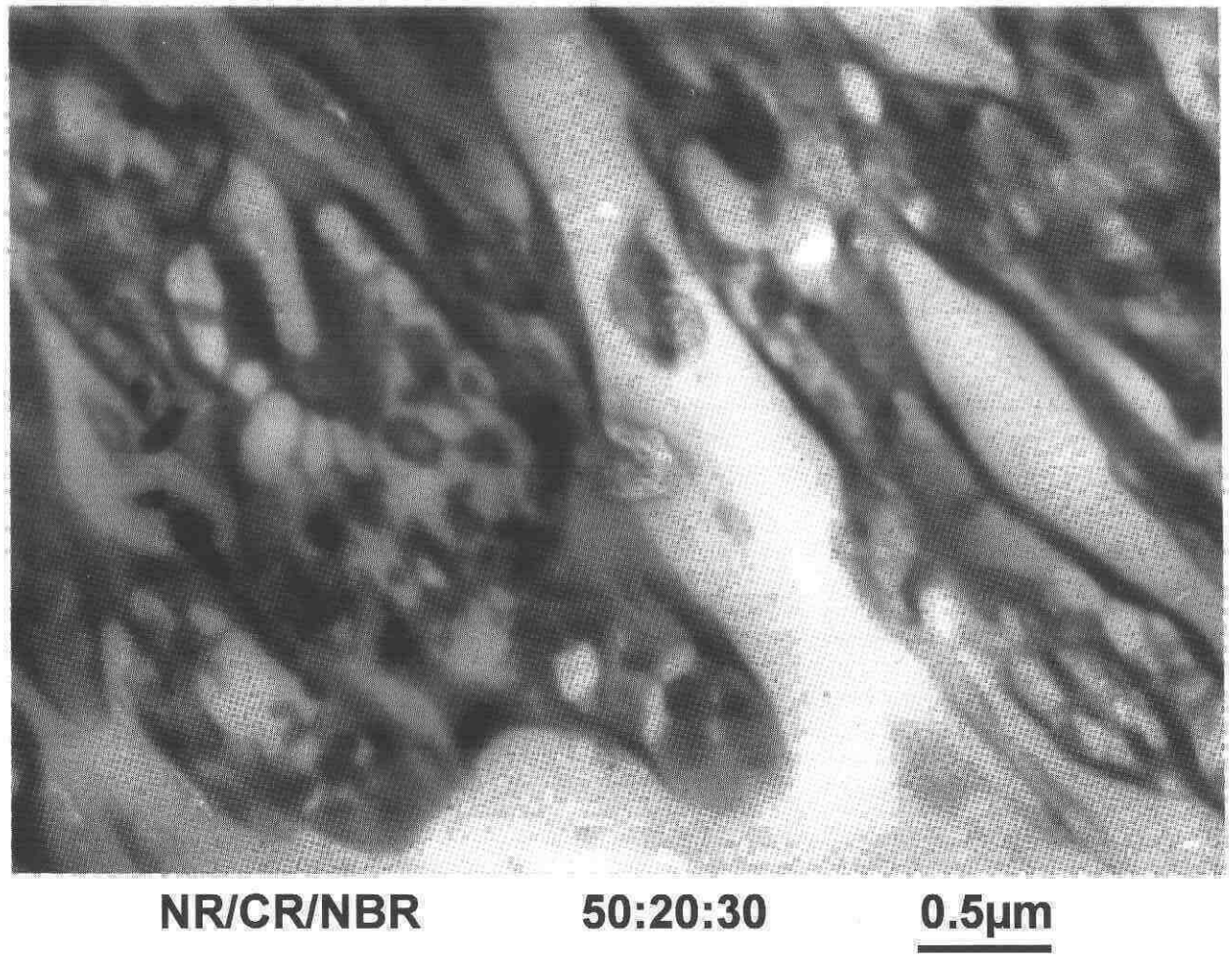

Figure 8. TEM micrograph of NR/CR/NBR 50:20:30.

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