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03

Specimen preparation for transmission electron microscopy of materials

Peter J. Goodhew



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The specimen: what we are trying to achieve

Electron microscopy is a powerful and fascinating tool for the investigation of structure and composition on a fine scale. Considerable skill is required to operate the microscope itself, to take high quality micrographs, and to interpret the resultant images. These topics are dealt with in companion handbooks. In this volume we shall be concentrating on the essential practical steps which must precede the microscopy itself and which are absolutely crucial to successful and meaningful use of a transmission electron microscope. Without a thin undamaged specimen even the most skilful microscopist is helpless.

Let us first state the primary objective of specimen preparation. It is to prepare and mount successfully in the microscope a thin specimen from which it will be possible to deduce accurately the structure, composition, and often also the behaviour of a larger sample of the material. There are several possible general approaches to achieving this objective. The most common is to thin a large piece of the material, for example by electropolishing, or to disperse it into pieces which are small enough to be transparent to electrons. This is the subject of Chapters 2 and 3. However, two alternative approaches are useful in a limited number of cases. It is sometimes possible to prepare the material directly in the form of a thin sheet, for example by vacuum deposition. Indeed, this often produces a marvellous specimen in all respects except the most important – it may not be representative of the material on a larger scale. However, if the object is to study thin deposited films then this simple technique is clearly highly appropriate. A third approach to specimen preparation is to extract from a larger specimen only certain components of its structure for study in isolation. This can be convenient but of course cannot give a total picture of the microstructure. The major technique of this type is carbon extraction replication, which is dealt with in Chapter 4.

1.1. Thinking ahead

Before deciding on a specimen preparation route (that is, if it is not already too late, before going straight to Chapter 3) it is wise to consider what transmission electron microscopy (TEM) techniques are to be used on the specimen. This will help to decide what are going to be the most important attributes of the final foil. For example, if large-scale low magnification information about the structure is required, uniformity of thinning and large size of thin area are paramount. On the other hand for weak-beam microscopy involving long exposures specimen stability

is very important. Again, if the specimen is to be analysed (by X-ray or energy loss techniques) it will be necessary to avoid segregation or preferential leaching of particular elements in the specimen. A further point is that if the electron beam is to be kept stationary on a very small region of the specimen (for microdiffraction, electron energy loss spectroscopy, or convergent beam diffraction) the specimen will need to be as free as possible from mobile surface contaminants. It is not easy to meet all these criteria so it is as well to determine in advance which is most important for a particular application.

A second way of thinking ahead is to assess the likely effect on the specimen of the various possible preparation procedures. This must be considered at all stages of preparation, and not just in the final stages when the specimen is obviously thin and delicate. Finally, it is clearly necessary to bear in mind what facilities and equipment are available locally. It is unlikely that all the equipment described in this booklet will be present in the laboratory. However, it can be demonstrated that a relatively modest outlay on reliable specimen preparation equipment is a good way of maximizing the effectiveness of the transmission electron microscope in every project. Most of the equipment described in this monograph costs less than 2 or 3 per cent of the value of the microscope it is intended to support.

1.2. The ideal specimen

The attributes of the perfect TEM specimen are listed in Table 1. Of course, these are never all realized simultaneously in practice; however, it is helpful to know what is being aimed at.

Table 1

The ideal TEM specimen is	Representative
	Thin
	Stable
	Clean
	Flat
	Parallel-sided
	Easily handled
	Conductive
	Free from segregation
	Self-supporting

Let us consider these attributes in turn. 'Representative' appears at the head of the list because without this all the other items are worthless. The specimen must accurately reflect the nature of the bulk material. We must be alive to the many possible ways in which the tiny area we finally photograph may not be typical. Among the more obvious problems are the introduction of dislocations or point defects due to mechanical damage during preparation, the loss of particular phases because of differential thinning, the unwitting selection of thin areas from only one phase in a two-phase material, or the non-random occurrence of particular crystal orientations in a thin polycrystalline foil. The most easily avoidable of these

problems, involving damage during preparation, are considered in the next two chapters as each technique is introduced. The more subtle problems of non-representativeness must be left to the intelligence and materials experience of the microscopist.

It seems obvious that a TEM specimen must be 'thin', but it proves rather difficult to quantify this statement. The concept of 'thin' must depend on the material (the electron penetration decreases as atomic number rises), on the accelerating voltage V available (useful penetration rises with V), on the imaging resolution required (high resolution requires very thin specimens), on the sideways spread of the electron beam that can be tolerated (in attempting for example to analyse a small region), on the size of second-phase particles which are required to be included within the foil, or on whether Kikuchi lines are required in the diffraction pattern. All that can safely be said is that the ideal thickness will probably lie between 100 Å (for lattice resolution) and several micrometres (for the high voltage electron microscopy (HVEM) of light elements). In practice most specimen preparation techniques lead to foils which taper from very thin to too thick, and the appropriate region for the type of image or analysis in hand must be chosen.

There are two aspects of specimen stability which are of concern. The response of the specimen to the electron beam should not lead to either chemical change or specimen drift. At the same time the ideal specimen should survive unchanged in air at room temperature for many years — it can then be re-examined at a later date. There is not a great deal which can be done about the latter — if mild steel is studied it will oxidize in damp air. The only solution is to keep prepared specimens in an appropriate atmosphere — in the case of mild steel in a desiccator in a refrigerator. Specimen stability in the microscope can be improved if the thin region of the specimen is supported by a much thicker region, as generally occurs with a disc specimen.

The cleanliness of a specimen is obviously of great importance since artefacts arising from dirty polishing solutions or simple laboratory dust are irksome and unsightly when superimposed on interesting microstructures. However, less obvious but more important are hydrocarbon layers which can give rise to contamination of beam-irradiated areas. Prevention of this effect remains one of the most difficult problems in TEM.

It is preferable for a specimen to be flat and parallel-sided for a variety of reasons. This will, in a crystalline specimen, eliminate thickness fringes and reduce the number of extinction contours. Also, if either the composition or the microstructure of the specimen is to be analysed quantitatively it is necessary to assume that the foil is of uniform thickness in the region photographed in order to arrive at the volume of the region. A similar consideration dictates that the specimen should contain no segregation of particular species to its free surfaces.

Finally, the specimen must be placed in and removed from the microscope without damage, and this is most easily achieved if it is self-supporting and can be handled with tweezers. If a support grid must be used then it will inevitably obscure some areas of the specimen (which, by Murphy's Law, will be the best thin areas).

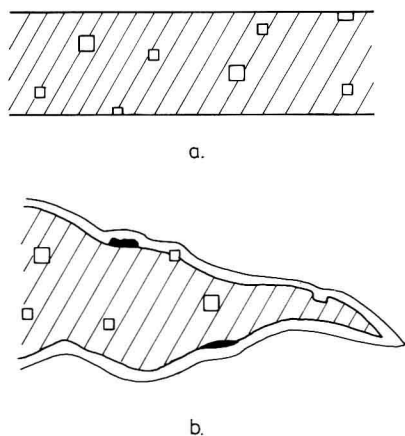


Fig. 1. (a) An ideal specimen which is flat, parallel-sided and undistorted. (b) A more usual specimen which tapers, droops and is covered with a layer of oxide, contaminant, and dirt. Some of its second-phase particles have been etched out and some stand proud of the foil.

If the specimen is electrically and thermally conductive then both charge build-up and too great a rise in specimen temperature can be avoided during examination.

It takes no clairvoyant to appreciate, before reading the rest of this monograph, that a specimen which fulfils all the criteria discussed above is only rarely produced. However, an appreciation of the ideal attributes of a specimen, and of the reasons why they represent an ideal, should help in the selection of techniques from the next three chapters which will be appropriate to the material under investigation and the reasons for its examination.

As a brief summary of this section a comparison of the 'ideal' and the 'typical' specimen is shown in Fig. 1. This is not intended to be entirely flippant!

Initial preparation of sheet or disc

The thinning of bulk material to electron transparency is generally achieved in two stages, which we shall call initial preparation and final thinning. Most of the final thinning techniques detailed in Chapter 3 work best on thin sheet or 3 mm diameter discs with flat or polished surfaces. In order to minimize the time taken by final thinning and to maximize the chances of ending up with a large parallel-sided thin area the initial preparation procedure should produce a sheet or disc about 0.1 mm (100 μm) in thickness. The lower limit on this thickness is set by the nature of the specimen material and its susceptibility to damage during preparation. A very hard material could usefully be prepared to a thickness of less than 50 μm , whereas a ductile single crystal will be difficult to thin below 200 μm without damage.

We shall consider initial preparation in three steps, illustrated in Table 2, although it is sometimes possible to omit some of these.

Table 2. *Common techniques for initial preparation*

(1) Cut slice	(2) Smooth faces	(3) Make disc
Saw	Hand polish	Punch
Acid saw	Mill	Spark machine
Wire slicer	Lap	Hollow drill
Slitting wheel	Chemical polish	Ultrasonic drill
Spark machine		Cut from rod
Cleave		
Microtome		

2.1. Cutting the slice

The first step is to cut a rough slice from the bulk specimen, bearing in mind when choosing the section that the final viewing direction will be perpendicular to the slice. At this stage the slice will probably have two rough surfaces and the thickness of the slice must be determined by the likely depth of damage caused by the chosen cutting technique. A conventional hacksaw, even with fine teeth, will probably do most damage and may easily affect the structure as far as 1 mm from the cut in a soft metal. Three less damaging techniques are the spark machine (used with a low energy spark), the diamond slitting wheel, and the rotating wire saw which carries an abrasive slurry through the specimen. The choice of technique depends on the material. Spark machining can only be applied to conductors and tends to be slow.

A slitting wheel can be much faster and is useful for non-conductors, but there is a risk of overheating the specimen. The wire slicer is a delicate device, widely used for cutting single crystals, but it imparts very little damage and progress can be monitored very easily. The least damaging, but most difficult, technique is the acid saw, in which a moving string carries acid (or some other appropriate solvent) through the cut. This is very gentle since the action is essentially chemical rather than abrasive, but it is consequently rather difficult to control and the preparation of a thin slice is not easy.

The four techniques are illustrated schematically in Figure 2. These devices are all available commercially in small sizes suitable for TEM preparation. A list of manufacturers and suppliers is given in the Appendix.

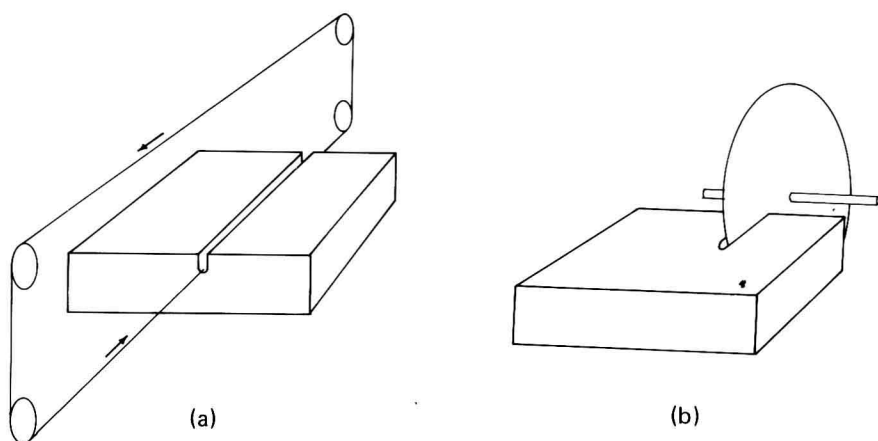


Fig. 2. (a) The acid saw, wire saw, and spark machine all employ a moving thread. In an acid saw this is a string which passes through an acid bath. In a wire saw it is a wire passing through an abrasive slurry. In a spark machine the whole device is immersed in paraffin and a spark is established between the moving wire and the workpiece. (b) A slitting wheel: the diamond-impregnated wheel passes through a slurry of lubricant and/or abrasive.

2.2. Preparing flat faces

Having cut a slice of thickness between 3 and 0.5 mm it is now necessary to prepare its faces as flat and parallel-sided as possible. The best way to achieve parallel faces is by machine milling or lapping, and the latter is preferable, if available, since the depth of damage can be reduced to quite a small value by using a fine abrasive. Certainly parallel-sided sheets of $100\mu\text{m}$ thickness or less can be produced from most materials by lapping with a 600 grit abrasive powder. Mechanical polishing by hand is a cheap and simple alternative to machine methods. If only a small sample is to be thinned (e.g. a 3 mm disc), then a very simple jig can be employed to control the thinning. Such a jig is shown in Fig. 3: it can be bought (see Appendix) or made in the workshop. With such a device it is possible to thin a disc to less than $50\mu\text{m}$ while maintaining highly parallel faces. The desired final thickness

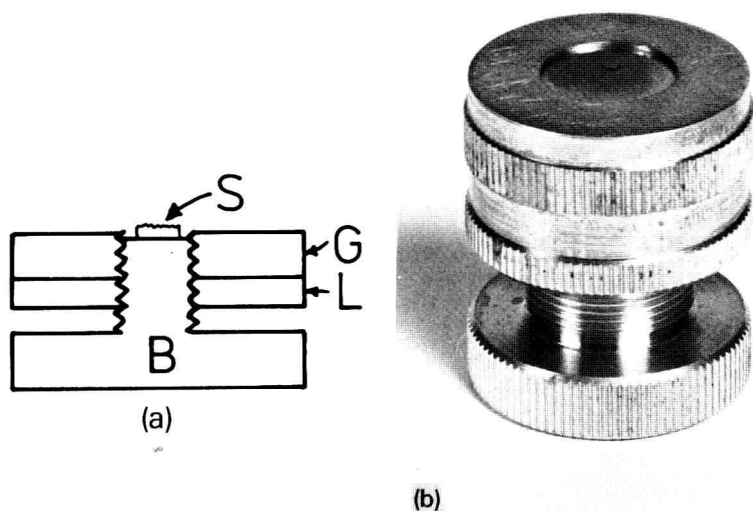


Fig. 3. A simple jig for hand polishing. The specimen (S) is glued to the central post (B) while a guard ring (G) is set to protrude by an amount equal to the desired final thickness. This is held in place by a lock ring (L).

can be set using the lock rings, and the thinning can then be done on conventional metallographic papers and wheels.

A major problem with all the mechanical techniques for producing smooth faces is to secure the slice to a substrate so that it can be handled. Each of the techniques described above can only be applied to one surface at a time, and the usual approach is to polish or lap one face and then to turn the slice over and re-fix it to the substrate with as thin a layer of adhesive as possible before finishing the second surface parallel to the first. Many adhesives are suitable but two of the most useful are a low-melting-point wax such as dental wax or a solution of polymethylmethacrylate (PMMA; Perspex) in ethyl acetate. In extreme cases double-sided adhesive tape can be used, but this is generally too thick and too soft for ideal security. Whatever adhesive is used, care must be taken to wash it off thoroughly or subsequent final thinning is likely to be uneven.

2.3. Chemical thinning solutions

The least damaging method of thinning a slice is by chemical polishing. This of course removes damage introduced by any previous mechanical stage but it is difficult to keep the faces of the slice perfectly parallel. Although chemical thinning machines have been built (see for instance Goodhew 1972) it is generally possible to polish both faces of a slice simultaneously in a simple beaker of thinning solution. If plenty of material is available, it is even possible just to immerse a slice in

the solution without any attempt to prevent preferential attack at its edges. The resultant slice is rather smaller but if the faces are acceptably polished this is not normally of great consequence. The key to successful chemical thinning is the correct choice of thinning solution. These solutions generally contain an acid, with if necessary the addition of a solvent for any solid oxides which may be formed. Table 3 gives a list of solutions which have been used successfully in the past. These should be considered as starting suggestions; it may be necessary to experiment with temperature or concentration in order to find the ideal for the material under investigation. Detailed references to the solutions listed in the

Table 3. *Chemical thinning solutions*

Material		Solution (use at room temperature unless otherwise stated)
Many metals		20% HNO ₃ , 80% methanol
Ag & alloys		50% HNO ₃ , 50% H ₂ O
Al & alloys	1	40% HCl, 60% H ₂ O + 5 g l ⁻¹ nickel chloride
	2	200 g l ⁻¹ NaOH in H ₂ O, 70°C
	F3	64% H ₃ PO ₄ , 18% HNO ₃ , 18% H ₂ SO ₄ , 80°C
	4	50% HCl, 50% H ₂ O + a few drops H ₂ O ₂
	5	94% H ₃ PO ₄ , 6% HNO ₃
Be	F	75% H ₃ PO ₄ , 7% chromic acid, 5% H ₂ SO ₄ , 13% H ₂ O, 60°C, slow.
Cd	1	20% HNO ₃ , 80% methanol
	F2	40% H ₃ PO ₄ , 60% H ₂ O
	F3	12 g KCN in 100 ml H ₂ O + 2 g Cd(OH) ₂
Co-Fe		50% H ₃ PO ₄ , 50% H ₂ O ₂
Cu & alloys	1	80% HNO ₃ , 20% H ₂ O
	2	50% HNO ₃ , 25% CH ₃ COOH, 25% H ₃ PO ₄
	3	40% HNO ₃ , 10% HCl, 50% H ₃ PO ₄
	4	60% CH ₃ COOH, 30% HNO ₃ , 10% HCl
Fe & steels	1	30% HNO ₃ , 15% HCl, 10% HF, 45% H ₂ O, hot
	2	35% HNO ₃ , 65% H ₂ O
	3	60% H ₃ PO ₄ , 40% H ₂ O ₂ , and other mixtures
	4	33% HNO ₃ , 33% CH ₃ COOH, 34% H ₂ O, 60°C
	5	34% HNO ₃ , 17% CH ₃ COOH, 32% H ₂ O ₂ , 17% H ₂ O; add H ₂ O ₂ just before use
	6	40% HNO ₃ , 10% HF, 50% H ₂ O
	7	5% H ₂ SO ₄ saturated with oxalic acid, 45% H ₂ O, 50% H ₂ O ₂ ; add H ₂ O ₂ just before use
	8	95% H ₂ O ₂ , 5% HF
GaAs, GaP	F1	1-4% Br in methanol
	F2	50% HCl, 50% HNO ₃
	F 6	42% HNO ₃ , 14% HF, 7% HCl, 37% H ₂ O
Ge	1	44% HNO ₃ , 26% HF, 27% CH ₃ COOH, 3% Br ₂
	F2	90% HNO ₃ , 10% HF
Mg & alloys	1	2-15% HCl in H ₂ O or methanol
	F2	2-15% HNO ₃ in H ₂ O or methanol
MgO		H ₃ PO ₄ , 100°C
Nb	F1	70% HNO ₃ , 30% HF
	2	34% H ₃ PO ₄ , 33% HF, 33% HNO ₃
	3	12% HNO ₃ , 11% HF, 27% H ₂ SO ₄ , 50% H ₂ O
Si	1	33% HF, 17% HNO ₃ , 50% CH ₃ COOH
	F2	90% HNO ₃ , 10% HF
Silica glass	1	35% HF, 50% CH ₃ COOH, 15% HCl
	F2	5% HF, 2% HCl, 93% H ₂ O
Ta	1	50% HNO ₃ , 50% HF
	F2	56% H ₂ SO ₄ , 22% HNO ₃ , 22% HF

Table 3. *Cont.*

Material		Solution (use at room temperatures unless otherwise stated)
Ti-Nb	1	75% HNO ₃ , 25% HF
	F2	25% HNO ₃ , 25% H ₂ SO ₄ , 25% HF, 25% H ₂ O, 10°C
U & alloys		50% HCl, 50% H ₂ O
UO ₂	1	25% HF, 25% HNO ₃ , 25% CH ₃ COOH, 25% chromic acid
	2	33% H ₃ PO ₄ , 33% HNO ₃ , 33% CH ₃ COOH
V		67% HF, 33% HNO ₃
Zn & alloys	1	60% HNO ₃ , 40% H ₂ O
	F2	30–50% H ₃ PO ₄ in ethanol
Zr, alloys	F	44% HNO ₃ , 12% HF, 44% H ₂ O

table can be found in the books of Goodhew (1972) and Thomas and Goringe (1980).

A few words of warning are necessary to those without experience in handling corrosive and oxidizing reagents, particularly those containing hydrofluoric and perchloric acids. Major points to be considered are listed in Table 4.

Table 4.

- (1) Look up any unfamiliar reagent in the laboratory safety handbook or a source such as Alderson (1975) or the *Handbook of Laboratory Safety*.
- (2) Remember that any reagent could be corrosive, poisonous, or explosive. Many electrolytes are unpleasant to inhale.
- (3) Always add acid to solvent, never solvent to acid.
- (4) Hydrofluoric acid (HF) penetrates both clothing and skin and is extremely dangerous. It attacks glass and must be used in polyethylene containers. It is essential to wear plastic gloves and apron.
- (5) Mixtures containing perchloric acid (HClO₄) are liable to explode on contact with oxidizable material (paper, wood, etc.) or even spontaneously. Never keep these solutions for long, even in a refrigerator.
- (6) Always label each solution with its composition and date.
- (7) Dispose of any unlabelled solutions.
- (8) Consult the laboratory supervisor to discover the procedures for disposal of waste solutions.
- (9) Use fume cupboards whenever possible.
- (10) In any case of doubt, ask for advice.

A particular problem with chemical thinning is stopping the reaction. Most solutions act quickly enough for the slice to be held with tweezers (preferably plastic!) so that it can be withdrawn from the solution to check that its faces are being polished rather than etched. A large beaker of solvent should be present beside the beaker of solution so that the slice can be immersed instantly to quench the chemical action. It is necessary to determine by trial and error how frequently the slice should be examined because in some cases it takes some time for the polishing action to be set up so that withdrawing the slice becomes counter-productive if carried out too often.

Table 5. *Names and formulae of reagents used in chemical and electrochemical thinning*

Name	Formula	Name	Formula
Acetic acid	CH_3COOH	Nitric acid	HNO_3
Acetic anhydride	$(\text{CH}_3\text{CO})_2\text{O}$	Ortho-phosphoric acid	H_3PO_4
Cadmium hydroxide	$\text{Cd}(\text{OH})_2$	Perchloric acid	HClO_4
Chromium trioxide	CrO_3	Potassium ferrocyanide	$\text{K}_4\text{Fe}(\text{CN})_6$
Ethanol	$\text{C}_2\text{H}_5\text{OH}$	Sodium hypochlorite	NaOCl
Hydrochloric acid	HCl	Sodium hydroxide	NaOH
Hydrofluoric acid	HF	Sulphuric acid	H_2SO_4
Hydrogen peroxide	H_2O_2	Water	H_2O
Methanol	CH_3OH		

2.4. Making a disc

Many automated final thinning techniques require a disc specimen 3 mm in diameter. Such a disc is easily handled, fits directly into the microscope without a grid, and provides good structural support for the thinnest areas of the specimen. It is occasionally possible to prepare the material initially as a rod of diameter 3 mm from which discs can be cut using a small slitting wheel or a dedicated mini-cut-off machine (see Appendix for suppliers). Such discs will usually be about 1 mm thick and can be thinned further using any of the techniques described in the previous section before final thinning. It is more usual, however, to dish ('dimple') them in the middle so that the outer rim remains 1 mm thick and can be handled with tweezers while the centre is 100 μm thick and is ready for final thinning.

Dimpling can be done by electropolishing or by ion bombardment, but in both cases it needs to be done more rapidly than final thinning or it will take too long. It is not necessary for the dimple to have a perfectly polished surface, and thus the dimpling procedure does not have to be as carefully controlled as does the final thinning. Automated electropolishers, such as the Tenupol described in the next chapter, generally have an operating manual which suggests appropriate electrolytes for rapid dimpling. Alternatively a specially designed ion-bombardment source (such as the Ion Tech 'Microrapid') which provides a high ion beam current and thus a fast thinning rate can be purchased. The temperature rise of the specimen must be watched, however, since a great deal of energy is concentrated in the disc by the ion beam. Ion thinning devices are described in more detail in Chapter 3.

If the material has been prepared as a slice then the 3 mm disc must be cut from a sheet of thickness about 100 μm . The quickest way to do this, if the material is ductile, is to punch out the disc using a hollow punch of internal diameter 3 mm. Such a punch needs to have some device for holding the sheet flat but this is not difficult to arrange. A simple jig made in the author's laboratory is shown in Fig. 4, but similar devices are also available commercially (Appendix). Clearly the punching action should not appreciably deform more than a thin rim of material

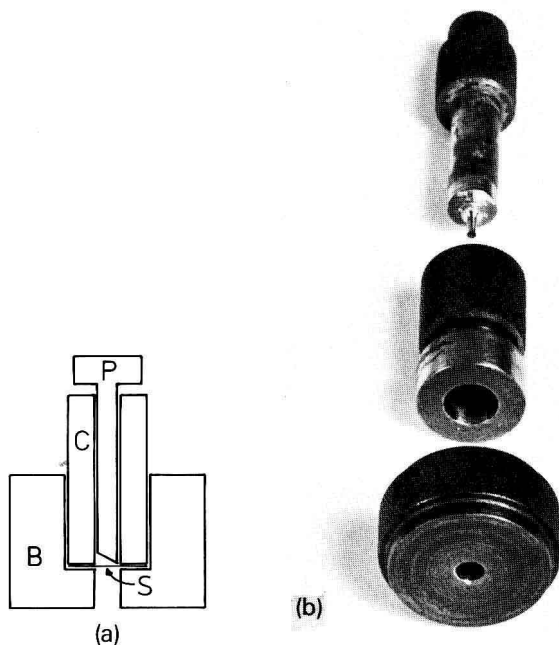


Fig. 4. A disc punch. The sheet specimen (S) is held down onto a base (B) by a block (C). The punch P runs through C and into a hole in the case when it is struck sharply with a hammer.

around the edge of the disc. This generally proves to be the case for ductile metals but the punching technique is not suitable for brittle materials. Unexpected damage may occur — it has been reported recently that after punching apparently successful discs from a steel, the thin area contained stress-induced martensite.

All the gentler techniques for cutting discs from sheet take much longer. The most commonly used methods use hollow drills. Diamond-tipped hollow drills can be purchased for use in conventional drilling machines or alternatively a tube of inside diameter 3 mm can be used in a spark machine. Finally, if the material is hard an ultrasonic drill with a hollow tool can be used. In such a machine the hollow tool is pressed very lightly onto the specimen sheet and is ultrasonically vibrated while immersed in a slurry containing abrasive powder. This works very well for silicon, for example, but is unsuitable for f.c.c. metals which simply have a circular groove forged into them.

The end product of initial preparation should be a parallel-sided sheet, a thin disc, or a dimpled thick disc. The techniques by which these can be finally thinned are the subject of the next chapter.

Final thinning

There are many ways of thinning a sheet or disc specimen down to its final electron-transparent thickness. Outside the biological sciences, however, two techniques dominate: these are electropolishing and ion beam thinning. This chapter therefore starts with a detailed description of methods based on these two approaches, continues with a shorter discussion of the less frequently used techniques such as chemical thinning and cleavage, and ends by considering how to thin specific regions of a specimen.

3.1. Electropolishing

The principles

The principle of electropolishing is quite simple. An electrolytic cell is established with the specimen as the anode and an appropriate potential is applied so that the specimen is dissolved in a controlled manner. Electropolishing is usually continued until a hole has formed in the specimen ('perforation'), when the regions around the hole should be thin enough for TEM. This sounds simple, and is simple once reproducible conditions have been established. However, there are so many experimental variables such as cell geometry, applied potential, and the composition, temperature, and velocity (if it is stirred or pumped) of the electrolyte, that some insight is needed into what should be happening in order to obtain good results quickly.

First consider what must be achieved. The electropolishing cell must polish the specimen, i.e. remove very fine-scale irregularities, it should smooth the specimen, i.e. remove larger-scale irregularities, and it must thin the specimen uniformly and fairly rapidly. The sequence is illustrated schematically in Figure 5. In order to produce this electropolishing action the electrolyte must generally contain an oxidizing agent together with reagents which will form a thin but stable viscous film. The fine polishing action is achieved by dissolution controlled by the length of the diffusion path through the viscous film to the electrolyte as shown in Fig. 6. High spots dissolve faster since they are nearer the free electrolyte and this results in a fine-scale smoothing which can generally be recognized as a brightening of the surface.

The viscous film must be kept thin and hence the electrolyte must contain, as well as an oxidizing agent and a film former, a solvent for the oxide-containing viscous film. Sometimes one reagent will act in all three ways and the electrolyte can be simple. An example is a dilute solution of perchloric acid in ethanol, which is close to being a universal electropolishing agent. On the other hand some