

Metallographic Sample Preparation Techniques

SAMAR DAS

National Metallurgical Laboratory, Jamshedpur-831007

E-mail : sdas@csnml.ren.nic.in

ABSTRACT

Microstructure plays an important role in controlling the properties in metals and alloys. Hence microstructural study called metallography has been extensively used for materials selection, failure investigation and materials development. The microstructure of metals are commonly observed under optical and/or electron microscopes, though other types of microscopes have been developed for specific uses. For any microscopic observation, the preparation of proper sample, which reveals the true microstructure is of primary importance. Numerous and diverse techniques have been developed not only to suit the material but also for the type of microscope to be used. The techniques also vary with the details to be observed. It is difficult to prepare a comprehensive survey of the techniques developed and practised today. An attempt has been made here, to briefly discuss the most commonly used specimen preparation techniques for optical, scanning electron and transmission electron microscopy together with their merits and demerits. Any specimen preparation method involves several steps. Proper care in each step is essential to avoid difficulty in the subsequent steps and to reveal the true microstructure. The choice of a technique depends mainly on the materials, the detail required to be observed and the type of microscope to be used.

INTRODUCTION

The study of microstructural details of metals and alloys is termed as metallography. The microstructure of steel was first observed by H.C. Sorby in 1864 at a very low magnification. Presently several types of microscopes such as transmission electron microscope, scanning electron microscope, Field ion microscope, Auger Microscope, Scanning Tunneling Microscope, Ultrasonic Microscope, Acoustic Microscope etc, are available for micro-structural, micro-chemical and crystallographic characterisation of metals and materials.

The type microscope used for characterisation depends upon the fineness and type of details required to be observed. For micro-structural characterisation of

metals and alloys Optical, Transmission and scanning electron microscopes are mostly used.

Modern optical microscopes with resolution limit of 200 nm and magnification upto 2000 x are used to observe details like grain shape and size, morphology of inclusions and precipitates, micro-segregation, micro-cracks, surface coating thickness and structure, weld defects etc. in metals.

Scanning electron microscope with resolution upto 5nm and magnification nearly 2,00,000 times are used for observing much finer details. Due its high depth of field, the scanning electron microscopes are extensively used in fracture study called fractography.

Transmission Electron microscopes with resolution limit upto 0.2nm and magnification upto 8,00,000 times are used for observing crystal defects such as grain boundary, dislocations, stacking faults, twinning etc. The capability of transmission electron microscope in producing selected area diffraction pattern helps in characterising such crystal defects.

Whatever may be the microscope used, the success of any metallographic study is primarily dependent on proper sample preparation. There are different types of sample preparation techniques for different types of microscopes. These techniques may vary with the sample material. Some common techniques used for sample preparation for optical metallography, scanning electron microscopy and transmission electron microscopy are discussed here.

SPECIMEN PREPARATION FOR OPTICAL METALLOGRAPHY

The primary requirement of an optical metallographic sample is a representative smooth and flat surface etched to reveal the micro-structural details. The size of the sample should be compatible with the microscope stage.

The steps involved in preparing such sample are (i) Cutting (ii) Mounting (iii) Grinding (iv) Polishing and (v) Etching.

Cutting : A sample of size nearly 20mm x 20mm x 20mm is cut from a representative area of bulk material. Such a size facilitate the handling during grinding and polishing operations. Care is taken not to alter the microstructure due to generation of excess heat or, application of high stress. Hand sawing, abrasive cutting with proper cooling and chemical or, electro chemical sectioning are preferred to faster cutting processes such as flame cutting, laser cutting, electro discharge machining etc.

Mounting : Embedding of sample in either hot or cold setting resin is called mounting. Mounting is necessary when (a) sample size is too small for subsequent preparation steps (b) Preservation of sample edge is must (c) A par-

ticular face of sample is to be observed (d) Multiple number of samples are to be prepared and observed in a single mount (e) Oblique mounting is required to magnify thin coatings. In hot mounting, the sample along with the resin powder is subjected to pressure ($\sim 30\text{MPa}$) and temperature ($\sim 150^\circ\text{C}$). Thermosetting phenolic resins cures and hardens with time, where as thermoplastic resins solidifies when cooled to room temperature.

In cold mounting a homogeneous liquid mixture of resin (monomar) and catalyst is poured into a metallic or, phenolic ring placed surrounding the sample on a glass slide. The two chemicals react at room temperature and solidify embedding the sample.

Cold mounting is used when the sample is porous or contains cracks. In vacuum impregnation the pores and cracks are filled without any air bubble. Hot mounting is preferred because it is faster and economically cheaper, provided the applied pressure and temperature does not alter the structure.

The mounting materials should be chemically inert to the sample, etchant or, other materials used during grinding and polishing. It should have low shrinkage, moderate viscosity and form bubble free solid to provide good bonding strength to the sample.

Grinding : The purpose of grinding is to remove the deformed materials and reduce the surface roughness caused by cutting operation. Successive grinding with coarse to fine abrasive particles results in a flat surface with fine scratches. Rotating wheels with adhesive backed emery papers, vibratory platens with emery powders or, hand grinding emery papers fixed on glass plates are conventionally used in grinding. Emery papers of grit sizes 60, 120, 220, 320, 400 and 600 are used successively in six steps. A 90° change in direction of abrasive cutting and cleaning of the surface, in between each step, produces better result in less time. Use of moderate pressure is preferred to minimize the deformed metal layer. The use of lubricant reduces the chance of embedding abrasive particles to soft metal (such as Al, Cu etc.) surfaces.

Polishing : Either manual or, electrolytic polishing is carried out to produce a perfectly flat mirror like smooth surface by removing the fine scratches produced during grinding.

Manual polishing is done by holding the flat ground surface on a cloth covered rotating wheels smeared with abrasive (Al_2O_3) suspension. $20\mu\text{m}$ and $10\mu\text{m}$ Al_2O_3 particle suspension and $5\mu\text{m}$ diamond paste is used in three successive polishing steps. Use of proper polishing cloth, appropriate speed of rotation and rotation of sample in a direction opposite to the rotation of wheel, produces a defect free perfectly smooth surface with minimum smeared metal due to plastic flow.

Electrolytic polishing is carried out by anodic dissolution in electrolyte cell. The cell consist of a container filled with electrolyte and stainless steel or platinum cathode. The sample as anode is placed in the cell and D.C. supply is connected to cathode and anode. Selective anodic dissolution of protrusions of grinding scratches, results in a mirror like smooth surface. The success of an electrolytic polishing depends upon.

- (1) Proper electrolyte bath composition and temperature
- (2) Proper voltage and current density
- (3) Uniform continuous agitation of bath
- (4) Proper timing for polishing
- (5) Proper shape size and orientation of cathode with respect to anode (sample)

Electrolyte bath composition depends on sample composition. It is generally a mixture of acids, ionizing solution and liquid medium for controlling the viscosity of electrolyte bath. Table-1 gives some suitable electrolytes for polishing common metals and alloys.

Table -1 : Electrolytic Polishing Baths

Material	Bath composition	Bath Temp.	DC Voltage
Aluminium and its alloys	HClO ₄ (70%) 20 C ₂ H ₅ OH - 80 ml	< 0°C	12-17
Copper and its alloys	HNO ₃ (conc.) 33 ml C ₂ H ₅ OH 67 ml	< -30°C	8-16
Iron, Steel and stainless steel	HClO ₄ (70%) 10 ml CH ₃ COOH (Glacial) 90 ml	~ 10°C	10-25
Titanium and its alloys	HClO ₄ (70%) 6 ml C ₂ H ₅ O ₄ 60 ml n-Butanol 34 ml	< -25°C	11-20

The current density versus voltage curve ABCD shown in fig. 1 depicts the plateau region BC suitable for electropolishing. Voltage below this causes etching, where as above this results in pitting of the surface. The curve EF shows the absence of plateau region due to high temperature of electrolyte.

The advantages of electrolytic polishing over mechanical polishing is that it produces a perfect smooth surface, free from deformed and flowed material layers rapidly without any surface heating. The chances of embedding abrasive particularly in soft metal matrix is avoided. But the edges and boundaries of imperfection like inclusions, cracks, etc. are dissolved at a faster rate than rest

of the matrix causing artefacts. The reaction products sometimes adhere to the surface which are difficult to remove by washing. Immediate washing the surface in running water and drying with alcohol is necessary to avoid further chemical attack and to produce a spotless reflecting surface.

Etching : Features, such as, pores, pits, cracks, inclusions and relief formation due to difference in hardness are visible in optical microscope in the as polished sample. But other microstructural details, such as, grain boundary, precipitate, etc., are revealed on the polished surface by a technique called etching. For metals and alloys three different types of etching employed are (i) Optical etching (ii) Chemical/Electrochemical etching and (iii) Physical etching.

Optical etching : Special optical techniques, such as, Dark field illumination, Phase contrast microscopy, Polarization microscopy and Interference microscopy are employed to make the microstructural features visible under a microscope. Further treatment of polished surface are not generally required, but the microscope must have the capability to adopt the special illumination and compension techniques.

Chemical etching : Treatment of the polished surface with chemical solvent (etechant) causes differential dissolution of microstructural components having different chemical composition. This is conventionally termed as chemical etching. The dissolution rate varies because of difference in electro-chemical potential of different phases with respect to standard potential of reference electrode. The differential chemical attack also occur at crystal imperfections such as grain boundaries, dislocations, deformed zones etc. Such varying chemical attack reveals the microstructural details under reflected light microscope. The list of chemical echants is given in Table 2.

When an external D.C. voltage is applied in this process with sample as anode and stainless steel or, platinum cathode electrolytic etching takes place. The same electrolyte used for electrolytic polishing is used with voltage range corresponding to the initial portion AB of the current density versus voltage curve shown in fig.1 When deposition of oxide layers and precipitates take place on polished surface a difference in colour is observed due to interference effect. This technique is called colour etching.

Physical etching : Revealing microstructural details by application of heat, sputtering or, bombarding by ion beam are classified as physical etching.

Surface diffusion, selective evaporation and condensation of matter towards low energy areas reveals the grain-boundaries in a heated polished surface. This is known as thermal etching.

Table 2 : Etchants for common metals

Material	Etchant	Time
Aluminium and its alloys	a) HF (40%)	0.5-10 ml
	H ₂ O	100 ml
	b) HNO ₃ (Conc.)	5-10 ml
	HCl (Conc.)	3-6 ml
	HF (40%)	2-4 ml
Cu and its alloys	H ₂ O	190 ml
	a) HCl (Conc.)	20-50 ml
	Iron (III) Chloride	5-10 ml
	H ₂ O/C ₂ H ₅ OH	100-120 ml
Cast iron, plain	a) HNO ₃ (Conc.)	1-10 ml
Carbon steel, Alloys steels	C ₂ H ₅ OH/CH ₃ OH	100 ml
	b) C ₂ H ₅ OH	100 ml
	Picric acid	2-4 gms
Stainless steels	HNO ₃ (Conc.)	15 ml
	HCL (Conc.)	30 ml
	Glycerol	45 ml
Titanium and its alloys	HNO ₃ (Conc.)	2-6 ml
	HF (40%)	1-3 ml
	H ₂ O	100 ml
Nickel and Nickel base super alloys	HCl (Conc.)	2-25 ml
	Iron (III) Chloride	5-8 gm
	C ₂ H ₅ OH	20-100 ml

When a polished surface is subjected to ion bombardment, selective dissociation of areas of different chemistry reveals the microstructure. The method is termed as Ion-beam etching. After the etching is over the specimen must be washed in alcohol to make the surface free from any chemicals and dried. When colour etching by deposition of oxide/precipitate layers are done, care should be taken not to wash off the layer.

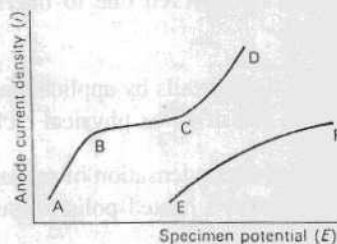


Fig. 1 : Current density versus voltage curve

SAMPLE PREPARATION FOR SCANNING ELECTRON MICROSCOPY (SEM)

Scanning electron microscopes are extensively used for

- i) Fracture Study
- ii) Microstructural study
- iii) Study of shape and size of powder particles and
- iv) Study of replicas

For fractography the sample is cut to suit the specimen holder. Care must be taken not to alter the fractured surface during cutting. It is then cleaned with some solvents e.g., alcohol, etc. to remove dust, oil, grease or, any extraneous material. Use of ultrasonic cleaner gives a better cleaning. If the sample is a non-conductor, it is made conductive by coating the surface with carbon or gold using vacuum/sputter coater. It is then screwed to the sample holder without touching the fractured surface. If necessary electrical conductivity between coating surface and holder is made by applying conductive carbon or silver paste.

For microstructural study, if the sample size is large a smaller representative sample is cut by some suitable method. In case of very small samples conductive mount can be made using resins with metal fillers. The sample is then ground and polished to obtain a flat polished surface. Details like, pores, microcracks, cavities and inclusions can be observed in the as polished sample. As polished surface can also be examined in SEM backscattered mode to get a compositional variation contrast.

The methods described in optical specimen preparation for mounting, grinding and polishing are also applied for SEM specimen preparation. Since an electropolished surface is free from deformed and flowed material layer, this method is preferred to manual polishing methods.

For microstructural examination using SEM secondary emission mode, the polished surface is etched either chemically or electrolytically in a similar manner as in case of optical microscopy. A deeper or stronger etch gives a better contrast.

If in-situ replicas are to be observed in scanning electron microscope, the replica is cut to a suitable size by sharp scissors or scalpel blades and fixed onto a SEM stub, with the replicated surface upwards. A thin uniform conductive coating on the surface is necessary to avoid charging during observation in SEM.

Powder particle specimens are made by dispersing a small quantity of powder in an unreactive solvent using an ultrasonic vibrator. One or, two drops of suspension is dropped on a clean glass slide and the solvent is allowed to evaporate. A piece of

self adhesive conducting tape is pressed uniformly on the slide and stripped out. The tape is then fixed on a SEM stub, keeping the particle containing side upwards. In case the particles are non-conducting a conductive coating is given as usual. If the particles are very discrete and does not have the tendency to agglomerate, the particles can be dispersed in the following way. A small quantity of powder is taken on cotton swab tip. The particles are then dropped on stub surface, smeared with adhesive, by holding the swab tip over it and giving a little jerk. Once the adhesive gets dried conductive coating is applied.

Powder particles can also be mixed uniformly with cold setting resin and a cold mount is prepared. The mount is then ground, polished and coated with conductive coating.

In all conventional scanning electron microscopy specimens, the following care must be taken.

- i) The specimen size should be compatible to the specimen holder used.
- ii) The specimen must be cleaned properly to make it free from any extraneous material.
- iii) The specimen surface should be conductive and connected to the holder to avoid charging effect.
- iv) The specimen should not contain any easily volatile components which evaporate due to vacuum in the specimen chamber.

Recent developments in scanning electron microscopes however rules out some of the above restrictions, such as environmental scanning microscopes allows the specimen chamber to be kept at a low vacuum and improved detectors allows badly conducting specimens to be observed without any conductive coating.

SPECIMEN PREPARATION FOR TRANSMISSION ELECTRON MICROSCOPY (TEM)

In transmission electron microscopy, the incident high energy electron beam, while transmitting through an electron transparent thin specimen, gets diffracted, where it encounters defects, such as, grain boundary, precipitate-matrix interface, twin boundary, dislocations, stacking faults etc.

The specimen should be thin enough, roughly 1000Å to 2000Å thickness, to allow sufficient amount of electrons to pass through the specimen. Otherwise more absorption of electron in the specimen results in a poor quality image. Generally lighter elements and higher energy beams allows higher side of thickness.

Preparation of such thin specimens involves several steps and chances of altering the microstructure or, introducing defects, called artefacts, are more. Due care is needed in each step to avoid such artefacts.

Among the three types, namely, thin film, thin foil and replica, of transmission electron microscopy specimens, thin foils and replicas are widely used for metallographic study.

Thin films can be prepared by physical vapour deposition technique. A measured quantity of material is heated sufficiently to melt in vacuum ($\sim 10^{-5}$ torr) using a vacuum evaporator. Thin film is formed by the deposition of vapour on a clean substrate. Properly cleaned glass slide, coated with some easily dissolving compound (e.g. NaCl or, soap solution) or a freshly cleaved crystal surface can be used for a substrate. Quartz crystal thickness monitor or, Angstromscope can be used to measure the film thickness. Film thickness can also be calculated by the formula

$$t = \frac{m}{4\pi r^2 \rho}$$

where t = thickness of film, m = mass of evaporant, r = distance between heater and substrate, ρ = density of evaporant

The film can be separated from substrate by dissolving the coating or, the crystal in some suitable solvent. The floating film is fished out of solvent on TEM grids and dried.

RF and DC sputtering, Ion beam sputtering, electron beam evaporation are some of the other methods, used for thin film preparation.

Carbon support grids for powder specimens are also prepared in vacuum evaporator with carbon arc attachment. Carbon film thickness can be calculated from the relation

$$t = \frac{d^2 l}{16 r^2}$$

where t = thickness of film, d = diameter of carbon rod tip, l = length of carbon rod evaporated, r = distance between arc and substrate.

Metallic thin foils are most exclusively used for microstructural study by transmission electron microscopy. The steps involved in making thin foils are (i) cutting (ii) preliminary thinning and (iii) final thinning.

The aim of cutting is produce a thin slice ($1\text{ mm} > \text{thickness} > 0.5\text{ mm}$). Electro discharge machining, Ultrasonic cutting, Diamond sawing, chemical sawing are some of the techniques which can be used to prepare a thin slice. In case of metals and alloys, low speed diamond sawing with proper cooling or, chemical sawing are preferred for producing a thin slice with minimum deformed layer and minimum generation of heat to alter the microstructure.

The cut slices are further thinned to a thickness of $\sim 0.1\text{ mm}$ either by, hand grinding on emery papers successively from coarse to finer grades or by chemical dissolu-

tion by dipping in suitable chemicals Table 3 gives some chemical solutions for a few common metals. Other chemical solutions can be found from the reference books at the end or from literature. Chemical dissolution is faster method but chances of forming pits are more. Alternate use of chemical dissolution and hand grinding produces better result in less time. Preliminary thinning is carried out till the thickness reaches nearly 0.1 mm. The thin slice is then cleaned properly and subjected to electrolytic polishing for final thinning.

Table 3 : Solutions for chemical dissolution

<i>Material</i>	<i>Solution Composition</i>	<i>Solution condition</i>
Aluminium and its alloys	NaOH, 20 gms, H ₂ O, 100 ml	Soln. temperature ~70°C
Copper and its alloys	HNO ₃ (conc.) 80 ml H ₂ O 20 ml	Soln. to be agitated vigorously
Copper-Aluminium alloys	HNO ₃ (conc.) 40 ml HCl (conc.) 20 ml H ₃ PO ₄ (conc.) 50 ml	—
Iron Carbon and alloy steels	HNO ₃ (conc.) 30 ml HCl (conc.) 15 ml HF 10 ml H ₂ O 45 ml	Soln. temperature ~ 70°C

The electrolytic polishing technique in general has been described earlier in optical specimen preparation methods. Three variations of this technique when employed for making electron transparent thin metallic foils are (i) Window technique (ii) Bollmann technique and (iii) Jet thinning technique.

For window technique a thin slice of size nearly 20mm x 10 mm x 0.1 mm is most suitable. A protective layer of lacquer of about 1mm to 2 mm width is applied around the edges, to avoid faster thinning at the edges. This is used as anode in the electrolytic cell as shown in fig. 2. After sometime of switching on the DC supply, perforations in the sample at the top level of electrolyte are formed, due to preferential dissolution. The sample is then withdrawn from the electrolyte and current is switched off. The sample is inverted and reintroduced into the bath and circuit switched on. Now preferential dissolution starts from top and bottom of sample and is allowed to continue till a narrow bridge portion is formed in the central zone of sample. The sequence of thinning is shown in fig. 3. The sample is taken out and circuit switched off. The sample is washed by carefully dipping in alcohol keeping the thin narrow bridge portion intact. This bridge portion is then cut with sharp scalpel, washed and dried using alcohol. It is sandwiched between two TEM grids for observation.

In the Bollmann technique, the cathode configuration is different. Two sharp pointed cathodes are placed on both sides of the sample (anode) as shown in Fig. 4. Initial perforation takes place in the anode near the cathode tips. The specimen is then shifted to form another hole in the nearby region. It is repeated to form several holes around the initial perforation and the bridge portions between the holes are cut by scalpel to get a number of electron transparent thin samples. The cut bridge portions are washed, dried and sandwiched between two grids for putting into TEM sample holder.

In jet polishing two cathodic jets of electrolyte are directed towards the centre of a 3mm dia x 0.1mm thick sample as anode. Once the cathodic jets hits the anode maximum dissolution at the centre of specimen takes place, causing a hole to be formed there. A schematic diagram of twin jet thinning unit is shown in fig. 5.

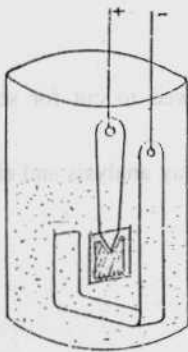


Fig. 2 : Electrolytic thinning bath in window technique

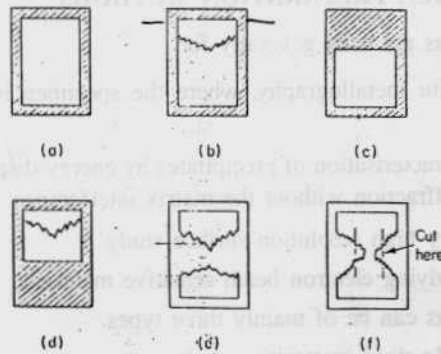


Fig. 3 : The sequence of operation in window technique

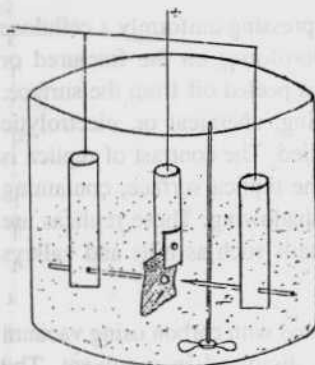


Fig. 4 : Electrolytic thinning bath in Bollmann technique

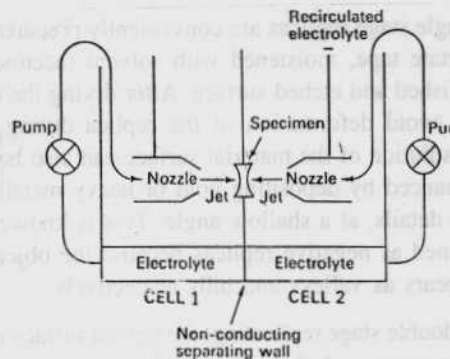


Fig. 5 : Schematic diagram showing twin jet thinning apparatus

A 3 mm disc punched out of preliminary thinned slice is cleaned and put into the anode holder. The anode holder is then slid into the jet chamber and electrolyte is pumped into the side chambers. The electrolyte passes through the small holes with cathodic connections, as jets and hit the sample centre from both side. Either a light source and photocell detector or Infra red source and IR detector placed on both sides of the sample detects any small perforation in the anode and automatically switches off the circuit and gives alarm to the operator. After repeated cleaning in ethanol and drying, the sample can be directly put in the TEM holder without any support grid.

Due to action of both jets a double concave type contour is formed in the central zone of anode disc and ultimately forms the perforation. The areas around the edges of this central perforation contain electron transparent thin areas.

REPLICA PREPARATION METHODS

Replicas are used generally for

- i) In-situ metallography, where the specimen is not allowed to cut for some reason.
- ii) Characterisation of precipitates by energy-dispersive X-ray analysis and electron diffraction without the matrix interference.
- iii) Very high resolution surface study.
- iv) Studying electron beam sensitive materials

Replicas can be of mainly three types

- i) Single stage or positive replica
- ii) Double stage or, negative replica
- iii) Extraction replica

Single stage replicas are conveniently prepared by pressing uniformly a cellulose acetate tape, moistened with solvent (acetone/chloroform) on the fractured or polished and etched surface. After drying the tape is peeled off from the surface. To avoid deformation of the replica during peeling, chemical or, electrolytic dissolution of the material surface can also be applied. The contrast of replica is enhanced by depositing gold or heavy metal on the replica surface, containing the details, at a shallow angle. This is known as shadowing. These replicas are termed as negative replicas because the object details such as hills and valleys appears as valleys and hills respectively.

In double stage replicating, the replica surface is coated with carbon using vacuum evaporator and then the cellulose acetate tape is dissolved in a solvent. The carbon replica floating on the solvent is washed carefully in some fresh solvent and fished out on TEM support grids.

For making extraction replica, carbon is deposited on the properly polished and etched surface in vacuum evaporator. The coated surface is then carefully scratched in grid formation by a sharp scalpel. The sample is then treated with a strong etchant to dissolve the matrix in contact with carbon film. The precipitates do not dissolve and comes off with carbon film floating in the etchant. The sequence of operations in extraction replica preparation is shown in Fig.6. These replicas are then transferred to alcohol containing dish and finally fished out on TEM grids and allowed to dry. By this method a carbon replica in which the carbides and precipitates are embedded obtained.

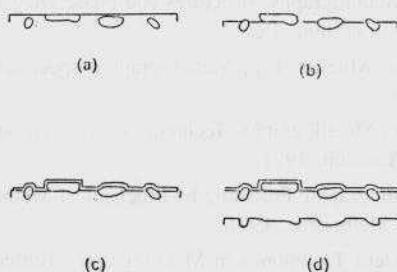


Fig. 6 : The sequence of operation in extraction replica preparation

In replica making steps, proper care is necessary to avoid introducing artefacts into the replica.

CONCLUSION

Since several techniques are available for metallographic sample preparation, one has to be very careful in selecting a technique. The main criteria which governs the choice of a technique are

- i) Material composition, its shape and size
- ii) The purpose of investigation
- iii) The details required to be observed or recorded
- iv) The type of microscope to be used for investigation
- v) The consumables and instruments available for preparation.

Once a technique has been selected, proper care in each step is needed for

- i) Final true microstructure and
- ii) To avoid difficulty in the subsequent steps.

REFERENCES

1. ASTM Standards, Standard Methods of Preparing Metallographic specimens, E-3-86. American Soc. for Metals, Vol. (03.01), 1986.
2. G. F. Vander Voort, Metallography principles and Practice, MacGraw Hill Book Company, 1984.
3. Gunter Petzow, Metallographic Etching, American Soc. for Metals, 1978.
4. K. C. Thompson, Russel and J. W. Edington, Ed. Practical Electron Microscopy in Materials Science, Monograph 5, Mac Millan Philips Technical Library, Eindhoven, Netherlands, 1975.
5. Metals Handbook, Metallography, structures and phase Diagrams. American Society for Metals, Vol. 8. 8th edition, 1973.
6. J. L. McCall, W. M. Mueller, Ed., Metallographic specimen Preparation, Plenum Press, New York 1971.
7. V. A. Philips, Modern Metallographic Techniques and Their Application, Willey Inter Science. New York/London, 1971.
8. L. E. Samuels, Metallographic Polishing by Mechanical Methods, Pitman Publishing Corp., London and Melbourne, 1971.
9. D. G. Brandon, Modern Techniques in Metallography, Butterworth Publishing Co., London, 1966.
10. ASTM Special Publication, Application of Modern Metallographic Techniques, No. 480, Symposium, Philadelphia, 1966.
11. P. B. Hirsch, A. Howie, R. B. Nicholson, D. W. Pashley and M. J. Whelan, Electron Microscopy of Thin Crystals, Butterworths, London, 1965.
12. G. Thomas, Transmission Electron Microscopy of Metals, John Willey and Sons, Inc., New York, 1962.
13. Mc.G. Tegart, The Electrolytic and chemical polishing of metals in Research and Industry, Pergamon Press, London, 1959.
14. G. L. Kehl, The principles of Metallographic Lab Practice, McGraw Hill Book Co, New York, 1949.
15. P. J. Goodhew, Specimen Preparation in Materials Science, North Holland Publishing Co.
16. D. H. Kay, Techniques for Electron Microscopy, Blackwell
17. V. Valdre, Ed. Electron Microscopy in Materials Science, Academic Press.