CHAPTER 6

METALLURGICAL EXAMINATION TECHNIQUES

The fact that metals are crystalline is explained in chapter 2. The type of lattice structure, the presence of impurity atoms and secondary phases, and the transformation of phases at different temperatures are factors that influence the properties of metals. Some of the common techniques for the examination of metals are discussed in this chapter.

6.1 Metallography

The examination of the metal surfaces with or without high magnification is called metallography. Two important subdivisions of this are the macrography and microscopy.

The structure and constituents of a metal or alloy can be revealed after suitable preparation. When the structure is revealed to the naked eye or at magnifications upto about 10 diameters, it is termed *macro-examination* or *macrography*. Sometimes the metals are subjected to special treatments to exaggerate flaws and structural irregularities. Metal features such as macro-segregation, inclusions and grain size can be studied and the information so obtained used to determine the mode of solidification and the previous processing history. Deep etching is frequently applied to show flow lines in forged and rolled sections. Such deeply etched surfaces can be used to produce prints by smearing them with printers ink and then pressing a white paper in contact with the surface. A hardness survey may be conducted on a macro-surface to get an idea of the uniformity of properties in different parts.

6.2 Sulphur Printing

A highly useful macro-examination consists in sulphur printing to reveal segregation of sulphur in steel. The record is obtained on bromide photographic paper by staining it with the evolved H_2S gas. Bromide paper is soaked in dilute H_2SO_4 (3%) for about 2 minutes and is inverted on the freshly prepared steel surface with its sensitive side down. The reaction between sulphides and acid results in the evolution of H_2S gas and this causes staining. After staining the paper is fixed in hypo. Brown coloration on the paper indicates local segregation of sulphides in steel.

Macro-examination methods offer advantages such as simplicity of operations and permit the examination of larger surfaces at any time. But the fact that parts being examined are to be sectioned imposes limitations to their application.

6.3 Microscopy

The most important area in metallography is the study of prepared

surfaces under the microscope employing magnifications beyond 10 diameters. The unaided human eye cannot distinguish details less than 0.1 mm apart and objects smaller than this have to be magnified at least to this dimension for recognition. Ordinary microscopes can yield a magnification of 50 to 2000. Microscopic examination can give precise information about the grain structure of metals, the nature of nonmetallic inclusions, segregated impurities and other secondary phases. It is used in the study of metal failures, routine process control and for the development of new alloys.

A variety of physical metallurgy problems have been solved by the microscopic examination technique and it is likely to continue playing an important role in this field. The efficiency of this method is being improved with the introduction of better objectives and eyepieces and the use of anti-reflection coatings. Reflecting type of microscopes permit examination of specimens from a longer distance and at a high temperature.

6.4 Metallographic Specimens

It has been recognized that successful micro-examination requires carefully prepared surfaces. The final polished surface must be free of distorted metal, contain all inclusions and micro-constituents and be free of marks and stains. The procedure to obtain a small specimen from bulk must ensure that it properly represents the whole mass. For a convenient polishing the usual specimen size is $\frac{1}{2}$ — 2 cm dia. Such specimens are obtained by cutting with a low speed saw or a cut-off abrasive wheel. During the cutting stage the specimen must not be overheated, or else the structure would change and misleading results obtained Flat and parallel surfaces are required so that a wider field of view remains in the depth of focus of the lens and refocussing is not necessary at different points.

Figure 6-1 illustrates a common type of cut-off machine. Many grades of abrasive wheels are available and in general hard wheels are used for soft materials. Submerged type of cut-off machines provided with a coolant circulation system are generally preferred.

6.5 Mounting

When the specimen is too small, fragile or of irregular shape to be conveniently handled, it is mounted or put in a clamp to facilitate polishing. Mounting materials that have been successfully used include sulphur, sealing wax, low melting point alloys (Wood's alloy M.P. 70°C) and thermosetting resins. Alloy mounting is not common due to the difficulty of satisfactory etching, since sacrificial corrosion of the mounting alloy may be ecountered.

Thermosetting resins such as phenol formaldehyde (bakelite) and

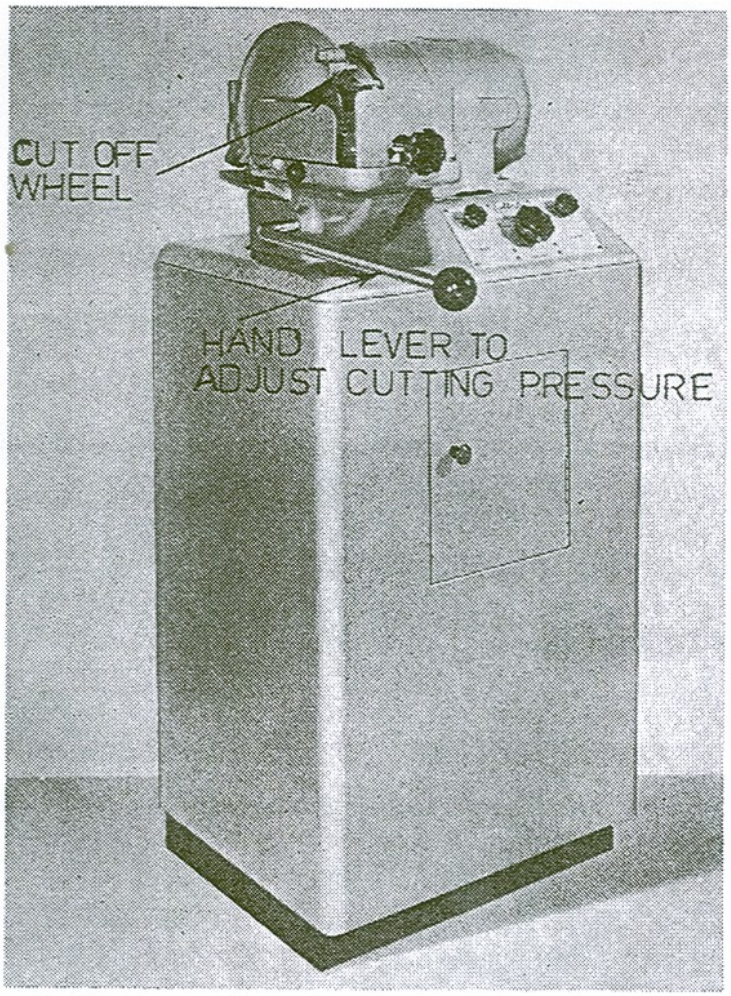

Fig. 6-1 Specimen cut-off machine. (Courtesy of Buehler Ltd. USA)

aniline formaldehyde are the most common mounting plastics. They possess a high resistance to attack by etching reagents. For most grades of bakelite powders a maximum temperature of 140°C is required with a corresponding pressure of 1.76×10^7 N/m² (1.14 Ton/in²). After setting the molding plastic is rapidly cooled by surrounding the die with chill blocks. The method of mounting consists in placing the specimen in the die with its face down, covering it with plastic powder and then applying the required heat and pressure. Figure 6-2 illustrates a hydraulic mounting press equipped with chill blocks.

Cold setting liquid resins are now available for metallographic mounting. The hardening temperature required for these resins is upto 50°C.

For specimens that are to be electrolytically polished and etched, conducting mounts are prepared. One method to prepare such mounts utilizes two mounting plastics, one of which is non-conducting while the other containing 20% of copper powder is conducting. The non-conducting powder is poured on the sides followed by the conducting powder and the specimen is mounted.

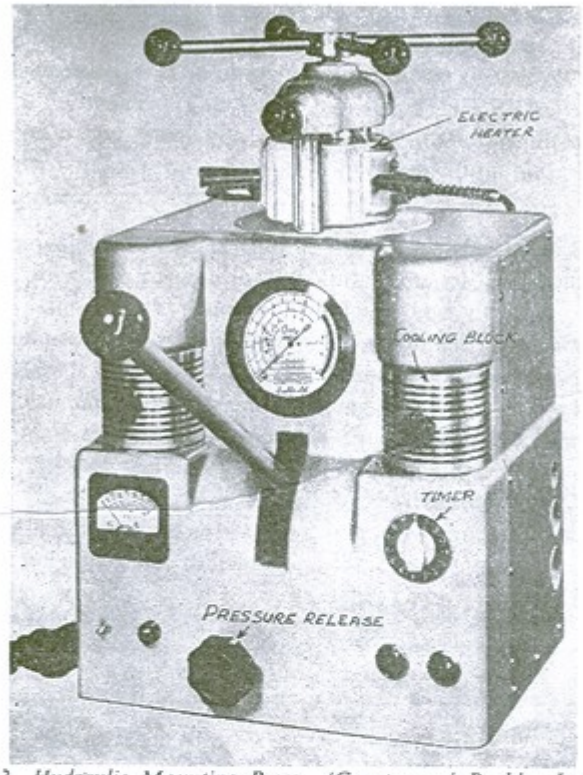

Fig. 6-2 Hydraulic Mounting Press. (Courtesy of Buehler Ltd. USA)

6.6 Grinding

After the specimen has been cut to shape and mounted it is subjected to smoothening operation. Silicon carbide abrasive paper or motor driven abrasive belts are recommended for this stage. The next step involves grinding on four grades of emery papers in the order of increasing fineness, starting with No. 1 and going in turn to 0, 00, and 000 papers. During grinding on each successive paper, the specimen is rotated to produce fresh scratches at 90° to those formed earlier. While polishing soft materials such as aluminium and lead, a little paraffin is applied to the paper to avoid the abrasive particles from getting embedded in their surfaces.

6.7 Final Polishing

Though the surface after initial polishing becomes bright, further treatment is necessary to obtain a scratch free surface for microscopic examination. This is achieved by polishing on a flat circular disc covered with a cloth that is impregnated with fine abrasive. The mechanical polishing discs or laps are covered with silk, canvas or selvyt cloth. The abrasives include fine alumina, chromic acid, magnesium oxide or iron oxide. For very hard materials such as sintered

carbides, final polishing is done with properly graded diamond dust of 0.5 micron size. In diamond polishing lubricants such as olive oil or mineral oil are used. The abrasives are poured on the revolving wheel as a slurry in water. The usual lap speed is 250-500 rpm. While polishing gray iron, steel and other ferrous materials firm pressure is used, but for soft materials such as copper and aluminium low pressure is to be applied.

The removal of scratches during final polishing takes 3-7 minutes. Excessive polishing results in the formation of additional disturbed metal, pits, and streaks due to dislodging and dragging of the non-metallic inclusions. After polishing the specimen is washed in running water, swabbed with wet cotton and finally rinsed in alcohol before drying. Automatic low speed polishing machines are now available to polish more than one sample at a time. Such machines have provision for holding the samples in special holders, which can revolve across the lap in a direction counter to that of the wheel. A loading device facilitates the adjustment of pressure on the sample. Figure 6-3 shows an automatic polishing machine.

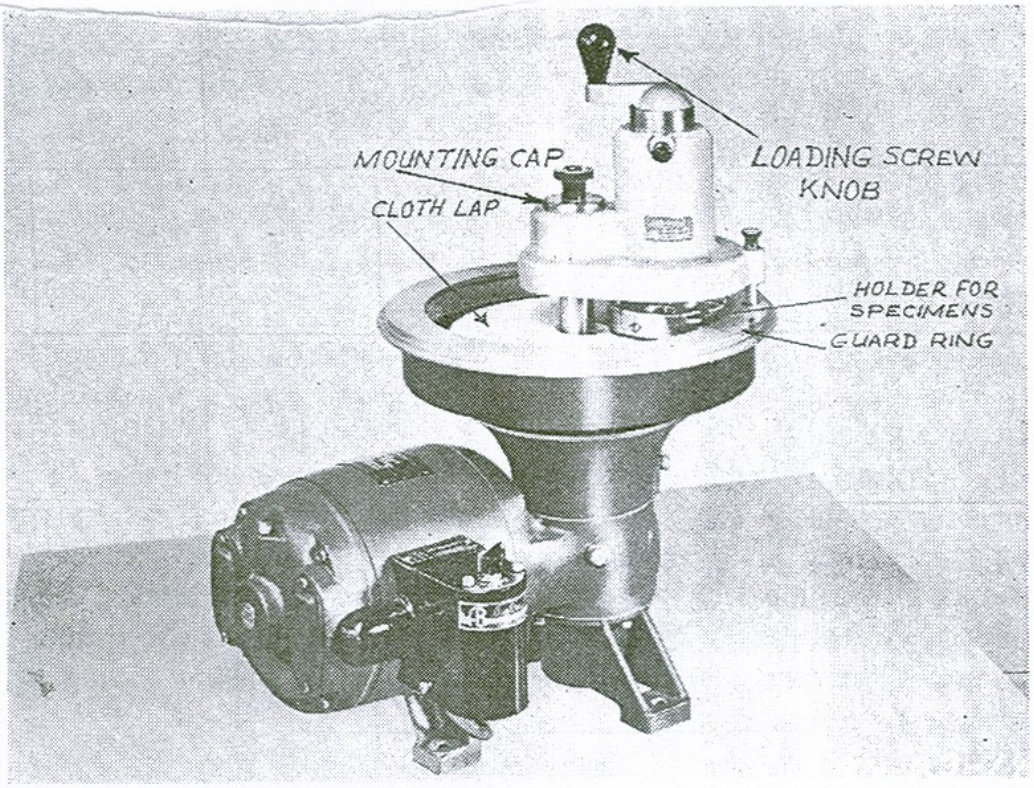

Fig. 6-3 Automatic Polishing Machine. (Courtesy of Buehler Ltd. USA)

6.8 Electrolytic Polishing

Hand polishing of metallographic samples is a tedious job and it is not always possible to produce ideal surfaces for examination at high magnification. Soft metals are difficult to polish due to the formation of disturbed metal. The repolishing and etching steps that are used in usual polishing of soft metals to get rid of the disturbed metal are not

required in electropolishing. Electropolishing is more economical and quick, but a serious disadvantage of the process is the complete or partial removal of nonmetallic inclusions by chemical reactions with the electrolyte.

Electrolytic polishing overcomes many other limitations of the mechanical polishing method. The specimen is made the anode in a solution that is specific for each type of metal. During electrolysis the surface layer is removed and this happens because the resistance between anode and cathode is lower at the peaks of rough regions than in the troughs. Thus at a certain voltage the current density at the peaks and ridges is greater than at the valleys and the metal dissolves faster at the peaks and the anode sample surface is polished. The optimum polishing conditions are revealed by the current density vs voltage curves. Curves similar to the one illustrated in figure 6-4 are obtained for many metal-electrolyte combinations, such as copper in phosphoric acid, zinc and tungsten in alkaline solutions, and magnesium in phosphoric acid-ethyl alcohol solution. Polishing occurs over a range of current densities. When resistance is plotted against voltage, a single maximum is obtained which corresponds to the best polishing condition.

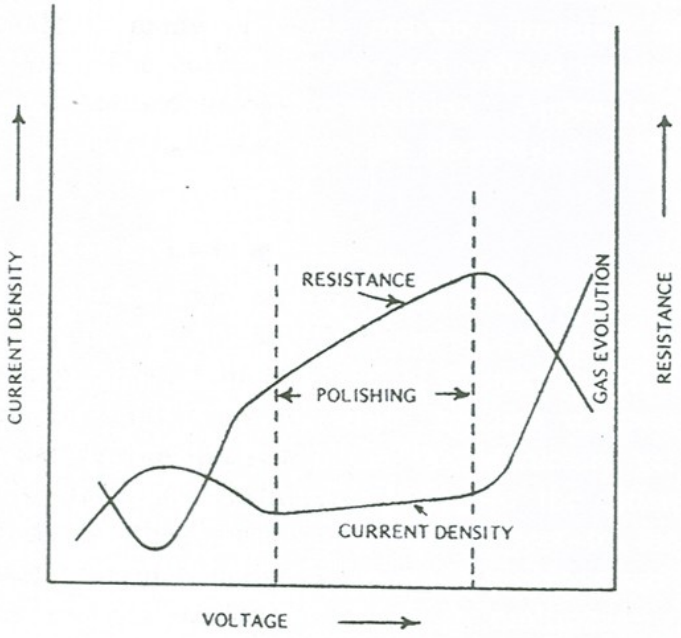

Fig. 6-4 Voltage vs Current Density and Voltage vs Resistance relationships for metal-electrolyte combinations.

Some experimental work is necessary to determine the optimum conditions for polishing a particular metal or alloy. With solutions of high resistance or of high viscosity, it is necessary to have mild agitation of the bath to avoid local overheating. Excessive agitation can destroy the anodic film and no polishing occurs.

The three main types of electropolishing solutions include:

(i) The phosphoric acid solutions suitable for copper alloys, cobalt, iron, zinc and magnesium.

(ii) The methyl alcohol-nitric acid solutions that are used for nickel alloys, aluminium, steels and copper alloys. These baths require cooling due to the higher current density used.

(iii) The perchloric acid-acetic anhydride type that are applicable to aluminium, tin, nickel, magnesium and stainless steel. Such baths must be used with extreme caution due to the danger of a violent explosion if adequate cooling is not provided or a spark appears. Mounted specimens in bakelite, plastic or bismuth are not used in this bath.

Appendix 2 gives the composition of electrolytes and cell data for electropolishing of some common metals.

6.9 Electrolytic Etching

Electrolytic etching is a very useful technique for developing the microstructure of some materials that are difficult to etch by chemical means due to the formation of a passive film. It consists in passing a direct current through an electrolyte with the sample acting as an anode. Cathode is made of some insoluble material, such as platinum or graphite. The current density and voltage required are quite different from those used in electro-polishing. Appendix 3 gives the composition and electrical data for the electrolytic etching of some common metals.

6.10 Chemical Etching

An unetched polished specimen when examined under the microscope does not show any structure except for some inclusions. During polishing a thin film of metal is formed by cold deformation and it covers the underlying structure. Etching removes this film of metal and the various phases in the microstructure get revealed. In pure metals grains are distinguished due to the differential attack, which depends on their orientation with respect to the plane of polished surface. Grain boundaries are made visible, since the rate of chemical attack in these regions is high. In multiphase alloys the attack on some phases is more than on others due to the difference in composition and orientation.

The etching reagents used for chemical etching contain organic and inorganic acids and other complex substances in appropriate solvents, such as water or alcohol. The type of the etching reagent used depends on the nature of the information required. Steel, for example, can be etched both with nital and picral. Nital etching can only reveal grain

boundaries in ferrite. If the object is to distinguish between the ferrite, cementite and pearlite phases in the structure, picral yields a much better result.

Appendix 4 gives the Composition of reagents and the etching conditions recommended for some common metals.

In addition to the electrolytic and chemical etching techniques certain other procedures have been used and these include magnetic etching and thermal tinting. In magnetic etching a fine iron powder is used for the identification of magnetic phases in the structure. Thermal tinting depends on the differential rate of oxidation of the phases in the structure on heating.

6.11 Metallurgical Microscope

With optical microscopes the limitation is that two points cannot be resolved and appear as a single blur, if the distance between them is less than $\frac{1}{2}$ the wave length of illumination. Thus the resolving power of an optical microscope should be a fraction of an angstrom unit. However, the currently available instruments can resolve only several angstroms.

The optical system of the microscope consists of two compound lenses. The one near the object is objective and the other kept 15 to 25cm away is called the eye piece. Metals being opaque are always examined by reflected light. Illumination of the specimen is carried out by the oblique or vertical methods and these are explained in figure 6-5.

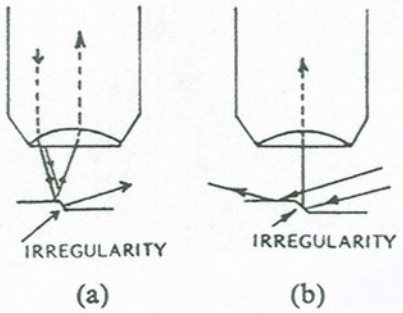

Fig. 6-5 Systems of Illumination. (a) Vertical illumination. (b) Oblique illumination.

In the case of oblique illumination light is reflected towards the objective only from the irregularities on the specimen surface and these appear as bright regions on a dark background. Most metallurgical microscopes use vertical illumination in which the light rays falling on a smooth surface vertically are reflected back, but the regions of irregularities cast these astray and thus smooth surfaces appear bright and others are dark. The stage of these microscopes is adjustable and can be moved to examine large areas.

The magnification depends on the focal length of the objective and the relative distances of the specimen, objective and the image. The eye piece has a low magnification and the image formed by the objective is examined through it. For large specimens and for photographic work, inverted binocular microscopes are preferred due to less eye strain. An inverted binocular microscope is illustrated in figure 6-6.

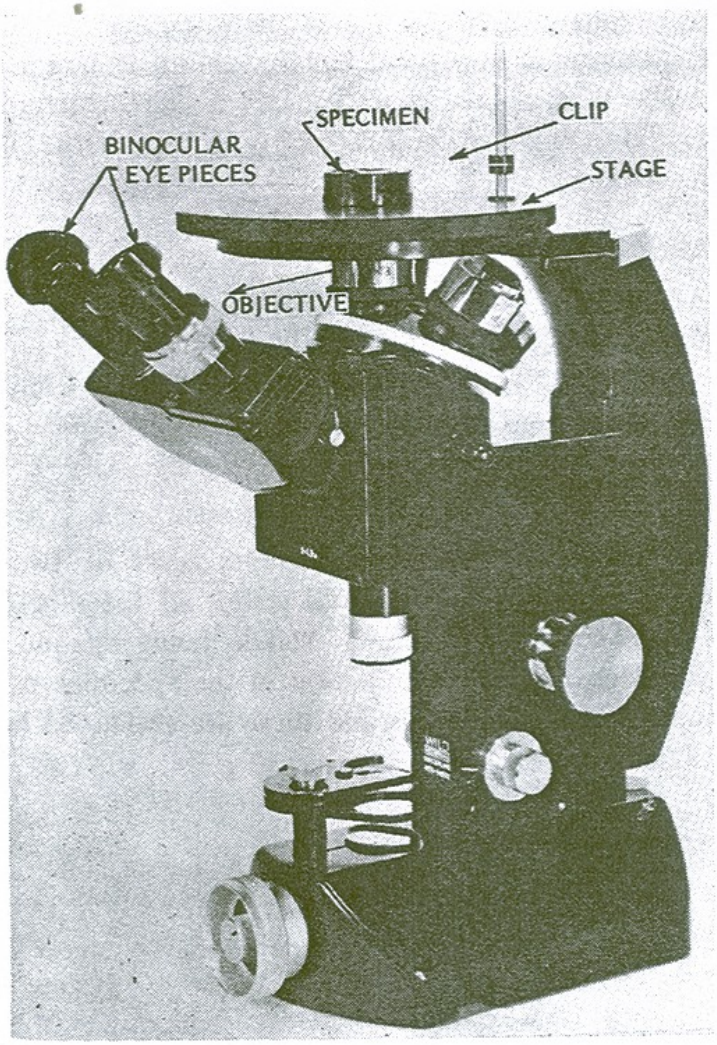

Fig. 6-6 Inverted binocular metallurgical microscope. (Courtesy of Wild Heerbrugg Ltd. Switzerland)

A *metallograph* is used to produce a photographic record of the micro-structure. It consists of an inverted microscope equipped with reflecting prisms or mirrors and a photographic camera mounted in the vertical, inclined or horizontal position. A typical binocular metallograph is illustrated in figure 6-7. The metallograph requires a very fine focussing facility and has a strong and stable construction. The procedure to photograph a projected image at any magnification in the camera is simillar to ordinary photography.

There have been many developments in the area of metallographic examination of metals. An interesting equipment introduced is the *flying spot microscope* in which the field of view is rapidly scanned by a spot of light. Nondestructive metallography is another innovation that facilitates the examination of inservice components. In this technique the surface under examination is polished and etched by the electrolytic methods and the examination is carried out not on the original surface, but on a replica obtained by using a special liquid varnish. A thin flexible and transparent varnish film is stripped from the surface by the aid of a wetting agent and is examined by the transmitted or reflected light. The quality of the image can be improved by aluminium or gold metallizing on the replica surfaces.

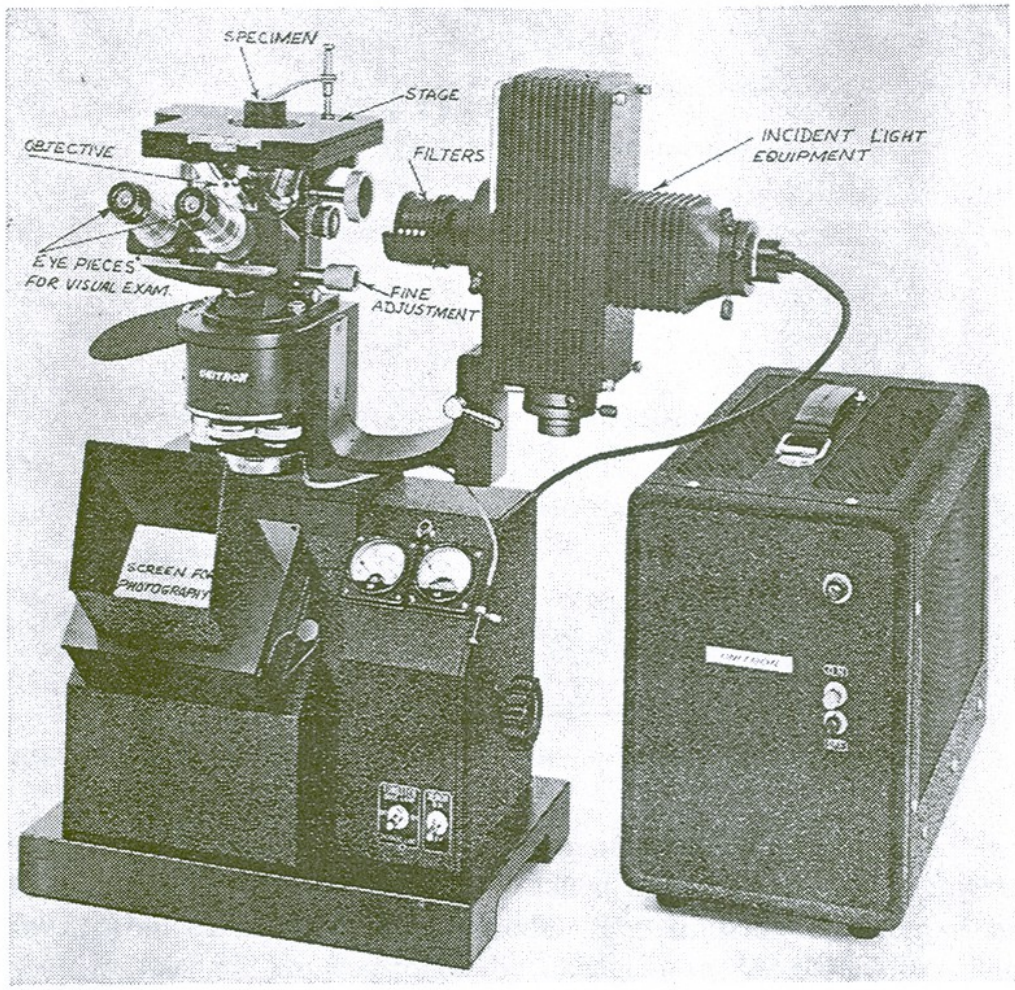

Fig. 6-7 A typical binocular metallograph. (Courtesy of Unitron Instruments Co., USA).

6.12 Electron Microscopy

It was said earlier that due to imperfections in the optical systems and the high wave length of illumination the resolving power of a light microscope is limited. Since electrons have wave lengths far below those of light, the resolving power is markedly improved when a

beam of electrons is used. The basic principle of magnification is similar to that of an optical system, except that instead of glass lenses magnetic and electrostatic fields are used to focus the electrons. Figure 6-8 shows the arrangement of the electron beam and the magnetic lenses in a transmission electron microscope.

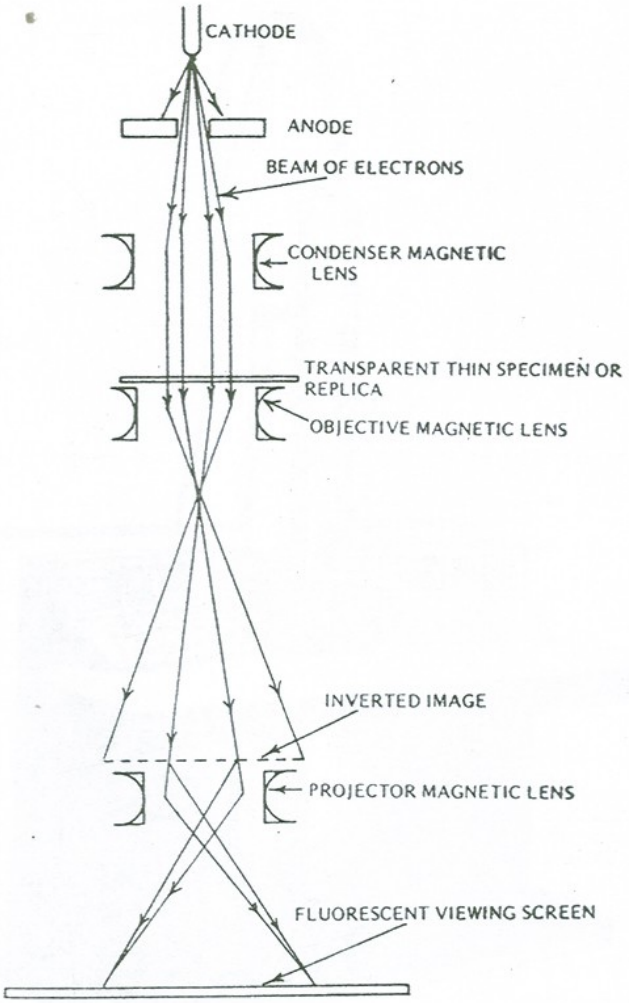

Fig. 6-8 Diagramatic arrangement of electron beam and magnetic lenses in a transmission electron microscope.

In the electron microscope a beam of electrons is generated in a heated tungsten filament, which is called electron gun and is directed towards an earthed cylindrical anode. The emission of electrons and their wavelength are controlled by changing the accelerating voltage. Magnifications upto 200,000 times can be obtained. The path of the electron beam is adjusted with the help of stabilizers and magnetic lenses. An interesting feature of the electron microscope is the greater depth of focus as compared to optical microscopy and this brings all parts of the specimen in focus at once. The greater depth of focus permits the use of stereography to obtain the three dimensional views of the specimen and the measurement of the height of various features relative to each other.

The electrons are invisible to the naked eye and it is necessary to use a fluorescent screen to observe the image as is done in a television screen. As the electrons cannot travel very far in air the electron source and the lens chambers have to be evacuated. A typical electron microscope is illustrated in figure 6-9.

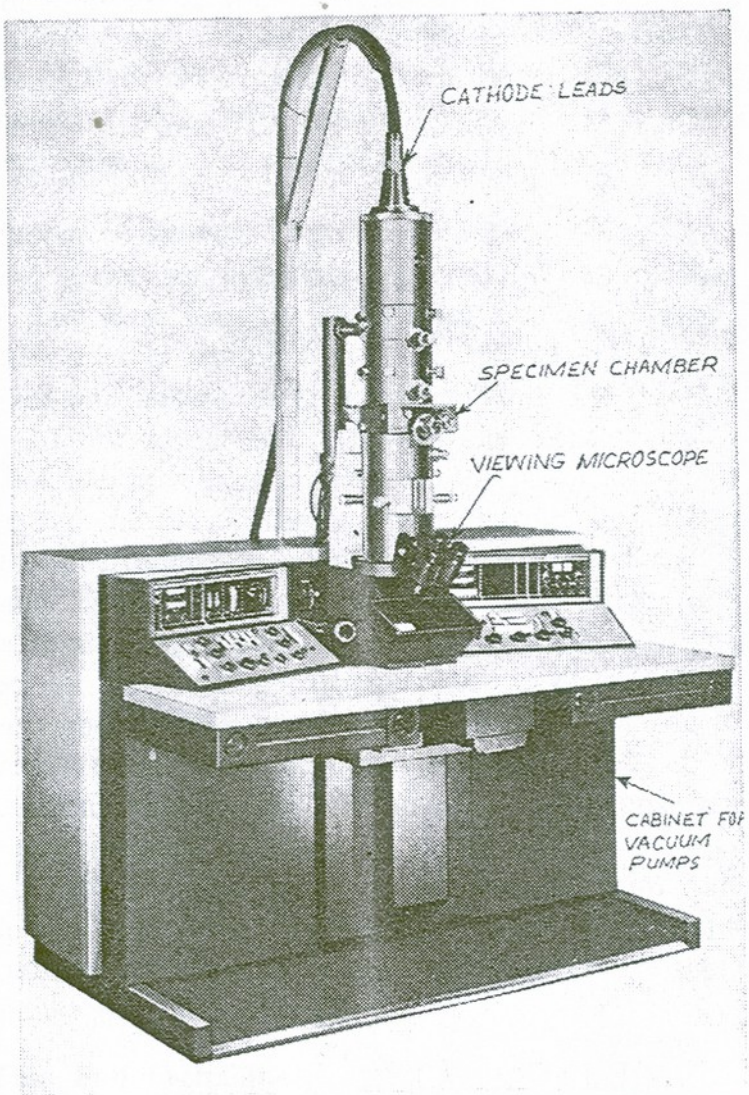

Fig. 6-9 A typical electron microscope. (Courtesy of Radio Corporation of America)

A serious limitation of the electron microscope is the difficulty of reflection of the electron beam. However, direct examination of the conventional microspecimens can be made with the scanning and emission electron microscopes. In the scanning instruments the metal surface is scanned by a focussed beam of electrons and the secondary emission is used to brighten the fluorescent screen and form an image. In the emission miscroscope the specimen is irradiated by electrons and the secondaries form an image by a lens system. The disadvantage of examination by the direct methods is the poor resolving power achieved compared with the transmission electron microscope.

Transmission electron microscopes are more often used for metallurgical examinations. All metals are opaque to electrons and bulk metal specimens cannot be used under transmission conditions. Specimens thinner than 1000Å, which are transparent to the electron beam are used. The preparation of thin specimens is extremely difficult and it is usual to employ methods that copy the surface contour of the metal specimen and to examine the copy after stripping it as a thin film. Two important procedures used for producing the contour copy are; (a) the replica technique, and (b) the thin film technique.

6.13 Replica Technique

A large variety of materials are used for the replication of metal surfaces, but what may be suitable for one metal may not give best results with another. Aluminium alloy replica can be made by anodizing the surface and subsequently removing the oxide film. Plastic replica method is more widely used. In this method a suitable plastic mixed with a volatile solvent is applied on the polished and etched surface. After drying, the plastic film is stripped from the surface and studied under the microscope. Collodin plastic dissolved in amyl acetate is often used for making a plastic replica. For high resolution carbon or silica replica are found to be more effective. A carbon replica is produced by evaporating carbon on to a plastic cast taken earlier from the etched surface and then dissolving the plastic to leave a thin structureless carbon film that has a positive contour of the original surface. In another method carbon is directly deposited on the etched surface and stripped by chemical or electrolytic means. The plastic replica can be handled more safely than the thin, inflexible carbon or oxide replicas. The single stage methods that produce a negative replica are now superseded by the two stage positive replica techniques that produce the surface features in their true perspective.

6.14 Thin Foil Technique

The thin foil electron microscopy affords a method for the study of imperfections and dispersed minor phases in the crystal structure of solids, which influence their strength, plasticity and other properties. It is possible to produce foils suitable for examination by transmission through electropolishing techniques following the sectioning of the specimen by chemical thinning, microtoming and some other methods. The thin foil method has provided experimental confirmation of the fundamental concept of the dislocation theory and is suited to the study of phase transformations, precipitation phenomenon and other metallurgical features such as ordering and vacancies.

Due to the great depth of focus an electron microscope can be used in the study of rough surfaces. Study of fractured surfaces by

this instrument can yield information relating to the structure of the material, the type of failure and intergranular features. It facilitates the determination of relation of microstructure vs bulk properties and of the surface conditions vs resistance to mechanical abrasion and chemical attack.

6.15 Field Ion Microscopy

The electron microscope with its 10 Å resolution has been a powerful addition to the metallographer's equipment, but there are still many features of the structure beyond its grasp. The next step would be that shows atoms as individuals occupying certain positions in the structure. This has already become possible with the introduction of field ion microscope that has a resolving power of 2-3 Å.

For this microscope the specimen consists of a fine wire, electropolished at one end and forming a sharp hemispherical tip. It is welded on the back to a tungsten electrode which is cooled with solid nitrogen. The assembly is evacuated to below 10^{-6} mm of mercury and a trace helium gas is allowed to leak inside. Facing the tip is a fluorescent screen that glows when bombarded by ionized atoms. The specimen is electrically charged relative to the screen to a potential of 5000-15,000 volts. When the specimen tip is negatively charged electrons are pulled out of it, but when the polarity is reversed free electron gas is slightly pulled into the metal, thus partly exposing the positively charged metal ions on the surface. The image of these ions is carried by the helium atoms to the screen after being ionized by giving up electrons to the metal atoms and then travelling straight to the screen along electrostatic lines of force. The magnification thus produced is equivalent to the ratio of the screen distance to tip radius; i.e. about one million. This microscope can be used to study grain boundaries, dislocations and lattice defects, such as vacancies.

6.16 X-Ray Metallography

X-ray analysis has been a very versatile method for the physical examination of metals. Originally the method was designed to study the atomic arrangement of metals, but its scope has been steadily expanding. Having a resolution far better than the finest microscope the X-ray diffraction method is used to study the atomic structure of metals and the identification of phases. Other studies by the diffraction technique include allotropy and thermal expansion, strain in cold worked metals, age hardening, grain size, and the grain orientation of the metals. Special cameras are available and records can be obtained on photographic films or by the use of a goniometer and gieger counter arrangements.

X-rays are generated when rapidly moving electrons strike atoms of matter. A simple form of X-ray tube consists of an evacuated chamber with two electrodes between which a high potential is applied. The cathode, usually a tungsten filament, acts as a source of electrons. The electrons are directed towards the anode (called target) and X-rays are emitted. As only about 1% of the energy of bombarding electrons reappears as X-rays, the target must be cooled by water or oil to avoid melting.

The nature of the X-rays is influenced by the applied potential and the target material. At voltages below a critical value the X-rays emitted consist of a continuous spectrum, known as the white radiations. On raising the voltage beyond a critical value the anode emits characteristic radiations and the particular wave length at which peaks are observed depends upon the target material. This happens when the electrons strike the target with enough speed to eject photo-electrons from the inner most electron shells. It is usually the characteristic radiations that are used for X-ray diffraction work after absorbing the white and other unwanted radiations. Filters consisting of thin foils of the suitable material are used for the purpose. The emission of white and characteristic radiations by a copper target and absorption of white radiations by a nickel filter are illustrated in figure 6-10.

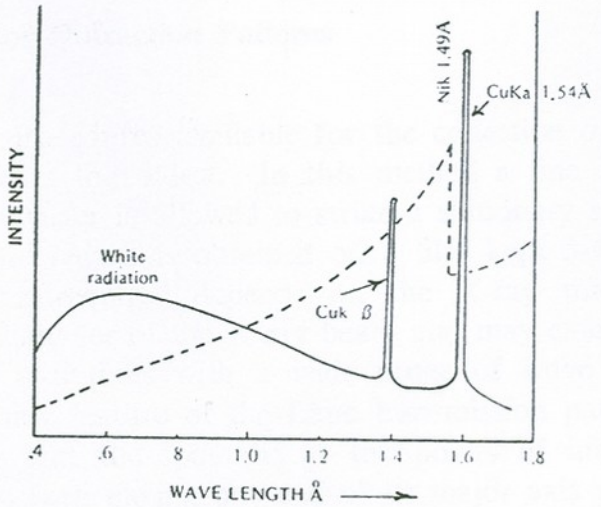

Fig. 6-10 The absorption of X-rays produced by copper target with a nickel filter. The broken lines show the mass absorption coefficient. The result is an increased intensity of Ka radiations relative to those of other wave length.

6.17 X-ray Diffraction

When the X-rays strike the plane of a crystal, the atoms act as point sources of radiations and the reflected rays have the same wave lengths as those of the incident radiations. The path difference between the reflected or diffracted beams is given by the Bragg's law. Figure 6-11 shows the path of the incident and diffracted beams on the

planes A and B at a glancing angle Q. The path difference between the diffracted beams derived from the same incident beam and diffracted from planes A and B is clearly equal to MO + NO or equal to 2d.SinQ, where d is the distance between planes A and B. For reflection to occur this path must be a whole number of the wave length and this is numerically stated by the Bragg's law as;

$2d.\sin Q = n\lambda$, where n is an integer and λ is the wave length of the beam.

In actual diffraction studies numerous parallel planes play their part in producing a diffracted pattern and when there is a slight disagreement in the phases of the diffracted beams from the successive planes, the regularity of these and their large number cause destructive interference.

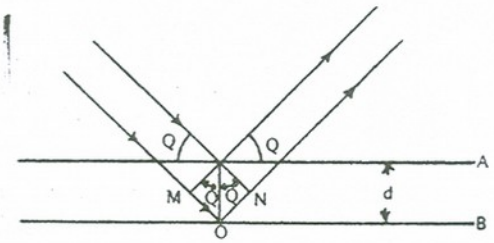

Fig. 6-11 Derivation of the Bragg's Law.

6.18 Production of Diffraction Patterns

Laue Method

Of the various procedures available for the collection of diffraction data Laue method is the oldest. In this method a fine X-ray beam about $\frac{1}{2}$ mm in diameter is allowed to strike a stationary single crystal and a photographic record is obtained on a film kept 3-4 cms away. The exposure time required depends on the X-ray tube, the film distance and the diameter of the X-ray beam and may extend to about an hour. White radiations with a wide range of wave lengths are used. An interesting feature of the Laue transmission pattern, shown in figure 6-12, is that the spots lie at the points of intersection of a series of ellipses each having one end of its major axis at the centre spot. The Laue technique is commonly used for the study of grain orientation, lattice distortion, and precipitation phenomenon. The analysis of these patterns is very cumbersome.

Rotating Crystal Method

Rotating crystal method is highly useful for crystal analysis. In this procedure a monochromatic X-ray beam falls at a single crystal rotating at a fixed axis, so that various sets of lattice planes come successively into reflecting positions. The pattern is considerably simplified by rotating the crystal along its different axes in turn. The photograph

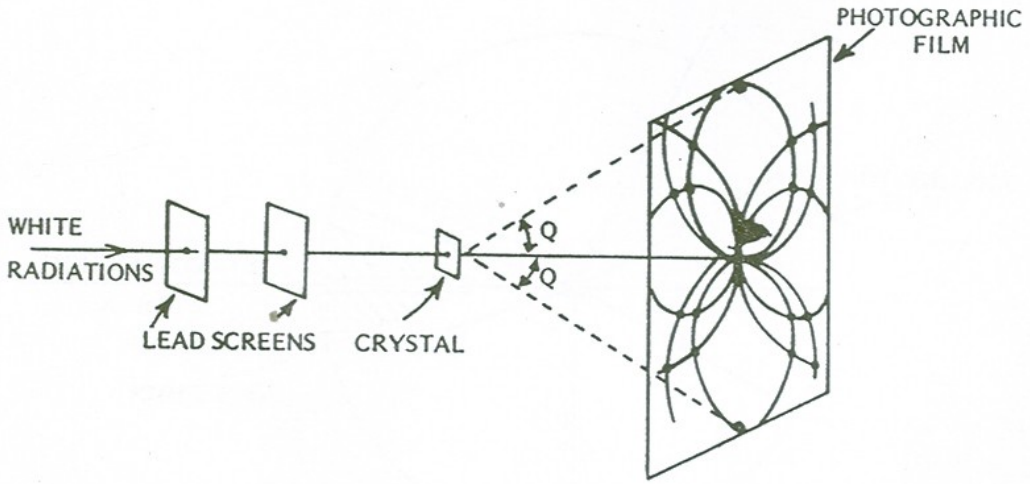

Fig. 6-12 Production of Laue patterns.

is recorded on a flat film kept a few centimeters away from the crystal or on a cylindrical film. The procedure permits the determination of lattice parameters and the orientation of grains in the crystal structure of metals.

Powder Method

The methods discussed so far require single crystals for diffraction studies. In the powder method the specimen consists of a fine powder in the bonded or compacted form or loose powder adhering to a fine hair. A polycrystalline material of fine grain size in wire or sheet form can also be used. It is necessary that a large number of small crystals are in every possible position with respect to the incident beam. The photographic film is either placed normal to the main beam or is bent in the form of a cylinder about an axis normal to the main X-ray beam. The plan of a typical powder camera along with a diffraction pattern obtained on an unrolled film is given in figure 6-13. When the flat film is kept normal to the incident beam, a series of concentric circles are obtained and from their radii the values of the corresponding Bragg's angles can be calculated. With a curved film the diffracted rays form arcs about the central spot on the film. The curvature of these arcs becomes less until they become straight lines and then it changes in the opposite direction. If the radius of the powder camera is known, it becomes simple to measure the Bragg's angles from the positions of diffractions on the film.

Since it is comparatively rare for two metal powders to give identical photographs, the patterns obtained can be used for identification. With impure specimens additional patterns get superimposed on the parent pattern. ASTM has produced charts giving data about the additional patterns and this facilitates the identification of the unknown materials.

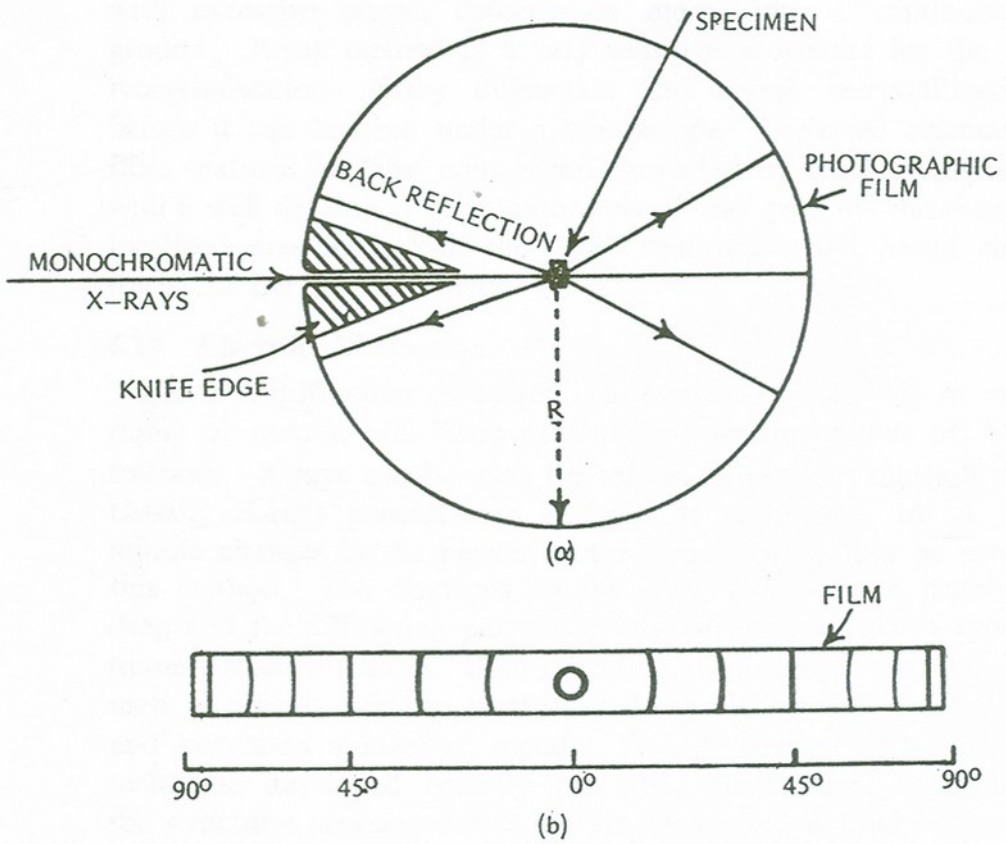

Fig. 6-13 (a) Typical powder camera arrangement for cylindrical film. (b) Curvature of lines on the unrolled film.

Back Reflection Method

This method is designed for the investigation of massive polycrystalline materials. The X-ray beam is allowed to pass through a hole in a flat photographic film to strike a specimen placed at a distance of about 7 cms. The film is suitably shielded with lead screens from the incident beam and the diffraction pattern is produced by back reflection only. If the type of the crystal is known, the lattice constants can be determined with a fair degree of accuracy. The grain size and preferred orientation can also be studied by this technique.

X-ray diffraction techniques can be used for the study of various effects on metals including elastic strain, plastic deformation, recrystallization and grain growth. Elastic strain may be produced by mechanical stresses and thermal shock. The Laue diffraction rings produced in a stress free polycrystalline material are very sharp, but these get diffused depending on the degree of displacement of atoms. The degree of lattice distortion can be best computed from the back reflection photographs. When the grains of a metal are fragmented, as during deformation and are preferentially oriented in certain directions, the spots on the photograph are drawn out into long narrow streaks, which

with excessive plastic deformation merge into a continuous background. X-ray method is a very sensitive procedure for the study of recrystallization. X-ray diffraction can reveal recrystallization long before it can be seen under a microscope. Preferred orientation and fibre textures are also conveniently revealed by these methods. Metals with a well developed fibre texture give X-ray patterns showing strongly localized arcs instead of the usual continuous and broad rings produced in the strained metal.

6.19 Electron Diffraction

Electron diffraction technique is also used for the study of crystallography of metals. Its basic principle is similar to that of X-ray diffraction. X-rays can be used for information about the bulk material. Usually X-rays penetrate to a depth of more than 10^5 Å and any minute changes in the phases or the structure may not be revealed by this method. The electrons on the other hand cannot penetrate very deep and the diffraction patterns produced are confined to those of extremely shallow regions. Both reflection and transmission techniques are used in practice and the method finds special applications in oxidation and corrosion studies of metals. A low energy electron diffraction technique developed recently can yield fundamental information on the structural arrangement of surface atoms and is used in the study of chemical reactions at the interface, nucleation, thin film growth and surface diffusion.

The electron microscopes are usually equipped with facilities for electron diffraction and this is achieved with little additional cost.

6.20 Fluorescent X-ray Spectrographic Analysis

Accurate quantitative analysis by the use of X-rays is possible for a wide range of alloys. It consists of placing the specimen against a strong X-ray beam to excite the elements in the specimen and emit characteristic radiations. The resultant radiations are analysed in a counter spectrometer by reflecting these from the surface of the crystal with known lattice spacings and recording the intensity of the reflected beam on a moving chart. The wave lengths present in the fluorescent radiations are calculated using the Bragg's law and the elements identified. It is necessary to have standards with known analysis so that the analysis of the sample can be determined by comparison of the radiation intensities. The chief advantage of the procedure is that a large area of the specimen is analysed and the inhomogeneity of composition within this region does not affect the result. Little specimen preparation is needed and the method is not destructive.

The technique is limited at present to the analysis of elements of higher atomic number than sodium. The method yields fast results and a few hundred samples can be analysed in a day.

6.21 Electron Probe Micro-analyser

Metallurgists can observe the micro-regions and minute phases in specimens, but their identification was always a matter of guess. The electron probe micro-analyser makes it possible to accurately determine the point to point chemical composition by means of X-ray emission spectroscopy. The method can provide useful data with respect to inclusion analysis, phase identification, diffusion in metals, depletion or enrichment of grain boundary regions and micro-segregation during solidification. Recent modifications in the instrument have increased its scope of application to rough surfaces and increased the speed of analysis.

The method involves irradiation of the sample with a focussed beam of high energy electrons. The X-rays emitted are directed to a crystal spectrometer and the wave lengths are sorted out. X-rays are measured from the characteristic peaks by rotating the spectrometer crystal and focussing the reflected beam on a proportional or gieger counter. Quantitative analysis is achieved by comparison with the standards, which consist of thin sheets of pure elements. The electron beam is focussed into a probe of 1 micron diameter and may be shifted to any position for analysis. Usually an optical microscope viewing arrangement is provided to correctly position the specimen into the path of the beam.

The scanning instrument produces scaler readings, X-ray spectra plots and images of the specimen in terms of X-ray emission or back scattered electrons. A simple arrangement of the various parts of a scanning micro-analyser is illustrated in figure 6-14. The elements that can be analysed by this instrument in air include elements from magnesium to uranium. This can be further extended by housing the spectrometer in vacuum.

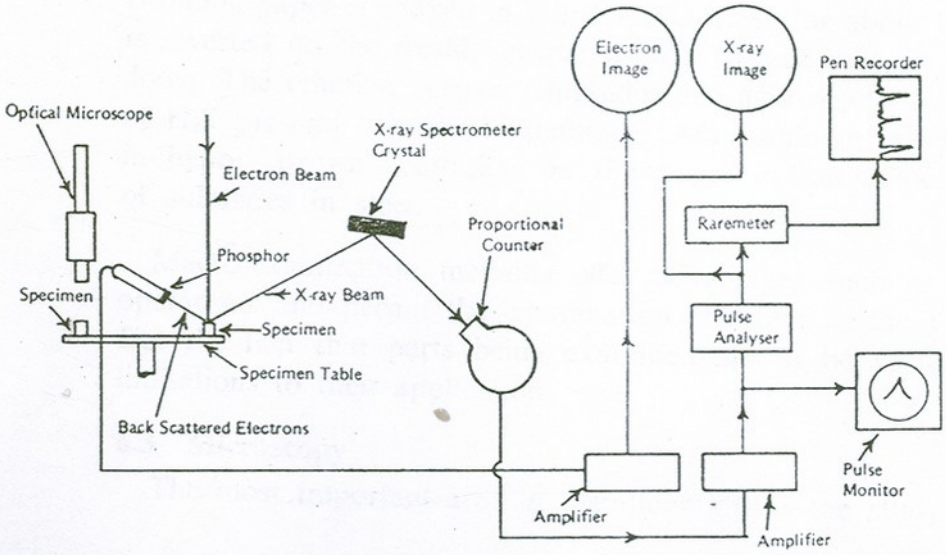

Fig. 6-14 Simplified diagram of X-ray scanning micro-analyser.