Modern Metallography

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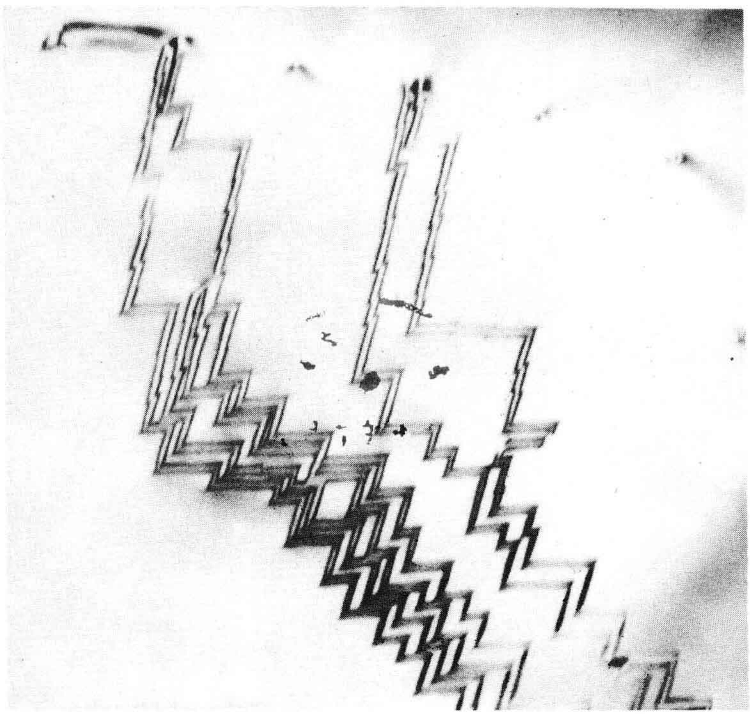

Transmission electron micrograph of antiphase boundaries in rutile.

PREFACE

IT IS now well established that the properties of metals are determined by the presence and behaviour of the defects they contain. These defects include precipitates, cracks, grain boundaries, dislocations, stacking faults and impurity atoms, the theoretical aspects of which have been treated in several books in recent years. There is however, a gap in metallurgical literature for a book suitable for students which deals with the practical techniques of studying defects in metals. It is hoped that the present book will at least partly fill this gap.

The book is primarily written for students of Metallurgy and Materials Science at Universities and Colleges of Advanced Technology, although it is hoped that practising metallurgists will find it useful. Since a comprehensive treatise on metallography would require the writing of several books, we decided to plan the present volume on three main principles: (1) to describe metallographic methods rather than observations; (2) to devote more space to recent methods than to well established ones. It is for this reason that our treatment of, for example, conventional X-ray techniques is by no means detailed; and (3) to consider in more detail the theories which lead to the experimental determination of quantities hitherto unknown, such as stacking-fault energy, Burgers vector, etc.

Finally, we would like to thank many colleagues who have helped during the preparation of this book, particularly those who have supplied original prints of micrographs. Due acknowledgement is given at the appropriate places in the text.

"If they wos a pair o' patent double million magnifyin' gas microscopes of hextra power, p'raps I might be able to see through a flight o' stairs and a deal door; but bein' only eyes, you see, my wision's limited"

(SAM WELLER–*Pickwick Papers*)

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CHAPTER I

REFLECTED LIGHT MICROSCOPY

INTRODUCTION

Metallographic examination is usually carried out with the aid of reflected light in order to investigate the nature and distribution of phases in metallurgical specimens, as well as to examine such lattice features as grain boundaries, slip bands, twins and cracks.

SPECIMEN PREPARATION

The specimen for microscopical examination is cut from the bulk metal sample and mounted either in cold-setting plastic or in thermo-setting bakelite using a metallographer's conventional mounting press. The surface to be examined is then ground by hand on a metal file until a suitable flat is obtained, and then on emery papers of decreasing coarseness from 320 grade down to 640 grade. To prevent over-heating, this operation is usually carried out under water or paraffin with the specimen held in such a way that each successive grinding operation is made at right angles to the one before it, and the specimen is not transferred to a new emery paper until the scratches produced by the previous paper have been removed by those of the one in use. A further necessary refinement of the grinding procedure is to wash the specimen carefully with water between successive papers to prevent coarse grit from being carried on to less coarse papers. After grinding on the finest grade emery paper, the specimen should be washed in water and then in alcohol, dried with a hair dryer, and finally polished on a selvyt cloth impregnated with an abrasive (alumina or diamond powder) held in a liquid suspension (water or paraffin). Polishing abrasives of various particle sizes from 10 microns down to $\frac{1}{4}$ micron are commercially available, and the best results are obtained using a series of cloths each impregnated with abrasive of successively finer grade. In almost all cases, a circular motion of the specimen on the selvyt cloth is recommended and is usually accomplished by carrying the selvyt cloth on a mechanical polishing wheel similar to that used by jewellers for lapidary work.

The mechanical polishing treatment involves the removal of surface projections of decreasing coarseness as the sequence file $\longrightarrow$ emery papers $\longrightarrow$ polishing wheel proceeds, which takes place by means of friction between the projections and the grinding or abrading medium. By its very nature, this mechanism produces both heat and plastic deformation in the surface layers of the specimen. In some cases this heat and/or deformation are undesirable, since it alters the thermal and mechanical history of the specimen under examination, but for the majority of specimens the affected surface layers are removed by etching, and hence the surface finally examined is representative of the bulk material.

Chemical and electrolytic polishing methods have been developed for certain metals and, provided a satisfactory surface can be produced, are usually to be preferred to mechanical polishing since the problem of surface damage is avoided. These methods of polishing are of special importance in electron microscopy and will be dealt with later.

THE REFLECTED LIGHT MICROSCOPE

Figure 1 is a ray diagram showing the formation of an image in the compound microscope. The basic components are two convex lenses, called the objective and eyepiece respectively. The object under examination O, is placed in front of the objective at a distance greater than its focal length f_o, but less than $2f_o$. A real inverted and enlarged image I_1, is formed at a distance greater than $2f_o$ from the objective on the side remote from the object. The distance between the objective and the eyepiece is made such that image I_1 is at a distance of less than f_e from the eyepiece, where f_e is the focal length of the eyepiece.

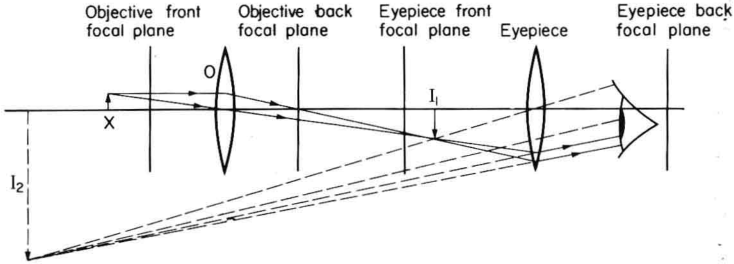

FIG. 1. Ray diagram showing how the image is formed in a compound microscope.

The eyepiece forms a virtual, upright and enlarged image I_2 of I_1, and it is this final image I_2 which is seen by the eye.

With low-power objectives the working distance (i.e. the space between the front surface of the objective and the specimen when the latter is in focus) is large enough to allow light, from a lamp held nearby, to illuminate the specimen. This is known as oblique illumination and is useful in the examination of surface projections (see Chapter III). The working distance is small for high-power objectives and it becomes necessary to illuminate the specimen with light passing vertically down through the objective. The principle of vertical illumination is illustrated in Fig. 2 where light from a source is reflected down through the objective by a half-silvered plane mirror held at an angle of 45° to the axis of the microscope. In some microscopes the half-silvered mirror is replaced by a right-angled glass prism which is offset from the axis of the microscope. Light reflected by the specimen and gathered by the objective passes through the half-silvered plane mirror to form the image which is viewed with the eyepiece.

A microscope cannot function properly unless the specimen is correctly illuminated; referring to Fig. 2, so-called "critical illumination" is achieved in the following way.

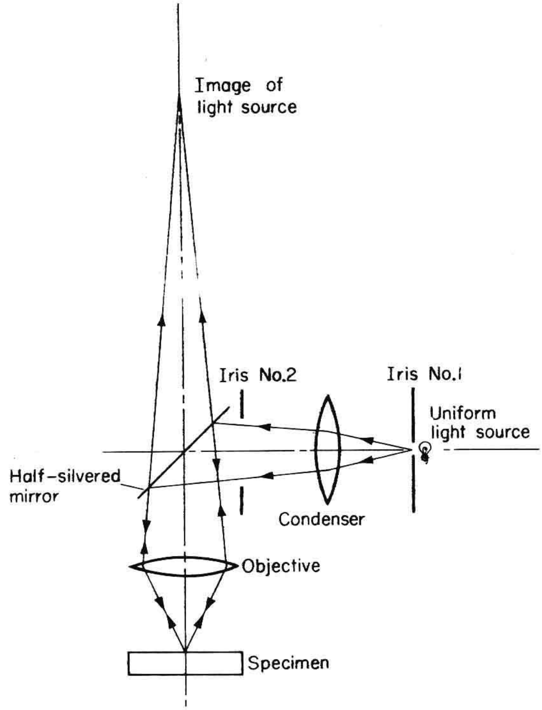

FIG. 2. Vertical illumination of a metallographic specimen.

1. The image of a uniform light source is formed at the specimen surface. The student should check that this is so by removing the microscope eyepiece and adjusting the position of the condenser lens until a sharp image of the lamp filament can be seen.
2. The aperture of No. 1 iris diaphragm is adjusted until it is just too big for the image of its outline to be seen when looking down the microscope. Under these conditions, the illuminated area on the specimen is controlled so that areas of no interest, e.g., the mounting medium, are not

illuminated and cannot scatter light into the objective, thereby "glaring" the image.

3. The aperture of No. 2 iris diaphragm is now decreased until only the area of interest can be seen. This effects an aperture control on the objective and ensures that the least possible amount of the less accurate part of the lens is used.*

For photographic work a more intense light source is

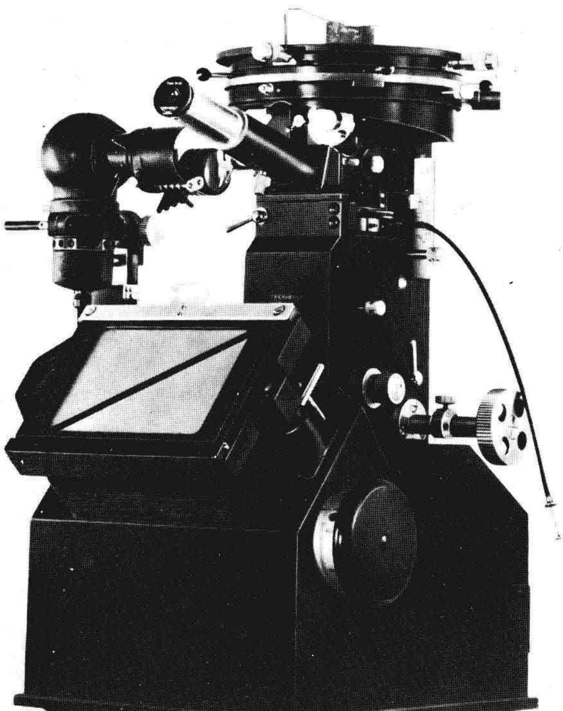

FIG. 3(a). Metallographic photo-microscope. (*Courtesy of Reichert.*)

*N.B. The quality of a lens is inferior at its outer edge than near its centre.

required. In Fig. 3(a) is shown a commercially available photomicroscope, and in Fig. 3(b) a ray diagram, to illustrate how the specimen under examination is illuminated and how the rays reflected by the specimen are received on the photographic plate.

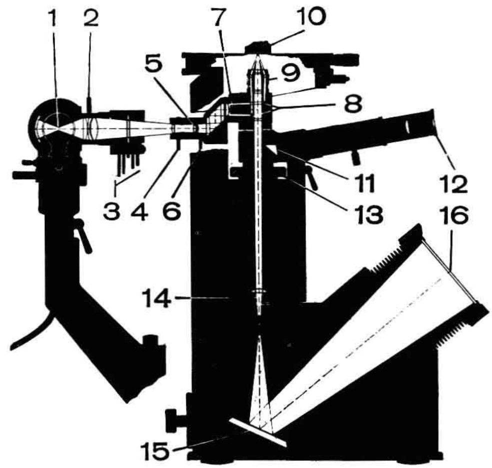

FIG. 3(b). Light path through the microscope shown in (a). 1 lamp filament. 2 condenser. 3 filter. 4 aperture-iris diaphragm. 5 deviating system. 6 field-iris diaphragm. 7 central diaphragm. 8 diverting mirror of opaque illuminator. 9 objective. 10 specimen. 11 diverting prism (swung out). 12 visual eyepiece tube (not used). 13 camera shutter. 14 photographic eyepiece. 15 diverting mirror of camera. 16 ground glass screen or photographic material (plate or film).

The most important component of the microscope is the objective, the chief function of which is to collect the maximum quantity of light coming from any part of the object. The numerical aperture (denoted by N.A.) of the objective is a measure of the light gathering power and is defined as

$$\text{N.A.} = \mu \sin \theta,$$