Some advantages of the optical and radiation pyrometers are:

1 Measurement of high temperature.
2 Measurement of inaccessible bodies.
3 Measurement of moving or small bodies.
4 No part of the instrument is exposed to the destructive effects of heating.

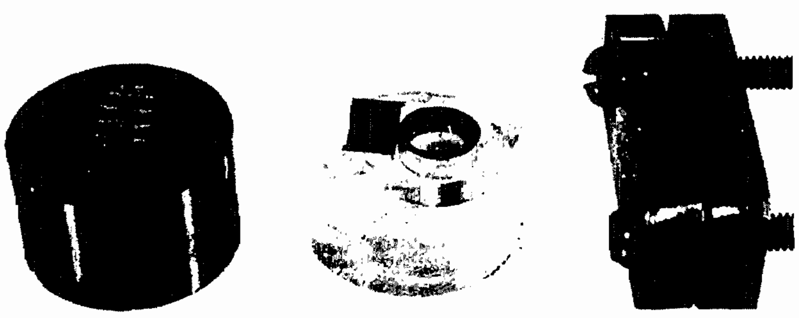
The principal disadvantages are:

1 Errors introduced because the photometric match is a matter of individual judgment.
2 Errors introduced by smoke or gases between the observer and the source.
3 Uncertainty as to the amount of departure from blackbody conditions.

METALLOGRAPHY

1·13 Introduction Metallography or microscopy consists of the microscopic study of the structural characteristics of a metal or an alloy. The microscope is by far the most important tool of the metallurgist from both the scientific and technical standpoints. It is possible to determine grain size and the size, shape, and distribution of various phases and inclusions which have a great effect on the mechanical properties of the metal. The microstructure will reveal the mechanical and thermal treatment of the metal, and it may be possible to predict its expected behavior under a given set of conditions.

Experience has indicated that success in microscopic study depends largely upon the care taken in the preparation of the specimen. The most expensive microscope will not reveal the structure of a specimen that has been poorly prepared. The procedure to be followed in the preparation of



(*a*) (*b*) (*c*)

Fig. 1·9 (*a*) Specimen mounted in Bakelite, enlarged 2X. (*b*) Specimen mounted in Lucite, enlarged 2X. (*c*) Specimen held in metal clamp, enlarged 2X.

a specimen is comparatively simple and involves a technique which is developed only after constant practice. The ultimate objective is to produce a flat, scratch-free, mirrorlike surface. The steps required to prepare a metallographic specimen properly are covered in Secs. 1·14 to 1·19.

1·14 Sampling The choice of a sample for microscopic study may be very important. If a failure is to be investigated, the sample should be chosen as close as possible to the area of failure and should be compared with one taken from the normal section.

If the material is soft, such as nonferrous metals or alloys and non-heat-treated steels, the section may be obtained by manual hacksawing. If the material is hard, the section may be obtained by use of an abrasive cutoff wheel. This wheel is a thin disk of suitable cutting abrasive, rotating at high speed. The specimen should be kept cool during the cutting operation.

1·15 Rough Grinding Whenever possible, the specimen should be of a size that is convenient to handle. A soft sample may be made flat by slowly moving it up and back across the surface of a flat smooth file. The soft or hard specimen may be rough-ground on a belt sander, with the specimen kept cool by frequent dropping in water during the grinding operation. In all grinding and polishing operations the specimen should be moved perpendicular to the existing scratches. This will facilitate recognition of the stage when the deeper scratches have been replaced by shallower ones characteristic of the finer abrasive. The rough grinding is continued until the surface is flat and free of nicks, burrs, etc., and all scratches due to the hacksaw or cutoff wheel are no longer visible. (The surface after rough grinding is shown in Fig. 1·10*a*.)

1·16 Mounting Specimens that are small or awkwardly shaped should be mounted to facilitate intermediate and final polishing. Wires, small rods, sheet metal specimens, thin sections, etc., must be appropriately mounted in a suitable material or rigidly clamped in a mechanical mount.

Synthetic plastic materials applied in a special mounting press will yield mounts of a uniform convenient size (usually 1 in., 1.25 in., or 1.5 in. in diameter) for handling in subsequent polishing operations. These mounts, when properly made, are very resistant to attack by the etching reagents ordinarily used. The most common thermosetting resin for mounting is Bakelite, Fig. 1·9*a*. Bakelite molding powders are available in a variety of colors, which simplifies the identification of mounted specimens. The specimen and the correct amount of Bakelite powder, or a Bakelite preform, are placed in the cylinder of the mounting press. The temperature is gradually raised to 150°C, and a molding pressure of about 4,000 psi is applied simultaneously. Since Bakelite is set and cured when this temperature is reached, the specimen mount may be ejected from the molding die while it is still hot.

Lucite is the most common thermoplastic resin for mounting. Lucite is completely transparent when properly molded, as shown in Fig. 1·9*b*. This transparency is useful when it is necessary to observe the exact section that is being polished or when it is desirable for any other reason to see the entire specimen in the mount. Unlike the thermosetting plastics, the thermoplastic resins do not undergo curing at the molding temperature; rather they set on cooling. The specimen and a proper amount of Lucite powder are placed in the mounting press and are subjected to the same temperature and pressure as for Bakelite (150°C and 4,000 psi). After this temperature has been reached, the heating coil is removed, and cooling fins are placed around the cylinder to cool the mount to below 75°C in about 7 min while the molding pressure is maintained. Then the mount may be ejected from the mold. Ejecting the mount while still hot or allowing it to cool slowly in the molding cylinder to ordinary temperature before ejection will cause the mount to be opaque.

Small specimens may be conveniently mounted for metallographic preparation in a laboratory-made clamping device as shown in Fig. 1·9*c*. Thin sheet specimens, when mounted in such a clamping device, are usually alternated with metal "filler" sheets which have approximately the same hardness as the specimens. The use of filler sheets will preserve surface irregularities of the specimen and will prevent, to some extent, the edges of the specimen from becoming rounded during polishing.

1·17 Intermediate Polishing After mounting, the specimen is polished on a series of emery papers containing successively finer abrasives. The first paper is usually No. 1, then 1/0, 2/0, 3/0, and finally 4/0.

The surface after intermediate polishing on 4/0 paper is shown in Fig. 1·10*b*. The intermediate polishing operations using emery paper are usually done dry; however, in certain cases such as the preparation of soft materials, silicon carbide abrasive may be used. As compared to emery paper, silicon carbide has a greater removal rate and, as it is resin-bonded, can be used with a lubricant. Using a lubricant prevents overheating the sample, minimizes smearing of soft metals, and also provides a rinsing action to flush away surface removal products so the paper will not become clogged.

1·18 Fine Polishing The time consumed and the success of fine polishing depend largely upon the care that was exercised during the previous polishing steps. The final approximation to a flat scratch-free surface is obtained by use of a wet rotating wheel covered with a special cloth that is charged with carefully sized abrasive particles. A wide range of abrasives is available for final polishing. While many will do a satisfactory job, there appears to be a preference for the gamma form of aluminum oxide for ferrous and copper-based materials, and cerium oxide for aluminum, magnesium, and their alloys. Other final polishing abrasives often used are diamond paste, chromium oxide, and magnesium oxide.

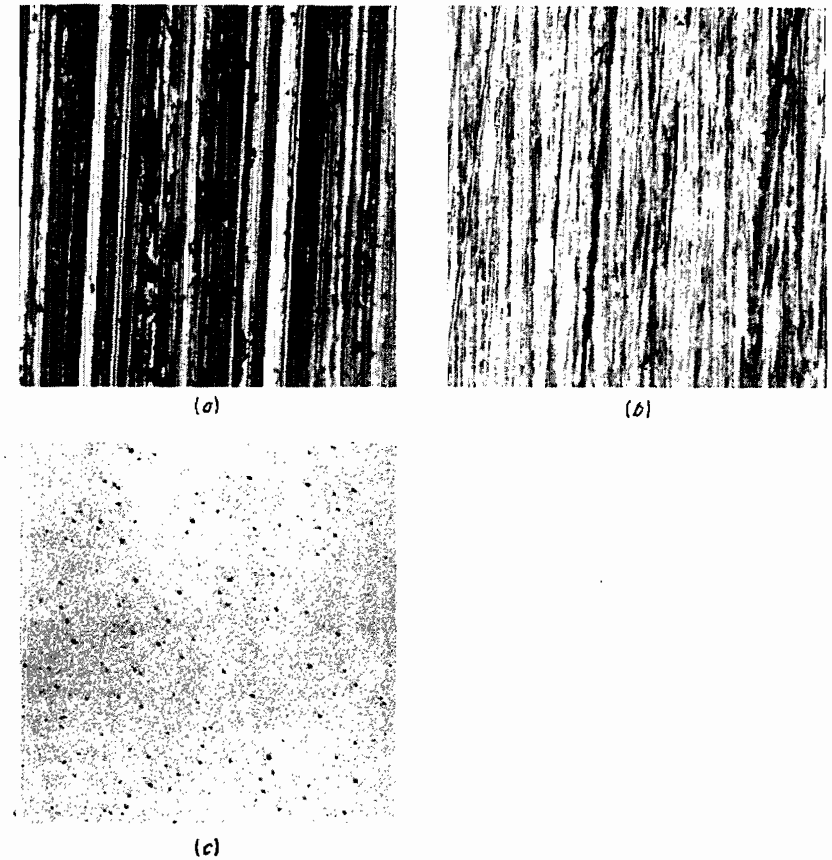


Fig. 1·10 (a) Surface after rough grinding, magnification 100X. (*b*) Surface after intermediate polishing on 4/0 paper, magnification 100X. (c) Scratch-free surface after final polishing, magnification 50X. Black spots are oxide impurities.

The choice of a proper polishing cloth depends upon the particular material being polished and the purpose of the metallographic study. Many cloths are available of varying nap or pile, from those having no pile, such as silk, to those of intermediate pile, such as broadcloth, billiard cloth, and canvas duck, and finally to a deep pile, such as velvet. Synthetic polishing cloths are also available for general polishing purposes, of which two, under the trade names of Gamal and Microcloth, are most widely used. A properly polished sample will show only the nonmetallic inclusions and will be scratchfree (Fig. 1·10*c*).

1·19 Etching The purpose of etching is to make visible the many structural characteristics of the metal or alloy. The process must be such that the various parts of the microstructure may be clearly differentiated. This is accomplished by use of an appropriate reagent which subjects the polished surface to chemical action.

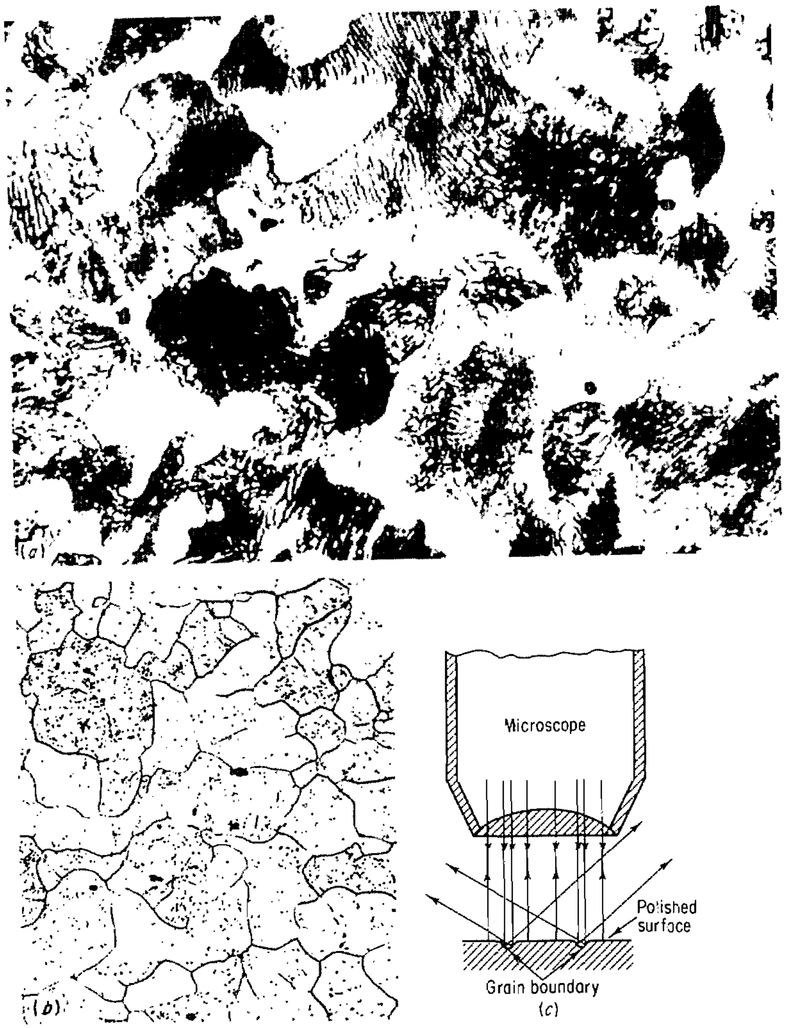


Fig. 1·11 (a) Photomicrograph of a mixture revealed by etching. (b) Photomicrograph of pure iron. (The International Nickel Company.) (c) Schematic illustration of the microscopic appearance of grain boundaries as dark lines.

In alloys composed of two or more phases, the components are revealed during etching by a preferential attack of one or more of these constituents by the reagent, because of difference in chemical composition of the phases (Fig. 1·11*a*). In uniform single-phase alloys or pure metals, contrast is obtained and grain boundaries are made visible because of differences in the rate at which various grains are attacked by the reagent (Fig. 1·11*b*). This difference in the rate of attack is mainly associated with the angle of the different grain sections to the plane of the polished surface. Because of chemical attack by the etching reagent, the grain boundaries will appear as valleys in the polished surface. Light from the microscope hitting the side of these valleys will be reflected out of the microscope, making the grain boundaries appear as dark lines. This is illustrated schematically in Fig. 1·11*c*.

The selection of the appropriate etching reagent is determined by the metal or alloy and the specific structure desired for viewing. Table 1·3 lists some of the common etching reagents.

1·20 Metallurgical Microscopes At this point it is appropriate to discuss briefly the principles of the metallurgical microscope. In comparison with a biological type, the metallurgical microscope differs in the manner by which the specimen is illuminated. Since a metallographic sample is opaque to light, the sample must be illuminated by reflected light. As shown in Fig. 1·12, a horizontal beam of light from some light source is reflected, by means of a plane-glass reflector, downward through the microscope objective onto the surface of the specimen. Some of this incident light reflected from the specimen surface will be magnified in passing through the lower lens system, the objective, and will continue upward through the plane-glass reflector and be magnified again by the upper lens system, the eyepiece. The initial magnifying power of the objective and the eyepiece is usually engraved on the lens mount. When a particular combination of objective and eyepiece is used at the proper tube length, the total magnification is equal to the product of the magnifications of the objective and the eyepiece. Figure 1·13*a* shows a table-type metallurgical microscope.

It is possible to mount a camera bellows above the eyepiece and use the table-type microscope for photomicrography. However, the bench-type metallograph illustrated in Fig. 1·13*b*, which is specifically designed for both visual examination and permanent recording of metallographic structures by photographic methods, will give superior photomicrographs.

The maximum magnification obtained with the optical microscope is about 2,000×. The principal limitation is the wavelength of visible light, which limits the resolution of fine detail in the metallographic specimen. The magnification may be extended somewhat by the use of shorter-wavelength radiation, such as ultraviolet radiation, but the sample preparation technique is more involved.

The greatest advance in resolving power was obtained by the electron

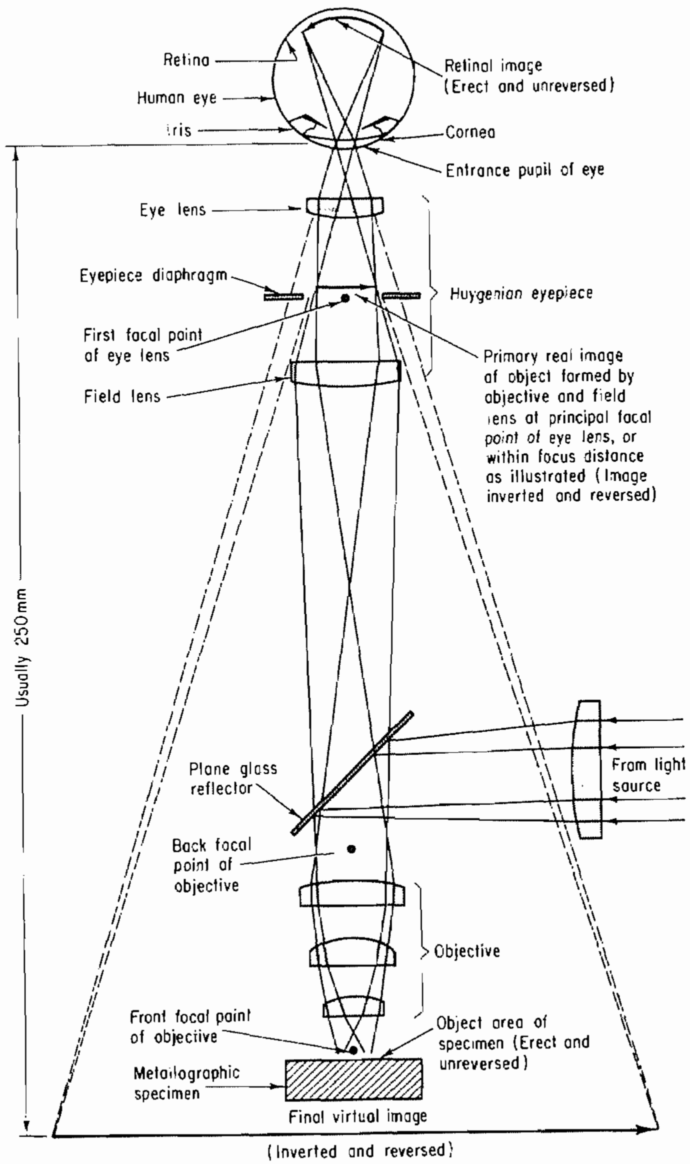


Fig. 1·12 Illustrating the principle of the metallurgical compound microscope and the trace of rays through the optical system from the object field to the final virtual image. (By permission from G. L. Kehl, "Principles of Metallographic Laboratory Practice," 3d ed., McGraw-Hill Book Company, New York, 1949.)

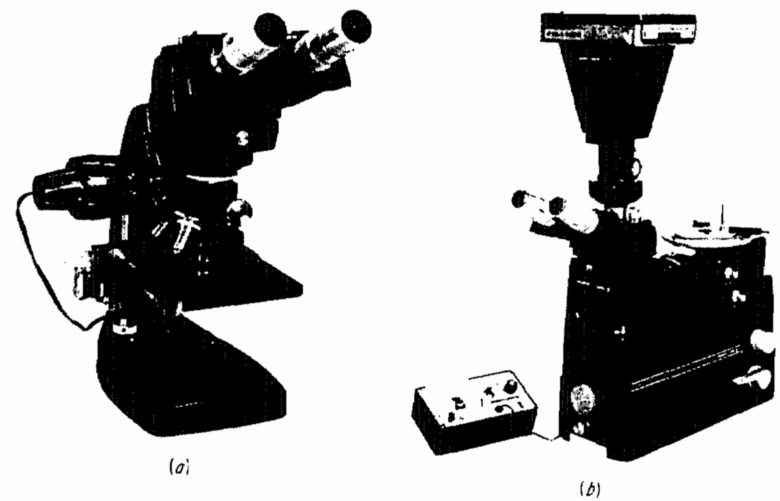


Fig. 1·13 (a) Metallurgical microscope. (b) Bench-type metallograph. (Bausch & Lomb, Inc.)

microscope. Under certain circumstances, high-velocity electrons behave like light of very short wavelength. The electron beam has associated with it a wavelength nearly 100,000 times smaller than the wavelength of visible light, thus increasing the resolving power tremendously. An electron microscope is shown in Fig. 1·14*a*.

Although in principle the electron microscope is similar to the light microscope (Fig. 1·14*b*), its appearance is very much different. It is much larger because of the highly regulated power supplies that are needed to produce and control the electron beam. The entire system must be kept pumped to a high vacuum since air would interfere with the motion of the electrons.

The lenses of the electron microscope are the powerful magnetic fields of the coils, and the image is brought into focus by changing the field strength of the coils while the coils remain in a fixed position. In the optical microscope the image is brought into focus by changing the lens spacing.

Since metallographic specimens are opaque to an electron beam, it is necessary to prepare, by special techniques, a thin replica of the surface to be studied. The specimen is polished and etched following normal metallographic practice. It is then placed on a hot plate with a small pellet of suitable plastic on the etched surface. As the temperature rises, the plastic begins to flow and pressure is applied to ensure intimate contact between the plastic and the surface. After cooling, the replica is carefully peeled off. To improve contrast, a thin coating of carbon or tungsten is evaporated onto the replica at an angle and from one side. Since the shadowed replica is fragile, it is supported on a disk of very fine copper-