

CHAPTER 2

THE PREPARATION OF SAMPLES FOR ORE MICROSCOPY

2.1 INTRODUCTION

The preparation of polished surfaces free from scratches, from thermal and mechanical modification of the sample surface, and from relief¹ is essential for the examination, identification, and textural interpretation of ore minerals using the reflected-light microscope. Adequate polished surfaces can be prepared on many types of materials with relatively little effort using a wide variety of mechanical and manual procedures. However, ore samples often present problems because they may consist of soft, malleable sulfides or even native metals intimately intergrown with hard, and sometimes brittle, silicates, carbonates, oxides, or other sulfides. Weathering may complicate the problem by removing cements and interstitial minerals and by leaving samples that are friable or porous. Delicate vug fillings also cause problems with their open void spaces and poorly supported crystals. Alloys and beneficiation products present their own difficulties because of the presence of admixed phases of variable properties and fine grain sizes. In this chapter, the general procedures of sample selection and trimming, casting, grinding, and polishing (and, in special cases, etching) required to prepare solid or particulate samples for examination with the ore microscope are discussed. The preparation of these *polished sections*, and also polished and doubly polished *thin sections*, which are useful in the study of translucent or coexisting opaque and translucent specimens, is described. These are matters about which all students of reflected-light microscopy should be aware, ideally through personal experience.

¹*Relief* is the uneven surface of a section resulting from soft phases being worn away more than hard phases during polishing.

2.2 PREPARATION OF POLISHED SECTIONS

The procedures involved in the preparation of polished sections vary in detail from one laboratory to another, depending on both the type of material to be studied and the actual objectives of the study. A typical sequence of steps is outlined in Figure 2.1 and is described in greater detail below.

2.2.1 Initial Preparation and Casting of Samples

Coherent materials are easily cut to size with a diamond saw. Lubrication of the saw blade by water, kerosene, or a cutting oil to prevent heating of the specimen during cutting is important. If the sample is suspected of containing phases that are either soluble (e.g., metal sulfates or chlorides) or reactive with water (e.g., some rare sulfides in meteorites), it will be necessary to conduct all cutting and subsequent operations using appropriate nonreactive fluids (oils,

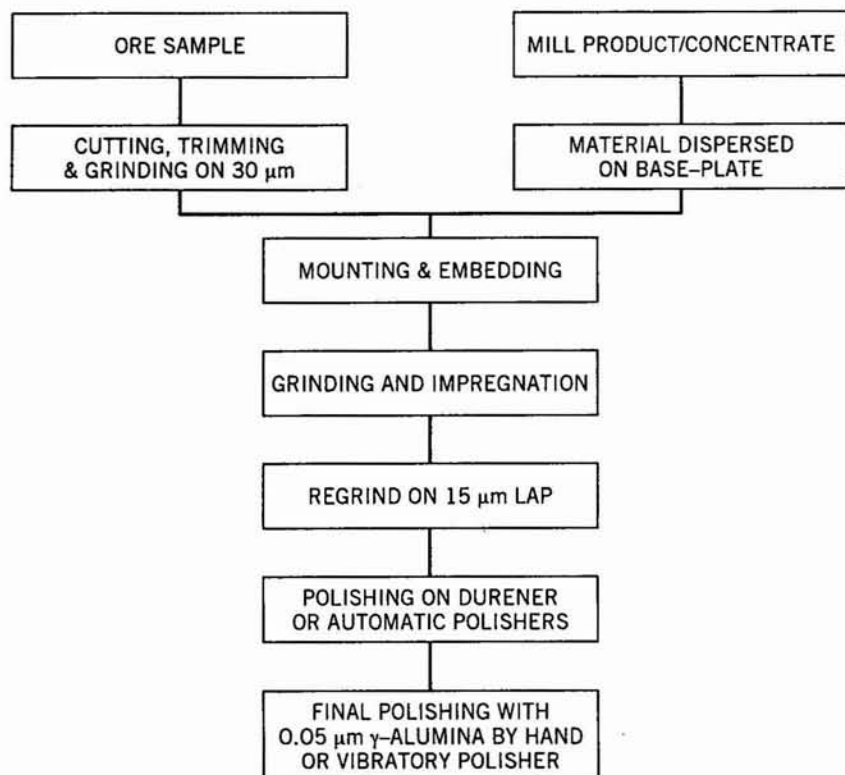


FIGURE 2.1 Flowsheet outlining the steps in the preparation of polished sections.

alcohols, kerosene, etc.). In all cases, it is necessary to remove cutting oils and water and to dry samples thoroughly before casting, because such fluids may react with the epoxy and reduce its bonding strength. Furthermore, any trapped fluids can, in time, bleed out onto the surface and create serious problems in conducting analysis where high vacuum is needed.

The size and shape of the sample to be mounted is somewhat arbitrary and must be dictated by the nature of the material and the intended study; most workers find that circular polished sections from 2.5 to 5 cm in diameter are adequate. The casting of samples may be in one- or two-piece cylindrical plastic, polyethylene, or metal molds (Figure 2.2) or in cylindrical plastic rings that remain a permanent part of the sample. When molds are used, a thin coating of wax or other nonreactive lubricating agent (e.g., silicon stopcock grease, or vaseline) is extremely helpful in the removal of specimens once the casting resin has hardened. Sample thickness is also arbitrary, but the total thickness of the polished section generally need not exceed 1–2 cm. The maximum thickness is often governed by the working height between the microscope stage and the lens.

If the specimen is coherent and has a low porosity, it may be cut to the desired size and polished directly. Commonly, however, specimens are cast in a mounting resin to facilitate handling and to minimize the problems of crumbling and plucking. The preparation of a flat surface on the specimen by cutting and grinding prior to casting is a very useful step, because it allows the sample to lie flat on the bottom of the mold; irregular specimens may trap air bubbles and require extensive grinding of the polished section to expose sufficient surface area. It is useful to bevel the edges of the surface to be polished; this helps to prevent material being dislodged from, or trapped between, the sample and mounting medium. Also, it may be desirable to cut and mount a sample in specific orientations, because *plucking* (the tearing of material from the surface during polishing) may occur when cleavage or fracture directions are appropriately oriented. If the specimen is friable, fractured, porous, or consists of loose grains or powders, impregnation is a useful procedure to prevent grain plucking and trapping of abrasive during grinding and polishing. Impregnation is readily accomplished by placing the mold containing the speci-

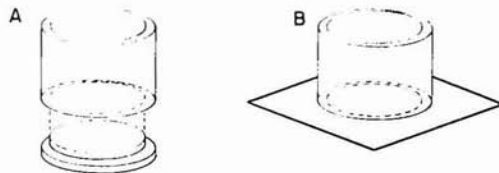


FIGURE 2.2 Sample cups used for the casting of cylindrical polished sections. (A) Two-piece cup. (B) Plastic ring that becomes a portion of the polished section after emplacement of the sample and the mounting resin.

men and mounting resin under vacuum for several minutes; this draws air from the voids, and the subsequent release of the vacuum tends to drive the mounting resin into the voids. Sometimes repeated brief vacuum treatments are needed to drive the resin into deep cracks and voids.

It is important that the procedures involved in specimen preparation preserve the precise mineralogical and textural character of the samples. The recognition in recent years that certain common sulfide minerals (e.g., anilite, Cu_7S_4 , and djurleite, $\text{Cu}_{1.96}\text{S}$) break down below 100°C and that many other ore minerals have unknown thermal stabilities points to the necessity of using cold-setting mounting media, requiring neither heat nor pressure for preparation. Older procedures that require elevated temperatures and high pressure (e.g., the use of bakelite as a mounting medium) and that are still commonly used in metallurgical laboratories should be avoided. Such procedures may not alter refractory ore mineral assemblages (e.g., chromite, magnetite, pyrite) but may profoundly modify low temperature or hydrous mineral assemblages. Even heating for brief periods at temperatures as low as 100°C will cause decomposition of some phases and induce twinning or exsolution in others. Accordingly, virtually all mineralogical laboratories now use cold-setting epoxy resins or plastics; these are readily available and easily handled. Most of the epoxy resins are translucent and nearly colorless, which facilitates labeling, because paper labels can be set in the resin with the specimen. Lower-viscosity mounting media generally penetrate cracks and voids in samples better than higher-viscosity ones—an important point when one is dealing with friable or porous samples. If samples are also to be used for electron microprobe analysis (see Appendix 3) or some other high-vacuum instrument, one must be careful to choose a resin that does not create problems in the instrument sample chamber by degassing or volatilizing.

2.2.2 Grinding and Polishing of Specimens

Once a sample has been cut to an appropriate size and cast in a mounting medium, it is ready for grinding and polishing. Large research or industrial laboratories commonly employ automated grinding and polishing machines capable of handling large numbers of specimens. Smaller laboratories are usually equipped with either fixed- or variable-speed rotary lap equipment designed to prepare one sample at a time. The procedures described below are general and apply to any laboratory; experienced workers invariably develop favorite "tricks" and minor modifications but follow the same basic steps outlined.

The purpose of grinding is to remove surface irregularities, to remove casting resin that covers the sample, to reduce thickness, to prepare a smooth surface for further work, and to remove any zone of major deformation resulting from initial sample cutting (Figure 2.3). Many workers have found that fixed abrasives (such as adhesive-backed emery paper or diamond embedded in metal or epoxy) are superior to loose abrasives, because the latter tend to roll and leave irregular depth scratches rather than planing off a uniform surface.



FIGURE 2.3 Schematic cross section of a polycrystalline sample illustrating the nature of deformation from the cut or coarsely ground surface down to the undeformed sample. The nature and depth of deformation varies with the nature of the sample.

Grinding and polishing compounds may be designated in terms of *grit*, *mesh*, or *micron* grain sizes; the equivalence of terms is shown in Figure 2.4 and in Table 2.1 Successive grinding steps using 400- and 600-mesh silicon carbide have proven adequate in preparing most surfaces, although the 400-mesh abrasive is really needed only for harder materials and many sections made up of soft sulfides can be started on a 600-mesh abrasive. It is important at each step to prevent any heating of the specimen or to avoid contamination by the carry-over of abrasive from one step to the next. Samples should be thoroughly washed and cleaned (with an ultrasonic cleaner, if available) between steps, and it is convenient to process samples in batches. This requirement for cleanliness also applies to an operator's hands, because abrasives and polishing compounds are readily embedded in the ridges of one's fingertips. Grinding and polishing laps should be covered when not in use to prevent contamination by stray particles and airborne dust. If samples begin to pluck, one is well advised to stop the polishing and coat or impregnate with a mounting medium and then begin again; otherwise, the plucked grains probably will contaminate all grinding and polishing laps and leave the plucked sample surface and all subsequent samples highly scratched. Careful attention to the grinding steps is important in the preparation of good polished surfaces, because these steps should provide a completely flat surface and progressively remove surface layers deformed in previous steps. Even in compact specimens, the fracturing from a high-speed diamond saw may extend 1 mm or more from the cut surface.

Grinding may be completed with a 600-mesh abrasive compound or with 1,200-mesh abrasive used manually on a glass plate; the matte-like surface of a typical sample at this stage is shown in Figure 2.5a. It should be noted that some features, such as grain size and shape in sphalerite, are actually more easily seen megascopically when a sample has a good 600- or 1,200-mesh matte-like surface. The ground surface readily reveals the different grains, because orientation-controlled cleavages or plucking reflect light differently. Final polishing removes the surface imperfections and the features they reveal. Rough polishing, involving the use of a fabric loaded with 15 and 6 μm abrasives, removes most or all of the remaining zone of surface deformation and the deeper scratches, and prepares the sample for final polishing; the appearance of the sample after the 15 and 6 μm steps is shown in Figures 2.5b and 2.5c. The first polishing is best accomplished using diamond abrasives

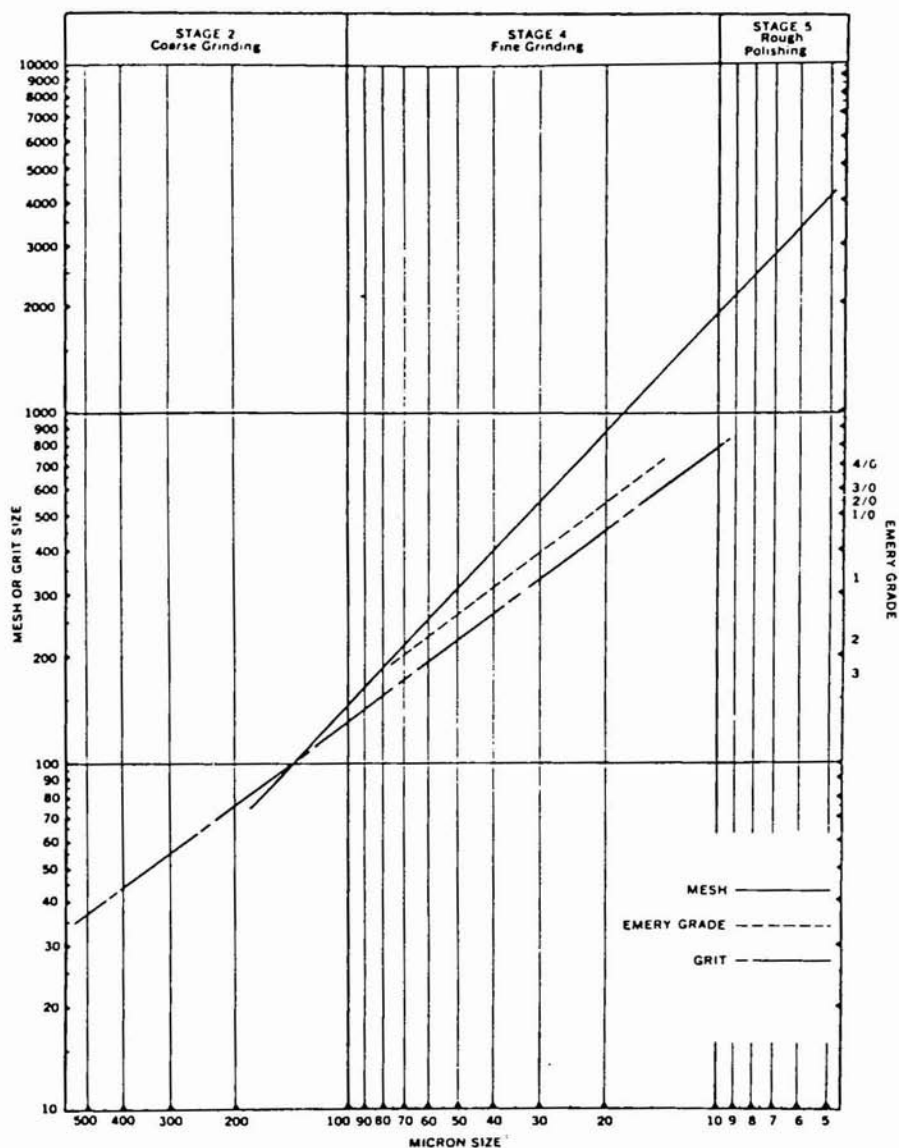


FIGURE 2.4 Relationship between grit, mesh, and micron size. Silicon carbide abrasive powders are generally classified in mesh or grit size. These are plotted on the left of the graph. The right-hand ordinate is scaled for emery grit designation. For conversion to micron size, locate the size range on the proper ordinate and transpose this point to the correct graph line while locating this point on the abscissa. Reverse this procedure to transpose micron to grit size. (Diagram courtesy of Buehler Ltd.)

TABLE 2.1 Dimensions of Mesh or Grit Size
Abrasives

Mesh or Grit Size	Microns	Inches
100	150	0.0059
200	75	0.0030
260	60	0.0024
325	45	0.0018
400	38	0.0015
600	30	0.0012
1,200	15	0.00059
1,800	9	0.00035
3,000	6	0.00024
8,000	3	0.00012
14,000	1	0.00004
60,000	1/2	0.00002
120,000	1/4	0.00001

embedded in a napless cloth. The embedding permits the diamond grains to plane the surface without rolling and causing irregular scratching or gouging. Use of a hard napless fabric without excessive weight pressing down on the sample minimizes the development of surface relief. The distributors of diamond abrasives recommend that a 1–2 cm strip of diamond paste be placed on the polishing lap at right angles to the direction of movement and that subsequent dispersal on the surface of the lap be done by dabbing the diamond about with a clean fingertip. Polishing with 6 μm abrasives is the most important stage and should be carried out fairly slowly with the surface just sufficiently lubricated for a smooth action and without too much weight applied to the section. Polishing is continued until no deep scratches are seen in even the hardest phases. It may be useful to study the section at this stage, since information lost later in polishing (e.g., grain boundaries of isotropic minerals) may be obtained. Again, care must be taken in this and subsequent steps to avoid excessive heating of the sample surface during the polishing.

Final polishing, using abrasives of less than 6 μm , removes only a very small amount of the specimen surface and should produce a relatively scratch-free surface (Figures 2.5d–2.5f). This step may be accomplished using 1 μm diamond embedded in a napless cloth, perhaps followed by 0.25 μm diamond cloth, or by using $\alpha\text{-Al}_2\text{O}_3$ (1–0.3 μm) or $\gamma\text{-Al}_2\text{O}_3$ (0.05 μm) suspended in water on a napped cloth. Some laboratories have also found that chromic oxide, magnesia, and cerium oxide can be useful in final polishing, especially of metallurgical samples. Napped cloths are very useful in the final polishing and when a quick buffing of a slightly oxidized surface is required, but such cloths very rapidly produce relief on samples containing several minerals of differing hardness.

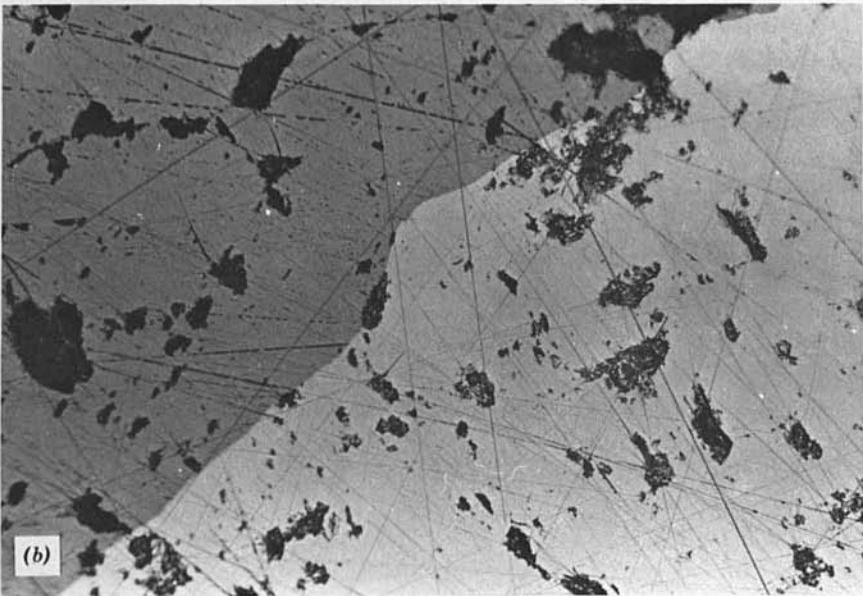
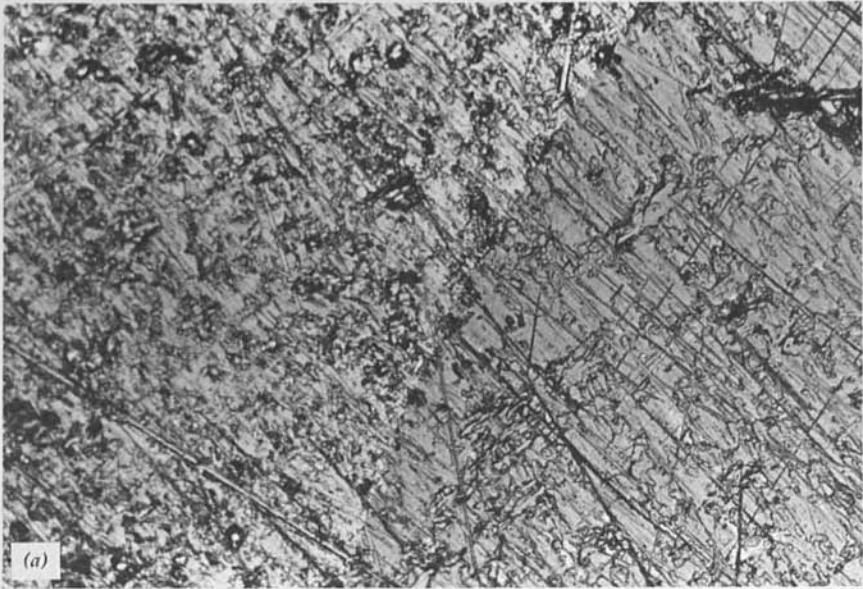


FIGURE 2.5 Polished surfaces of coexisting chalcopyrite (light) and sphalerite (dark) after abrasion by (a) 600-mesh silicon carbide; (b) 15 μ diamond; (c) 6 μ diamond; (d) 1 μ diamond; (e) 0.05 $\mu\gamma$ - Al_2O_3 on microcloth; (f) γ - Al_2O_3 on silk in a vibropolisher but without the intermediate steps of 15, 6, and 1 μ diamond (width of field = 520 μm).

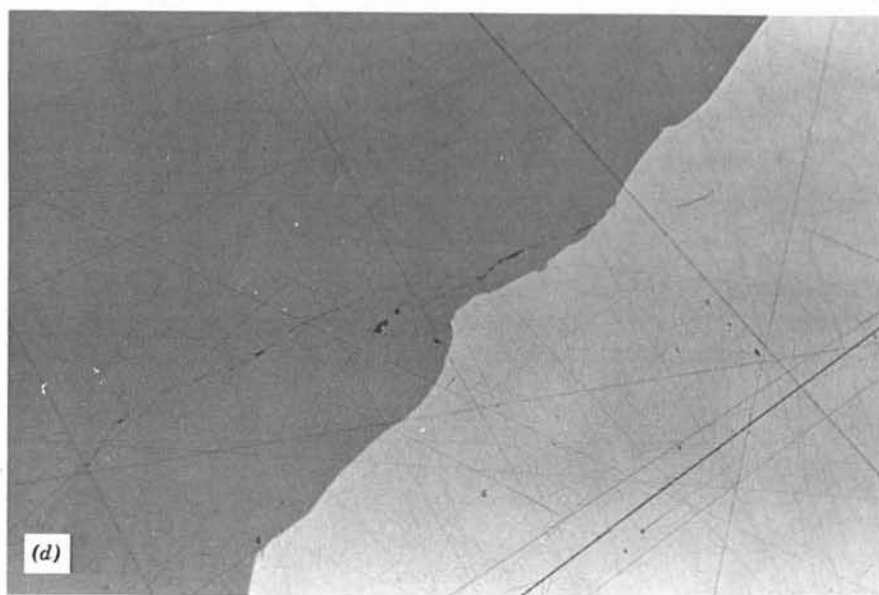
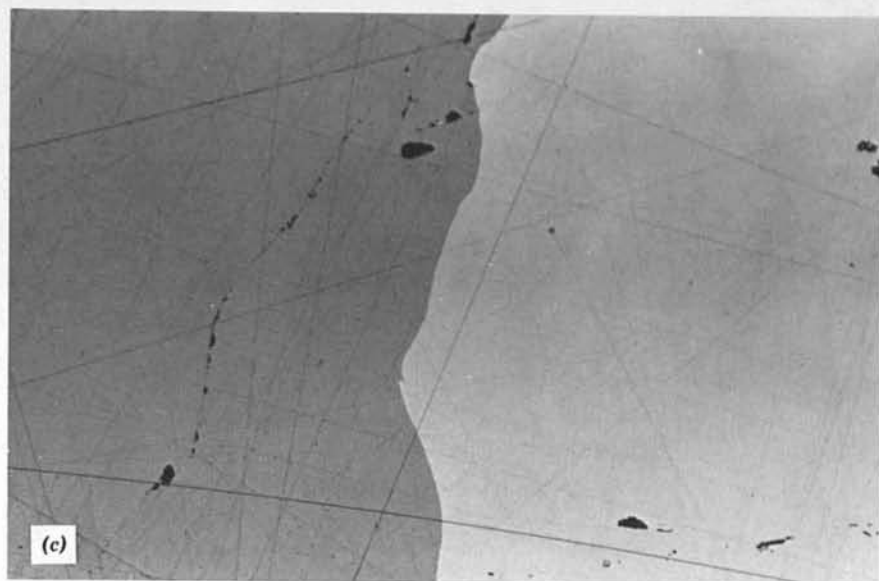


FIGURE 2.5 (Continued)

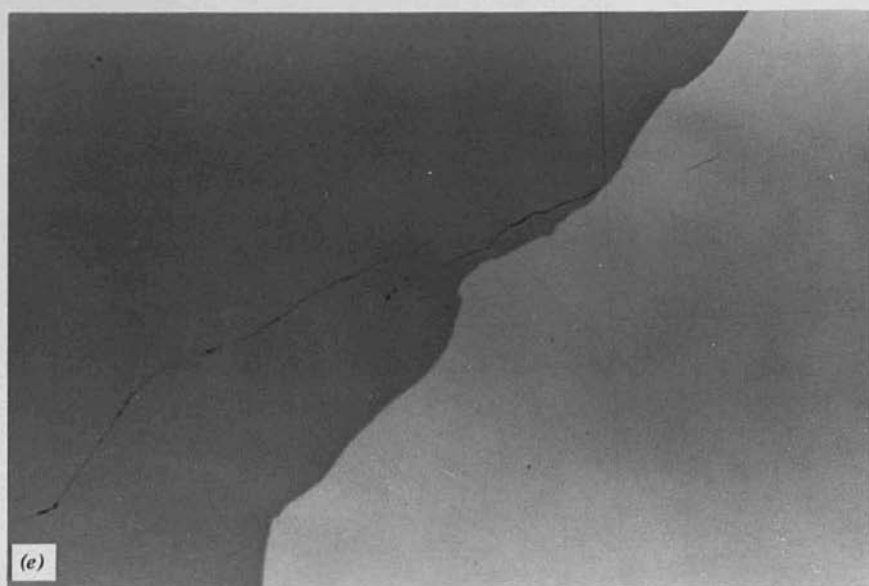


FIGURE 2.5 (Continued)

Some laboratories prepare sections using machines capable of polishing six or more specimens simultaneously on grooved cast-iron, copper, or lead laps (see the following section). Grooved laps require constant resurfacing but, if properly maintained, allow preparation of excellent relief-free sections. Specific instructions accompany each type of machine, but the polishing steps broadly parallel those outlined above.

Vibropolishing equipment (see the following section) is efficient and effective in the final polishing of many types of specimens. The quality of the surface and the time necessary to achieve a satisfactory polish depends on the prior grinding steps, the hardness, and the homogeneity of the specimen. The lap of the vibropolisher is usually covered with a taut piece of finely woven silk, nylon, or other hard fabric; the unit is filled with water to a depth of 2–3 cm and a small amount of 0.3 μm α -alumina or 0.05 μm γ -alumina. Because these very fine grained compounds remove surface layers only slowly, careful prior grinding down to the 600-mesh abrasive size, or finer, is necessary to ensure flatness and uniformity of sample surface; otherwise, polishing, which for the common sulfides and oxides takes only a few hours, will require several days and even then produces only a mediocre polish.² A drawback of vibropolishing units is the tendency for polished surfaces to develop considerable relief (Figure 2.6), especially when soft and hard minerals are intergrown. Such relief may obscure contact relationships between minerals, may make the identification of soft rimming phases and inclusions difficult, and may handicap attempts at photomicroscopy and electron microprobe analysis. Nevertheless, the simplicity of operation, easy maintenance, and capability of handling many samples simultaneously make vibropolishers useful.

2.3 GRINDING AND POLISHING EQUIPMENT

Several types of equipment are commonly used in the grinding and polishing of specimens for ore microscopy. Most, if properly operated, are capable of preparing adequate surfaces, free from scratches and with a minimum of relief. A few of the more popular varieties of equipment (as shown in Figure 2.7) include the following.

1. *Multiple-specimen automated grinding-polishing machines* (Figure 2.7a). These units hold six or more samples, which are mounted on spindles that rotate clockwise as they move on a lap that rotates counterclockwise. Polishing is accomplished on grooved metal (usually lead) laps coated with a thin layer of abrasive mixed with oil.
2. *Variable-speed manual grinding and polishing machines* (Figure 2.7b). These are popular units because of their rugged design and easy adaptability to perform a wide variety of tasks. Samples are manually (or

²Samples are generally held in stainless steel or brass weights in order to increase the polishing speed and to insure that samples do not merely slide about without receiving any polish.

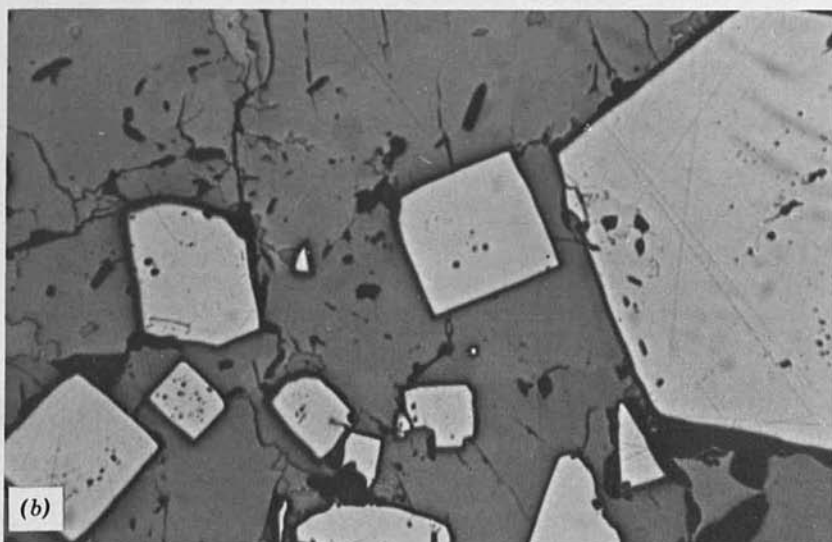
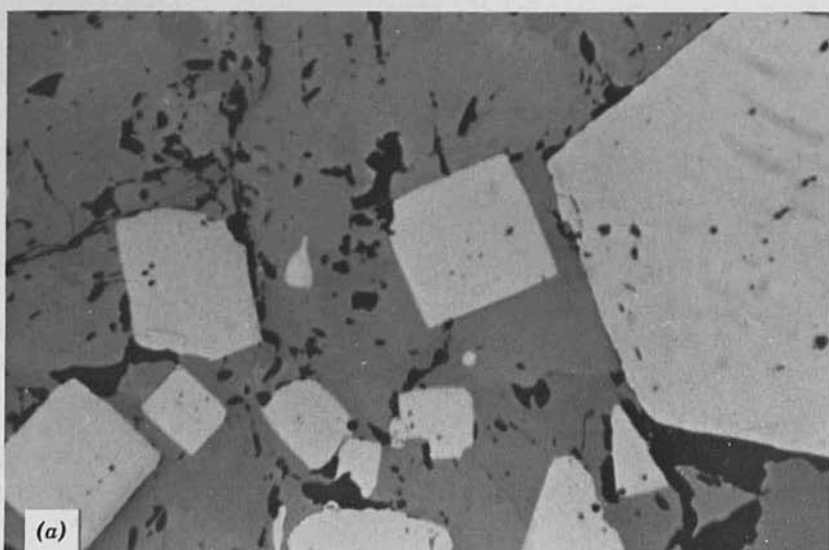


FIGURE 2.6 Comparison of the relief developed in the same sample, which contains a hard mineral (pyrite as cubes) and a soft mineral (pyrrhotite) when polished: (a) by polishing with 15, 6, 1 μ diamond followed by 0.05 μg - Al_2O_3 on microcloth (briefly) and (b) by using a vibropolishing machine charged with 0.05 γ - Al_2O_3 . The relief is evident in the photomicrograph as the dark shadow around the edges of the pyrite (width of field = 100 μm).



(a)



(b)



(c)

FIGURE 2.7 Examples of the types of equipment that are commercially available for the preparation of polished sections: (a) large automated Dürener grinding and polishing machine capable of preparing several samples simultaneously; (b) variable-speed manual grinding and polishing machine, Buehler Ecomet 3; (c) automated grinding and polishing machine capable of handling six to eight samples, Buehler Ecomet 4 with Universal Sample holder; (d) Syntron electro-magnetic vibrating polishing machine, FMC Corporation. (All photographs courtesy of the manufacturers.)



FIGURE 2.7 (Continued)

sometimes mechanically) held as they are ground and polished on interchangeable fixed abrasive paper- or fabric-covered laps. Most units have variable-speed motors, although less expensive models may possess only one or two fixed speeds. Most polishing is done on napless cloths in which the polishing compounds become embedded. Final polishing or buffing of surfaces commonly employs a napped cloth.

3. *Small automated polishing machines* (Figure 2.7c). These units have become very popular because of their low price, compact size, and ease of operation. Samples are held on an arm that moves them in an eccentric motion on paper or fabric lap surfaces that adhere to glass plates. The glass plates are contained in individual bowls that are conveniently interchanged between steps.
4. *Vibropolishing equipment* (Figure 2.7d). Vibropolishing equipment is convenient in the preparation of large numbers of sections with a minimum of operator attention. Polished sections are held in cylindrical brass or stainless steel weights and ride about the circular lap as a result of the vibratory motion. The lap surface is covered with a silk, nylon, or other hard cloth that is stretched taut and covered with an abrasive slurry or water; a small amount of MgO or Al_2O_3 is dispersed on the lap. Samples polish slowly but effectively and require virtually no attention except watching the water level and the wear of the polishing cloth.

2.4 PREPARATION OF GRAIN MOUNTS AND MOUNTS FOR SPECIALIST ANALYTICAL METHODS

Loose mineral grains from placer deposits or extracted from larger specimens by crushing may be cast into polished sections in several ways. The most direct technique is that of pouring embedding resin into the mold and then dispers-

ing the mineral grains over the surface and allowing them to sink to the bottom. Three potential problems of this technique are (1) the trapping of air bubbles (2) the stratification of mineral grains due to differential rates of settling through the resin, and (3) differential exposure of grains of differing sizes. Air bubbles can usually be removed by using the vacuum impregnation techniques described in Section 2.3. Stratification can sometimes be avoided by merely dispersing grains such that all, or at least representatives of all, types of grains rest on the bottom of the mold. Alternatively, one can cast the grains and then section the grain mount vertically to expose any layering present. This vertical section through the grain mount may be polished directly or recast in another section such that the layering of grains is visible.

If one is dealing with only one or a few selected grains (e.g., in placer gold samples) or of grains of differing sizes, the most efficient technique is often to place the grains either on a glass slide or on a polished section "blank" and to cover them carefully with a few drops of resin. In this manner, the grains may be placed in a pattern for easy recognition and their relative heights controlled so that all are exposed simultaneously during polishing. Grains may even be specifically oriented, if desired, by the use of small supporting devices or by carving or drilling appropriately shaped holes into the underlying polished section blank.

The mounting of specimens for such specialist techniques as image analysis, micro-PIXE analysis, or secondary ion mass spectrometry (SIMS) may require some special methods, as noted by Laflamme (1990). For image analysis, it is very important that the section be truly representative of the sample and that contamination through trapping of polishing compounds (particularly lead) in holes (such as may be left by air bubbles in epoxy) at the polished surface be avoided. In micro-PIXE analysis, the standard techniques may be used, but, in order to maximize the efficiency of the analysis, selected monomineralic areas may be cut (as 2–4 mm cubes and using a low-speed saw) from a suite of samples already studied in polished section. These samples can be remounted as arrays with as many as 40 such cubes and including analytical standards. In this way, costly instrument time is more efficiently used. Both in micro-PIXE and SIMS analysis, problems of the conducting away of heat and electrical charge can be overcome by using graphite (20–25 wt %) mixed with the mounting medium (araldite or equivalent). In all such specialist techniques, as further discussed in Appendix 3, sample mount shape, diameter, and thickness will be dictated by the sample holder and sample chamber of the instrument being used.

2.5 PREPARATION OF POLISHED (AND DOUBLY POLISHED) THIN SECTIONS

The thin section, a ~0.03 mm thick slice of rock, has for many years been the standard type of sample employed in petrological microscopy. The need to examine the opaque minerals in rocks and to be able to analyze all of the

minerals in thin sections using the electron microprobe has led to the preparation of polished thin sections by simply polishing the upper uncovered surface of a thicker-than-normal thin rock slice (Figure 2.8).

Although the study of most ore samples still involves conventional polished sections, there has been an increased awareness that important textural information in some ore minerals is not readily visible in conventional polished sections, thin sections, or even polished thin sections. In polished sections or polished thin sections, it is evident that most sphalerite, cassiterite, cinnabar, tetrahedrite, tennantite, ruby silvers, rutile, and copper oxides transmit light, but the diffuse light scattering from the rough underside of the thin section makes recognition of some internal features difficult. The difficulty is readily overcome if doubly polished thin sections are employed. These are prepared either by cutting standard thin-section blanks, which are polished and then glued to a glass slide, or by gluing conventional polished sections to a glass slide. The excess blank or the polished section is then cut away from the slide by a *wafaring* saw (a slow-speed saw is especially useful, because it minimizes the amount of fracturing and distortion within the sample). The thin slice on the glass slide is then gently ground until it transmits sufficient light to suit the worker's needs, and it is given a final polish in the normal manner. The result is a section that combines certain advantages of both the polished section and the thin section by allowing simultaneous study of ore and gangue minerals, and a section that may reveal internal structure otherwise missed (see Figure 2.9 and Chapter 7). Excellent examples are given by Barton (1978) and by Eldridge, Barton, and Ohmoto (1983).

The preparation of samples for fluid inclusion studies is very similar and is described in detail in Chapter 8.

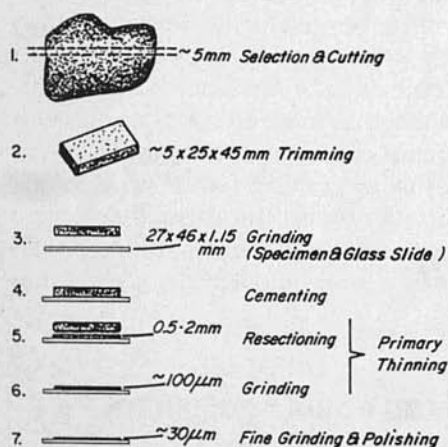


FIGURE 2.8 Steps in the preparation of polished thin sections: (1) specimen selection and cutting to ~5 mm in thickness; (2) trimming to ~25 × 45 mm; (3) grinding of the specimen side to be cemented with epoxy on a preground glass slide; (4) cementing of specimen to glass slide; (5) resectioning to 0.5–2 mm thickness using a low-speed saw; (6) rough grinding by hand with SiC or with an automatic grinder to ~100 μm; (7) final thinning by hand using SiC of ~35 μm, followed by polishing to a final thickness of 25–30 μm.

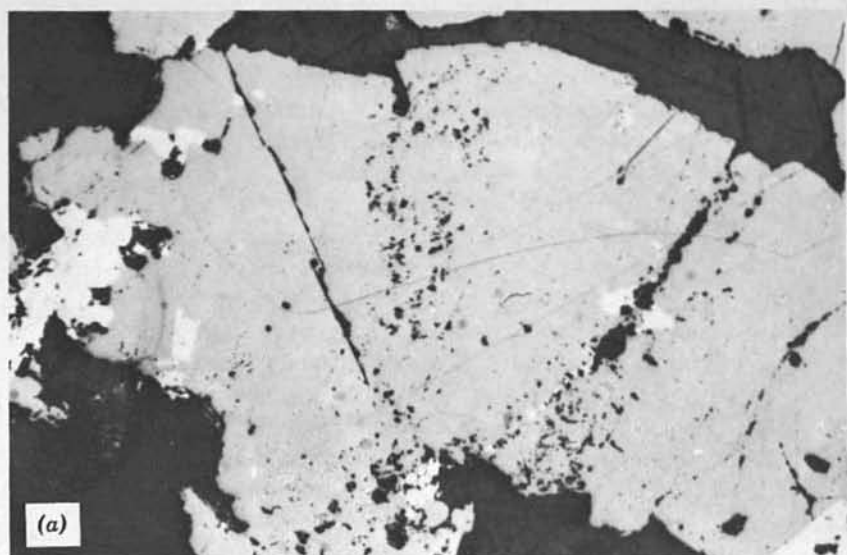


FIGURE 2.9 Comparison of the structure visible in the same sample of sphalerite when viewed: (a) in normal reflected light and (b) in transmitted light in a doubly polished thin section. (Figures courtesy of P. B. Barton, reproduced from *Min. Geo.* 28, 296, 1978, with the publisher's permission.)

2.6 ELECTROLYTE POLISHING AND ETCHING TECHNIQUES

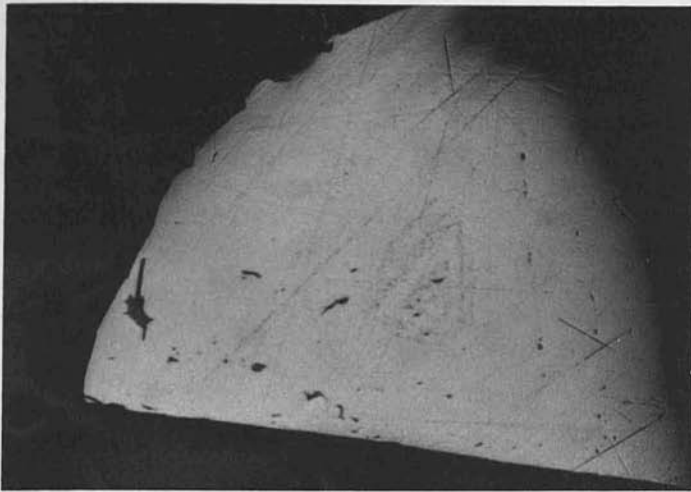
Metallurgists, and to a lesser extent mineralogists, have supplemented conventional polishing methods with electrolytic and chemical-mechanical techniques. In the former, electrically conductive samples, ground only as far as 600-mesh abrasive, are polished with $0.05\ \mu\text{m}$ Al_2O_3 suspended in an electrolyte fluid. The sample is attached as anode and the polishing lap as cathode in a 25 V DC circuit. Polishing occurs by the removal of material from the sample by both mechanical and electrical stripping. Chemical-mechanical polishing employs a fine polishing compound in a chemically reactive fluid (commonly an etchant). The surface is prepared by the combined mechanical polishing and the chemical etching. Care must be taken when using chemical polishing techniques, because they may selectively remove some phases (e.g., chromic acid/rouge may result in the loss of native silver or bismuth).

Chemical etchants were widely used in mineral identification prior to the development of the electron microprobe (see Appendix 3). Although now less widely used for identification, etchants are still useful to enhance mineral textures. Reaction of the specimen with various chemicals in liquid or vapor form will often reveal mineral zoning, twinning, and grain boundaries in apparently homogeneous material and will accentuate differences in phases that are nearly identical in optical properties. A typical example of etching to enhance growth zoning is illustrated in Figure 2.10a and 2.10b, in which pyrite is shown before and after etching with concentrated HNO_3 . In Figure 2.10c, the distinction between monoclinic and hexagonal pyrrhotite is made by etching with ammonium dichromate-HCl solution. Monoclinic pyrrhotite is also distinguished by use of a magnetic colloid, which will coat grains of this mineral while leaving grains of hexagonal pyrrhotite uncoated. Metallurgical laboratories have found for many years that a wide variety of etchants bring out alloy microstructures that are otherwise invisible. It is not possible to record all of the numerous etch reagents described in the literature, but Table 2.2 includes many of the most useful etchants used in mineralogical and metallurgical studies. (*Note:* Several of these etchants contain strong acids or oxidants; their preparation should be done in a chemical fume hood while wearing safety glasses and observing standard chemical laboratory safety procedures. Care must be taken during use so as to avoid chemical burns to the hands, clothing, or laboratory equipment.)

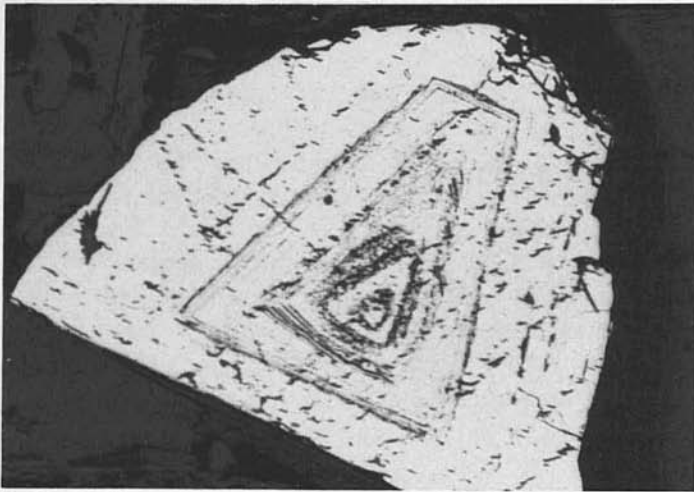
2.7 HOW TO ACHIEVE HIGH-QUALITY POLISHED SURFACES

A number of key points essential to the preparation of high-quality polished surfaces are worth emphasizing, as pointed out by Laflamme (1990):

1. Ensure that the specimen for preparing the polished surface is representative of the sample as a whole.



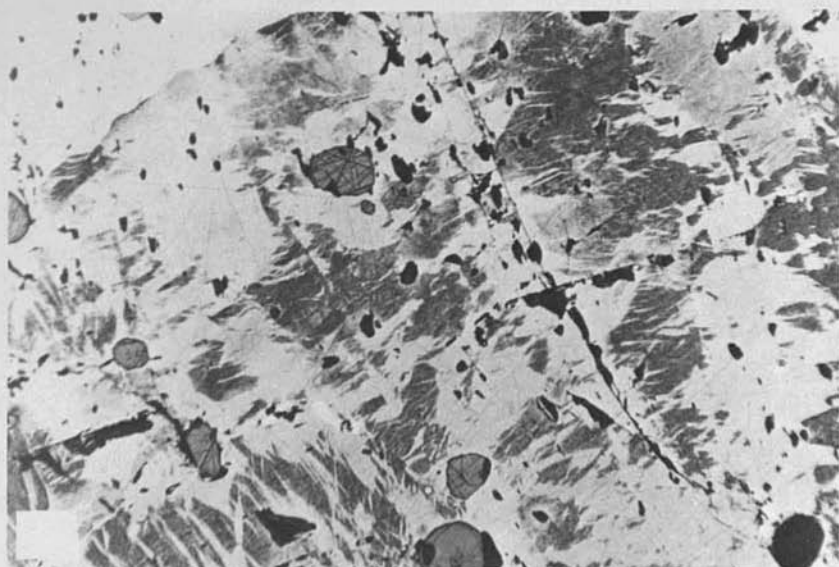
(a)



(b)

FIGURE 2.10 Etchants may be used to enhance structures within grains, as shown in (a) a freshly polished pyrite grain, and (b) the same grain after etching with concentrated HNO_3 , Mineral District, Virginia (width of field = 1.2 mm). (c) Etchants may also enhance the difference between hexagonal pyrrhotite (dark) and monoclinic pyrrhotite (light), Sudbury, Ontario (width of field = 1,300 μm). (Reproduced from *Econ. Geol.* **66**, 1136, 1971, with the publisher's permission).

2. Use a cold-setting mounting medium; epoxy is ideal because of its low shrinkage and high adhesive strength.
3. Ensure cleanliness throughout the entire polishing procedure.



(c)

FIGURE 2.10 (Continued)

4. Fixed abrasives are generally superior to loose abrasives for use during the grinding operation, which is by far the most important stage in preparation.
5. Impregnation followed by careful grinding will prevent contamination problems and will produce a higher-quality polish; coarse grinding (with $>30\ \mu\text{m}$ abrasive particles) is best avoided to minimize damage to the specimen.
6. An oil-based lubricant is needed if the specimen is suspected of containing water-sensitive minerals, and washing should then be done using ethyl alcohol.
7. Lead contamination can be a serious problem if metal laps are used; when techniques such as imaging (and image analysis; see Chapter 11) using back-scattered electrons are employed, then cloth or paper laps should be used with diamond abrasives.
8. Vibropolishing is recommended only as a final polishing step, particularly for soft minerals and metals.
9. The quality of the final polished surface depends directly on the amount of care and attention devoted to details at each stage of preparation.

To conclude then, high-quality polishes can be obtained on most materials, provided that care, constant attention, and especially cleanliness are employed during all stages of the polishing process. Without good-quality polished surfaces, any attempts to apply the qualitative and quantitative methods of ore microscopy and ore petrography that are described in this book will be of little, or limited, value.

TABLE 2.2 Etchants Useful in the Enchantment of Mineral Textures^a*Some General Etchants*

HNO₃-concentrated; 1 part HNO₃ to 1 part H₂O; 1 part HNO₃ to 7 parts H₂O
 HCl-concentrated; 1 part HCl to 1 part H₂O; 1 part HCl to 5 parts H₂O
 H₂SO₄-concentrated; 1 part H₂SO₄ to 1 part H₂O; 1 part H₂SO₄ to 4 parts H₂O
 HI-concentrated; 1 part HI to 1 part H₂O
 HBr-concentrated
 Aqua Regia—1 part concentrated HNO₃ to 3 parts concentrated HCl
 HCl—1 part concentrated HCl to 3 parts dilute (100 g/l) thiourea solution
 Picric acid—4 g in 100 cm³ ethyl alcohol
 KMnO₄—2.5 g in 100 cm³ H₂O; 1 part to 1 part concentrated HNO₃; 1 part to 1 part concentrated H₂SO₄; 1 part to 1 part KOH
 (NH₄)₂Cr₂O₇—400 mg in 25 cm³ of 15% HCl
 KCN—20 g in 100 cm³ H₂O
 FeCl₃—20 g in 100 cm³ H₂O; 50 g in 100 cm³ H₂O
 SnCl₂—1 part saturated solution to 1 part concentrated HCl
 KOH—saturated solution
 H₂O₂—16%
 NH₄OH—1 part concentrated NH₄OH to 1 part 16% H₂O₂; 5 parts concentrated NH₄OH to 1 part 3% H₂O₂
 For pyrrhotites and iron-rich sphalerite:
 1. 400 mg (NH₄)₂Cr₂O₇ dissolved in 25 cm³ of 15% HCl solution^b
 2. 50% HI
 For pyrite:
 Concentrated HNO₃ followed by brief concentrated HCl
 For monoclinic pyrrhotite: Magnetic colloid
 1. Dissolve 2 g FeCl₂·4 H₂O and 5.4g FeCl₃·6 H₂O in 300 cm³ of distilled water at 70°C.
 2. Dissolve 5g NaOH in 50 cm³ of distilled H₂O.
 3. Mix the two liquids; filter black precipitate and rinse with distilled water and 0.01N HCl. Place black precipitate in 500 cm³ 0.5% sodium oleate solution and boil for a few minutes; the resulting colloid should be stable for several months.

^aUse these chemicals with extreme caution. See note in text.

^bAlthough this etchant has commonly been used to distinguish between hexagonal and monoclinic pyrrhotite, which mineral has received the darker stain varies from one author to another.

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