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Application of Petrographic Techniques To Assess In Situ Leach Mining Potential

By Susan Brink, Bernhardt Saini-Eidukat,
Drummond Earley III, and Rolland Blake

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The Bureau of Mines is investigating the potential of in situ leach mining (ISLM) a variety of Cu, Mn, and Au-Ag ores. Geologic characterization of ore samples from laboratory leaching experiments is conducted both prior to and after leaching to assess the effects of leaching on the chemistry, mineralogy, and texture of the rocks.

The identification and chemical compositions of ore, gangue, and reaction products are determined by a combination of techniques such as reflected and transmitted light microscopy, X-ray diffraction, electron diffraction, electron microprobe analysis, and back-scattered electron imaging with a scanning electron microscope. The distribution of the desired metal in various mineral phases can be illustrated using elemental X-ray maps. The potential of reactive gangue minerals to rob Cu from leach solutions is investigated with the aid of transmission electron microscopy (TEM).

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

Å	angstrom	mm	millimeter
°C	degree Celsius	μm	micrometer
cm	centimeter	nm	nanometer
g/L	gram per liter	ppm	part per million
kPa	kilopascal	wt pct	weight percent
min	minute		

APPLICATION OF PETROGRAPHIC TECHNIQUES TO ASSESS IN SITU LEACH MINING POTENTIAL

By Susan Brink,[1] Bernhardt Saini-Eidukat,[1] Drummond Earley III,[1] and Rolland Blake[1]

ABSTRACT

The U.S. Bureau of Mines is investigating the potential of in situ leach mining a variety of Cu, Mn, and Au-Ag ores. Geologic characterization of ore samples from laboratory leaching experiments is conducted both prior to and after leaching to assess the effects of leaching on the chemistry, mineralogy, and texture of the rocks.

The identification and chemical compositions of ore, gangue, and reaction products are determined by a combination of techniques such as reflected and transmitted light microscopy, X-ray diffraction, electron diffraction, electron microprobe analysis, and backscattered electron imaging with scanning electron microscopy. The distribution of the desired metal in various mineral phases can be illustrated using elemental X-ray maps. The potential of reactive gangue minerals to rob Cu from leach solutions is investigated with the aid of transmission electron microscopy.

Porosity and permeability are measured to determine how effectively the host rock can transmit leach solution. Textural relationships between ore, gangue, secondary reaction products, and fluid channelways are determined by petrographic examination and are documented by optical photomicrographs and secondary electron imaging. Representative sample selection is described, and several epoxy impregnation and sample mounting techniques are discussed.

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INTRODUCTION

As part of its program to assure an adequate supply of metals and minerals from domestic sources, the U.S. Bureau of Mines is conducting research to evaluate the potential to mine Cu, Mn, and Au-Ag deposits in situ. In situ leach mining (ISLM) involves selectively leaching a target metal from a nonrubbled ore body by introducing and recovering the leach solution through boreholes (*1*).[2] The advantages of ISLM over conventional open pit and underground mining methods include less hazardous working conditions for miners; the ability to recover deep or low-grade ore, which would otherwise be economically unfeasible; and lower environmental impact (*1-2*).

Recent investigations conducted by the Bureau include leaching experiments on lengths of diamond-drill-core specimens of Cu oxide and Mn oxide ore using dilute sulfuric acid (H_2SO_4), 10 to 50 g/L (*3*), and dissolved sulfur dioxide (SO_2) solutions, respectively. The leaching experiments are designed so that the leaching fluids flow only along the natural permeable paths within the rock. Fluid chemistry derived from core-leaching experiments is combined with petrographic examination of both preleached and postleached ore to assess the effects of solution-rock interaction and to evaluate the potential of an ore body for ISLM (*4*).

In addition to core-leaching experiments, the Bureau is also conducting basic studies in mineral-fluid reactions. Specialized sample preparation techniques and analytical methods have been used to characterize mineral dissolution, absorption reactions, and reaction kinetics. For example, the Bureau is currently investigating the uptake of Cu by biotite under simulated leach-mining conditions (*5*).

Ore-control engineers and geologists at conventional mines use standard ore assays of powdered rock samples or column leach tests on aggregate material to determine the amount of recoverable target metal in a sampled area of an ore body. However, more detailed information is needed for ISLM because leach solution access to ore minerals is largely limited to natural permeability paths within the rock, unless artificial fractures are induced at depth by hydrofracing or other means. In ISLM, metal recovery is dependent upon the chemical reactivity of ore and gangue mineralogy and on the texture and distribution of these minerals within the host rock with respect to the permeability of the host rock both before leaching and as the rock is leached.

It is not only important to identify ore minerals and determine their location and accessibility to leach solutions within a rock, but also to determine the chemical composition of the ore minerals. The composition of both ore and reactive gangue minerals affects metal recovery and also the potential buildup of unwanted chemical components in the recycled lixiviant. For example, unleached chrysocolla from the Santa Cruz deposit, Pinal County, AZ, was found to contain variable amounts of Cu, as determined by microprobe analysis (table 1). A photomicrograph of a preleached Cu ore sample (fig. 1) shows compositionally different blue and yellow varieties of massive chrysocolla. This information can be used during drill-core logging to identify zones of low Cu chrysocolla within an ore deposit scheduled for ISLM, and thus provide better estimates of Cu recovery.

Figure 1.—Photomicrograph of fracture-hosted chrysocolla mineralization from Santa Cruz oxidized porphyry Cu deposit. Mottled yellow chrysocolla vein (*A*) and 0.14-mm wide blue chrysocolla veins (*B*) fill 1.4- to 2-mm-wide microbreccia fracture and are associated with quartz fragments (clear), goethite mineralization (dark orange to opaque) (*C*), and jarosite (mottled brown) (*D*).

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[2] Italic numbers in parentheses refer to items in the list of references at the end of this report.

Table 1.—Electron microprobe analysis of Santa Cruz chrysocolla

Variety	CuO	SiO_2	Other[1]	Total[2]	H_2O[3]	Cu/Si
Yellow . .	33.4	45.1	4.2	72.2	22.8	1.08
Blue	22.8	25.0	2.3	40.1	59.9	.97

[1] Consists mostly of Al_2O_3, Fe_2O_3, CaO, and MgO in order of decreasing abundance.

[2] Summation of all oxide components that were analyzed.

[3] By difference. Microporous nature of chrysocolla may in part account for the low total weight percent of oxide.

Petrographic and chemical characterization of a potential ISLM deposit is dependent on representative sample selection and preparation. In laboratory studies, preleached thin sections are made from the end sections of the drill core that is to be leached. Following leaching, thin sections are also made from a variety of horizons within the leached core. Since it is not possible to petrographically analyze the same portion of rock both before and after leaching, the observations and data collected from preleached specimens are averaged for the length of drill core studied.

PETROGRAPHIC AND ANALYTICAL TOOLS FOR GEOLOGIC CHARACTERIZATION

ELECTRON BEAM TECHNIQUES

Scanning electron microscopy (SEM) and the electron microprobe allow examination of samples at high magnifications. Both instruments rely on observation of the products produced by the interaction of a focused electron beam with the sample. These interaction products include secondary electrons, backscattered electrons, and characteristic X-rays, which provide information on the sample's composition and topography (*6*).

Scanning Electron Microscopy Imaging

Two common types of SEM imaging can be used to gain information on the composition and texture of the sample: backscattered electron imaging (BEI) and secondary electron imaging (SEI). In both modes, the image is formed by rastering the electron beam across the sample, detecting any electrons that are emitted from the sample, and displaying detected electrons on a video monitor, which is rastered in sync with the beam. The difference between the two types of imaging is the type of electron that is detected.

BEI is best used to distinguish compositional differences in a sample. The type of electrons detected in BEI are high-energy electrons that have elastically scattered from the sample material. The average intensity of backscattered electrons from a particular mineral is proportional to the mineral's mean atomic weight. Thus, a BEI image of an Au grain, in a sample from the Cyprus Copperstone Deposit near Quartzite, AZ, appears much brighter than most silicate images and its location is easily discerned in a quartz-filled fracture (fig. 2) or when disseminated in fine-grained gangue minerals. BEI is being used to determine if sulfur dioxide can selectively dissolve Mn from an Mn ore from the Algoma-Zeno deposit, Crow Wing County, MN, containing an intimate mixture of pyrolusite (MnO_2), hematite, quartz, and other gangue minerals. Figure 3 is a high-magnification image of the Mn ore and shows the textural relationship between hematite, quartz, and pyrolusite across a leach front. The lower part of the image shows that Mn has been selectively removed, leaving quartz and hematite behind. Thus, the use of BEI to distinguish ore and gangue minerals at high magnifications is important in evaluating fine-grained or optically indistinct ore for ISLM.

SEI detects electrons produced as a result of interactions between high-energy beam electrons and weakly bound conductive electrons in the sample. This interaction produces low-energy electrons, which carry little information on the composition of the sample, but can provide information on the topography of the sample. For example, the location and texture of a porous Au grain from the Phoenix Mine, Idaho Springs, CO, lying in the pore space between several pyrite grains within a fracture (fig. 4) indicates that this type of Au mineralization may be accessible to leach solutions. Gold mineralization, which is encapsulated by a pyrite or quartz grain rather than occurring between gangue mineral grains, is not amenable to ISLM. Therefore, by discerning the texture of grain boundaries, SEI is used to determine if ores with certain textures can be effectively reached by leach solutions during ISLM. The topographical nature of natural porosity, as well as induced porosity and permeability of an ore sample, due to mineral dissolution during leaching, is evaluated using SEI. An SEI (fig. 5) of the same leached sample of Mn ore (fig. 3) shows the increased porosity due to leaching of pyrolusite.

X-Ray Elemental Maps

X-ray spectrometry is used in conjunction with image-processing software to create maps of the chemical distribution of elements in a specimen. An X-ray map is a composite of spot analyses (on the order of 1 μm in diameter) made across a specimen in a grid pattern. Each selected element being analyzed is assigned a color code. The concentration of an element across a sample,

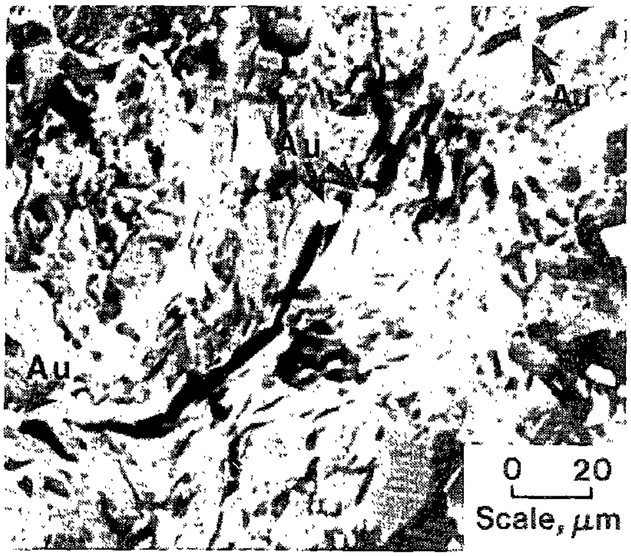

Figure 2.—BEI showing four Au grains in open fracture of schist from Cyprus Copperstone Mine. [From Blake and Earley (*11*)].

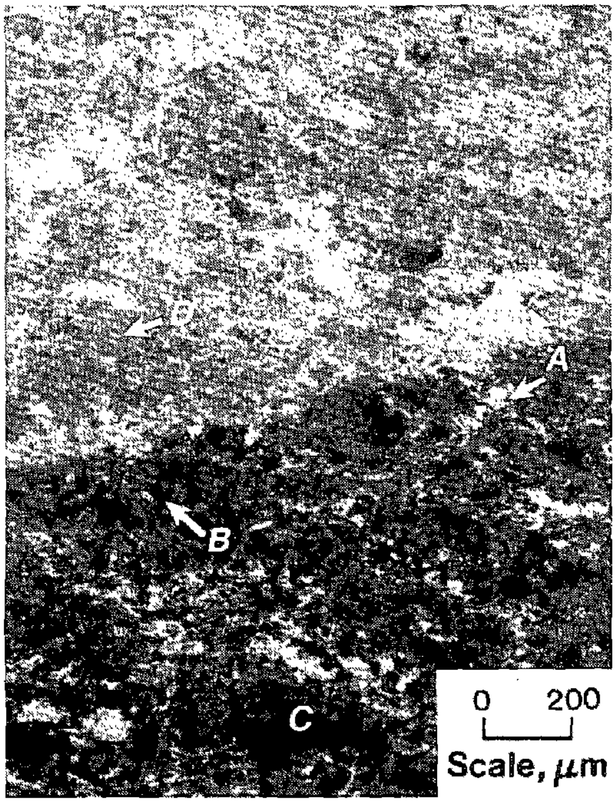

Figure 3.—BEI of Mn ore from Algoma-Zeno deposit showing textural relationship between hematite (*A*), quartz (*B*), and pyrolusite (*D*) across leach front. (*C* indicates hole.) Lower half of image shows that Mn has been selectively removed, leaving quartz (medium dark gray) and hematite (light gray) unleached.

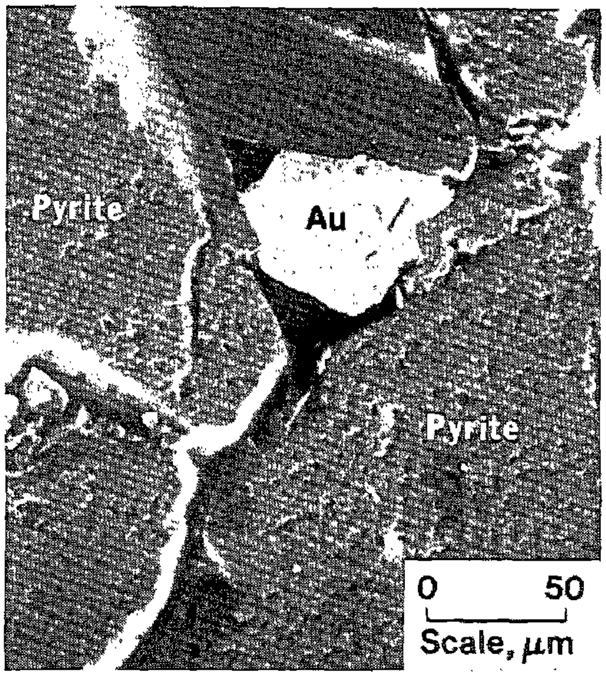

Figure 4.—SEI showing Au between pyrite grains from Phoenix Mine.

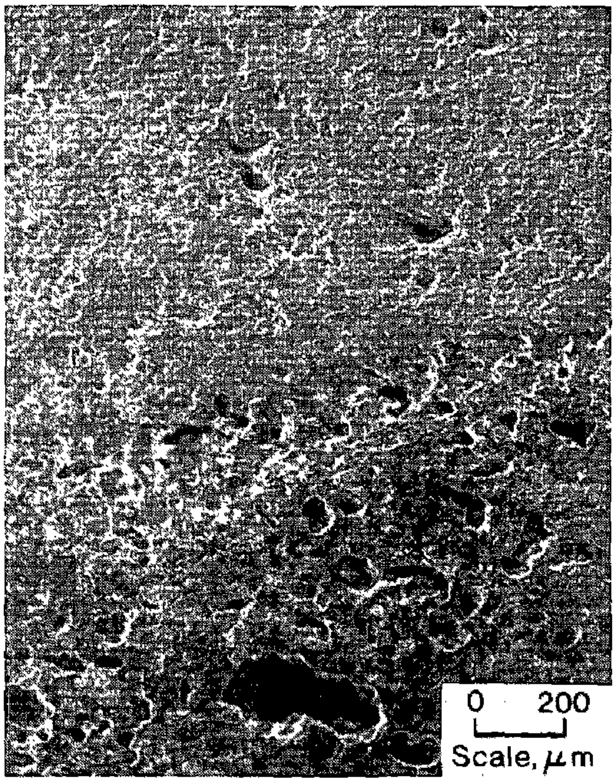

Figure 5.—SEI of Mn ore from Algoma-Zeno deposit (shown in figure 3) showing resulting porous topography after leaching Mn from lower half of mixed pyrolusite-hematite ore.

therefore, is proportional to the intensity of the assigned color. The software can be used to enhance color intensities and to overlay patterns of the various elemental maps on a BEI or SEI. These methods allow the distribution of certain minerals to be compositionally identified and compared with topographical features of the specimen.

X-ray maps can be used to graphically illustrate the distribution of fine-grained ore minerals in a rock matrix and to determine compositional zoning and the location of impurities within an ore mineral. For example, the location of Mn, Fe, and Si concentrations in a sample from the Prince mine, Lincoln County, NV, is shown in figure 6. Combined with SEM images, these maps can provide insight into the processes by which ore minerals are leached or why leaching is being hindered. For example, subtle sources of metals, such as Cu-bearing biotite from the Santa Cruz deposit, Pinal County, AZ (fig. 7), have been investigated using X-ray maps. Figure 7 shows the heterogeneous distribution of Cu (red) within a biotite grain. A quantitative compositional profile (fig. 8), determined by microprobe analysis, shows that this biotite contains as much as 1 to 2 wt pct CuO which is concentrated along tabular zones that are parallel to the basal cleavage. The Cu that resides in biotite is somewhat resistant to leaching as the biotite crystal itself must be consumed in order to recover its Cu content (7). Thus, the potential recovery by ISLM of metals disseminated in other mineral phases can be evaluated using X-ray maps and microprobe analysis of preleached and postleached specimens.

The solubility of gangue minerals and possible formation of byproducts or precipitates resulting from leach fluid reactions with the rock strongly influence the permeability of the rock as well as the overall leach fluid chemistry by increasing acid consumption and the concentration of unwanted ions. For example, a compositional X-ray map (fig. 9) shows the preferential distribution of alunite ($KAl_3(SO_4)_2(OH)_6$) crystals (yellow) in the sericite-altered areas (light blue) of potassium feldspar ($KAlSi_3O_8$) grains (pink) from the Santa Cruz oxidized porphyry Cu deposit. When this compositional map is compared with the BEI of the same area as shown in fig. 9, the fractures containing leach fluid channelways (black) and amorphous

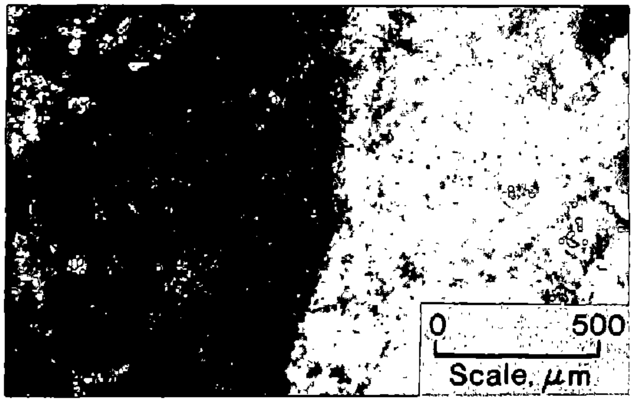

Figure 6.—X-ray compositional map showing location of Mn (pink), Fe (dark red), and Si (green) across leach front in sample from Prince mine. Manganese has been dissolved from coronadite mineral structure leaving Pb (white), which was not leached. Black dots are voids. Fracture closely corresponds to leaching front.

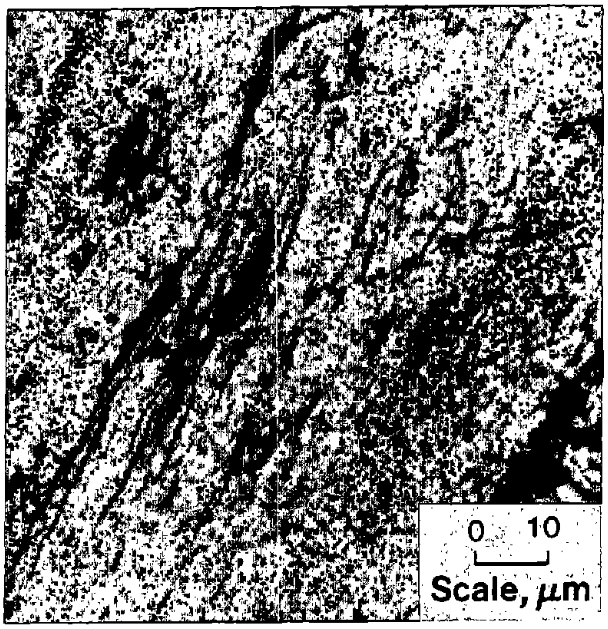

Figure 7.—X-ray map overlying BEI of biotite grain from Santa Cruz deposit showing distribution of Cu (red) within grain.

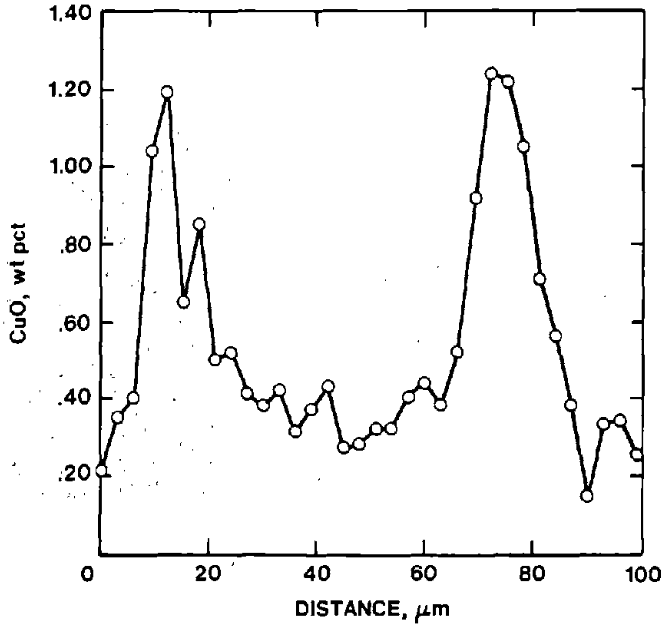

Figure 8.—Compositional profile across biotite grain from Santa Cruz deposit (shown in figure 7) quantitatively showing zones of high Cu concentration.

silica (SiO_2) residue left behind after leaching chrysocolla (medium gray) can be seen (fig. 10). It was hypothesized that alunite precipitation or dissolution was controlling the Al concentrations in the leach fluid during leaching experiments (*7*). However, the occurrence of euhedral alunite in preleached as well as postleached samples indicates that it is not a precipitate formed during the leaching experiment. The euhedral crystals that occur within the leached fracture do not appear to be noticeably dissolved by the leach fluid. Thus, X-ray maps are useful to evaluating the ISLM potential of ores by illustrating the occurrence and distribution of metals and ore-gangue minerals within host mineral phases and aggregates of alteration products both before and after experimental leaching of the ore.

Electron Microprobe Analysis

The electron microprobe is used to determine the qualitative or quantitative chemical composition of individual mineral grains on a microscopic scale. The bombardment of a sample with a beam of electrons produces X-rays with energies characteristic of the elements present in the sample. The number of each characteristic X-ray is counted for a given sample and compared with the number emitted when a mineral of known composition is under the beam. In the study of ISLM, the electron microprobe is used to determine the composition of preleached and postleached ore and gangue minerals and to quantify the effects of leach solutions on these minerals. The presence and composition of secondary reaction products can also be investigated.

The utility of the microprobe can be illustrated with an example from a leaching test of a Prince mine, Lincoln County, NV, Mn ore with aqueous sulfur dioxide. A preleach chemical analysis showed that the ore contained about 3 wt pct Pb, and although a large percentage of the Mn was removed, very little of the Pb was measured in the effluent leach solution. Since common Pb minerals were not observed in preleached samples by optical microscopy nor identified by X-ray diffraction (XRD) analysis, the Mn oxides were chemically analyzed using the electron microprobe and found to contain up to 30 wt pct PbO (lead oxide). BEI of the postleached ore showed the presence of highly reflective areas, which, when analyzed with the microprobe, were shown to consist almost entirely of Pb (fig. 6). Thus, the difference in the experimental solubility of Mn and Pb shown by high-magnification SEM images suggests that sulfur dioxide leaching of Pb-containing Mn oxides probably will not mobilize Pb during ISLM, a result that can be looked on as favorable from an environmental perspective even if negative from a Pb-recovery perspective.

The electron microprobe can accurately determine the major and minor chemical constituents of minerals, but determination of trace element content (less than about 500 ppm) requires special consideration (*8*) or the use of more sensitive instruments such as the ion or proton microprobe (*9*).

Transmission Electron Microscopy

Transmission electron microscopy (TEM) is similar to transmitted light microscopy except that an electron beam rather than light passes through ultra-thin specimens (<100 to 200 nm) during analysis and the sample image is

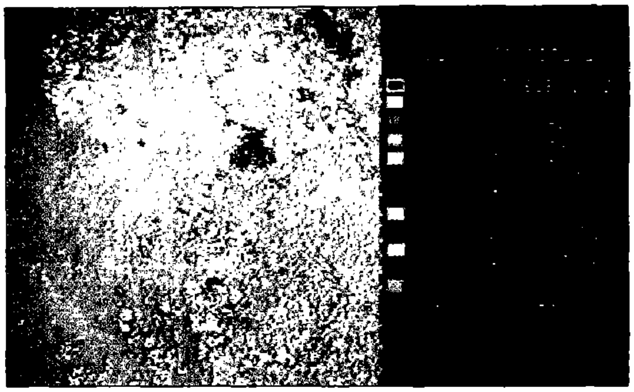

Figure 9.—Computer monitor screen displaying X-ray compositional map showing distribution of alunite crystals (yellow-green) within sericite-altered (light blue) potassium feldspar (pink) grains from Santa Cruz oxidized porphyry Cu deposit. Dark blue areas are epoxy-filled holes in sample. Width of image is 500 *μ*m long.

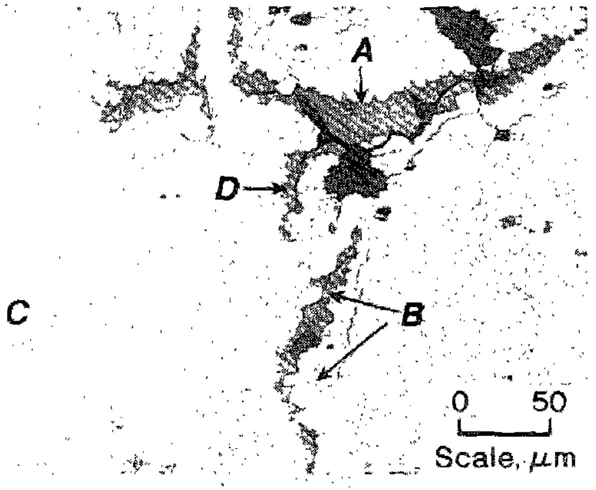

Figure 10.—BEI of same area as that shown in figure 9, showing fractures that contain amorphous silica residue (gray) (*A*) left after leaching chrysocolla, euhedral alunite crystals (white) (*B*), unaltered potassium feldspar (white fields) (*C*), leach fluid channelways (black) (*D*), and sericite-altered potassium feldspar (light gray fields).

generated when the diffracted electrons hit a fluorescent screen. The resolution of TEM is much greater than SEM, and features as small in size as 10 Å can be seen. Qualitative chemical analysis and electron diffraction, which is similar to XRD, is also possible using the transmission electron microscope.

An example where TEM was used for ISLM research is the Bureau's Cu-biotite investigation (*5*). In this investigation, flakes of biotite were reacted with acidic copper sulfate ($CuSO_4 \cdot 5H_2O$) solutions in flowthrough reaction vessels. SEM-electron microprobe analysis of the reaction products showed that trioctahedral micas can sorb up to 1 to 2 wt pct Cu in less than 1 month of reaction time (*5*). The technique of TEM was used to document the mode of Cu occurrence in the reacted biotites. The X-ray map shown in figure 11 shows that Cu has migrated 100 to 200 µm into the crystal after only 2 weeks reaction time. The Cu contents (shown in red) are as high as 1 wt pct. TEM analysis shows that submicroscopic native Cu inclusions, 10 to 200 nm in diameter (figs. 12-13), occur in the Cu-bearing areas of reacted micas. Copper crystallites are deposited preferentially along expanded interlayer sites of the biotite crystal (fig. 14) as a result of biotite alteration in acidic copper sulfate solutions.

The Cu distributions observed in this experimental study are similar to those observed in natural Cu-bearing biotites. The fact that biotite can obtain anomalous Cu values during weathering implies that biotite could potentially rob Cu-bearing solutions of their metal value during ISLM. Since biotite is a common and sometimes abundant mineral in the host rock of Cu deposits, it may be necessary to pacify biotite in order to avoid lowered Cu recoveries during ISLM.

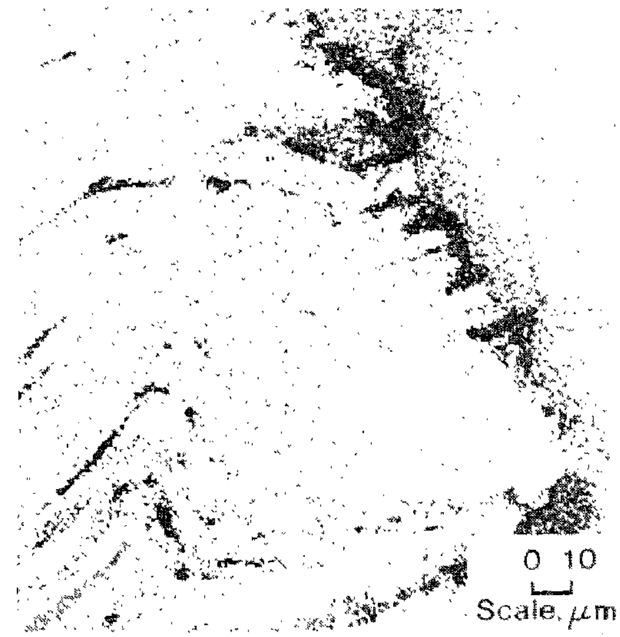

Figure 11.—X-ray map superimposed on BEI of biotite flake reacted with acidic copper sulfate solution for 2 weeks at low temperatures (25° C) and pressures (atmospheric). Image shows distribution of Cu (black dots) and S (medium gray dots). Medium gray fields indicate where Cu and S occur together.

CATHODOLUMINESCENT MICROSCOPY

Cathodoluminescent (CL) microscopy can be used to detect and distinguish minerals that have very similar optical properties in transmitted, polarized, and reflected light. CL microscopy can also aid in accurate modal analyses of fine-grained or otherwise indistinguishable ore mineralization that occurs in intimate ore-gangue mixtures. Ore-gangue textures important to ISLM and compositional zoning of certain ore and gangue minerals that cannot be observed by other petrographic techniques can greatly aid the interpretation of the potential for success of ISLM on a particular ore. In combination with the electron microprobe, CL microscopy can also be used to detect the presence of contaminating trace metals in ore samples such as Pb in an Mn ore.

Figure 12.—High-resolution TEM showing native Cu inclusions (dark ellipsoids) in biotite from Santa Cruz deposit (shown in figure 1). Image is oriented such that the view is along cleavage planes of mica crystal. Inclusions are far too small to be seen under the petrographic microscope or SEM.

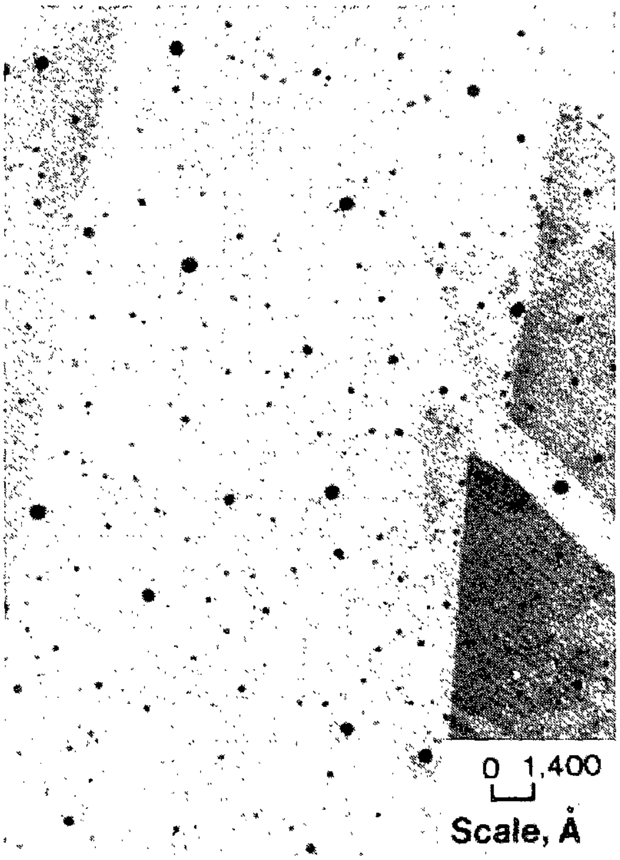

Figure 13.—TEM image of native Cu inclusions in biotite. View is down c-axis of biotite crystal (i.e., perpendicular to cleavage). Image illustrates discoidal shape of inclusions.

Cathodoluminescence is the emission of visible light from a mineral when it is bombarded by an electron beam. This visible radiation is due to electron transitions and occurs when energized (excited) d-orbital electrons of an atom fall back to a lower orbital level. The instrument most often used for this type of petrography consists of a cathode gun, which provides a diffuse electron beam; a vacuum pump; and a vacuum chamber, which fits on the stage of a conventional petrographic microscope. The vacuum sample chamber accepts uncovered, standard, polished thin sections 30 μm thick and polished thin sections 10 to 12 μm thick. The specimen surface does not need to be carbon coated, but should be free from mounting media, stain, and oil since the electron beam only penetrates a short distance. A thermal-resistant epoxy should be used to mount the thin sections in order to avoid vaporization at high voltages. CL images formed on electron microscopes can be combined with other types of images to provide compositional information such as trace element zoning in minerals.

Since the cathodoluminescence of most minerals is a function of their trace element content, it is not generally possible to predict in advance whether a mineral will luminesce or what color of light will be emitted. Activator elements within a mineral are centers of easy activation, which produce visible light and include Mn^{2+}, U, Pb^{2+}, Cu^{2+}, Zn^{2+}, Ag^{+}, Mg^{2+}, and several rare-earth elements (Eu^{2+} and Tb^{2+}). "Quencher" elements absorb excitation energy, thereby quenching luminescent properties in a mineral. The most common quenchers are Fe^{2+}, Ni^{2+}, and Co^{2+}. Combinations of activator elements will produce intermediate colors.

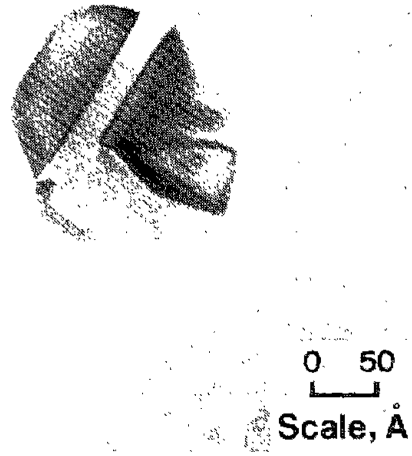

Figure 14.—High-resolution TEM showing closeup of Cu inclusion in biotite. Crystallite displays twinning that is characteristic of native Cu.

Luminescence of minerals in a thin section can be easily compared with features shown in transmitted normal and polarized light. Once the CL characteristics of specific minerals within an ore suite have been well established, CL microscopy can be an excellent timesaving analytical tool to identify and distinguish ore and gangue mineralogy, determine modal analysis, and characterize textural relationships important to ISLM.

X-RAY DIFFRACTION ANALYSIS

In contrast to chemical analysis, which only provides information on the chemical content of a sample, XRD analysis is used to determine the exact identity of minerals by their crystal structures. XRD can be best used in the identification of minerals that appear similar under the optical microscope, such as some of the Mn oxides, or which are mixtures of extremely fine-grained minerals, such as clays.

XRD analysis consists of reflecting a small beam of monochromatic X-rays off a powdered sample at continuously varying angles. The unique combination of spacing and angles between atomic planes in a mineral provides a "fingerprint" pattern, which is compared with patterns of known minerals. XRD analysis is a relatively insensitive technique for the determination of relative amounts of minerals in a sample, and thus point counting should be used for modal analysis.

POROSITY AND PERMEABILITY MEASUREMENTS

Hydrologic models of ore deposits are constrained by field measurements of permeability that typically involve well-to-well tracer tests and pressure measurements. These techniques are well suited for the study of large-scale hydrologic processes. Demonstration of pervasive fluid flow in the deposit on the basis of hydrologic modeling does not, however, mean that the target minerals are necessarily contacted by the lixiviant. Texture and spacial relationships will ultimately determine the accessibility of leach solution to ore minerals. The small scale or "matrix" permeability and porosity of rock samples can be measured quantitatively and compared with textural information to verify that solutions can pass through observed microcracks and other voids.

At the Bureau, matrix porosity and permeability of rock specimens are quantitatively measured using a pycnometer and permeameter, respectively. These instruments were used on carefully cored and trimmed cylinders of rock, which are approximately 2 cm in diameter and 4 cm in length.

Matrix porosity is determined indirectly using a two-step process:

1. The "geometric volume" of a cylinder is measured using calipers or Hg immersion. In this measurement, the interior void space of the sample is included in the measured volume.
2. In the next step, a gas such as He is forced into the pores of the sample in the sealed chamber of a porosimeter at known pressure (P) and chamber volume (V). By varying the chamber volume and measuring the change in pressure, the sample's matrix volume can be calculated using an expression that can be derived from Boyle's law ($P_1V_1 = P_2V_2$). The volume that is measured in this step does not include the pore space of the sample. Porosity is then calculated by subtracting the sample's, matrix volume from the geometric volume.

Matrix permeability is measured by forcing water through the cylindrical rock samples at a constant pressure. Axial flow is induced by jacketing the sample in a plastic doughnut-shaped gasket that is inserted into the metal sleeve of the pressurizing apparatus. Water flows from the injection side of the core under pressure through the core and is collected at a basal outlet. The volume of water collected and the experimental run time are recorded and used to calculate the permeability (k) using Darcy's law:

$$k = (U \cdot V \cdot L)/(A \cdot P \cdot t),$$

where U = viscosity of fluid,

V = volume of fluid that passes through specimen in time, t, at fixed pressure, P,

and A = cross-sectional area of specimen that is normal to fluid path.

Matrix porosity and permeability measurements would seem to be especially important for assessing the feasibility of applying ISLM to precious metals deposits. Because Au, Ag, and Pt occur in much lower concentration and in much smaller grain sizes than most other styles of mineralization, precious metals have a much greater potential for "hiding" in impermeable zones. Quantitative measurement of matrix porosity and permeability helps in accessibility assessments when the observed ore occurrences are along microfractures and grain boundaries. The baseline porosity of low-permeability rocks is also important information if permeability enhancement techniques such as hydraulic dilation are employed (*10*).

TRANSMITTED AND REFLECTED LIGHT MICROSCOPY

A well-known and commonly used geologic tool, the light optical microscope can magnify images approximately 10 to 1,500 times. Polished ore minerals are identified by their color, crystal habit, behavior in normal and in polarized light, etc. Microscopically observable features that are of interest for ISLM research include the association of ore minerals to microfractures; fracture frequency, width, length, and interconnectivity; and ore and gangue mineral size, grain boundary relationships, and effective surface area. Most of these features can be photographed through the microscope to preserve a record for future examination and to enhance a written description of their occurrence. Light microscopy is essential to understanding the ore and gangue minerals of a deposit at the microscopic level, which is the scale at which the leach fluids and minerals interact.

Example 1: Fracture-Hosted Mineralization

Experimental results have shown that most Cu-mineralization (atacamite and chrysocolla) occurring as veins and filling microbreccia fractures is easily accessed by leach solutions (*4*, *7*). Amorphous silica, which remains after leaching chrysocolla with sulfuric acid solutions, generally contains no Cu, but may sorb some components (SO_4^-, H^+, Cu^{2+}) from pregnant leach solutions along leach fluid channelways, as determined by microprobe analysis.

A leached sample of a chrysocolla-mineralized microbreccia from the Santa Cruz deposit, Pinal County, AZ, shows residual amorphous silica rimming some of the rock fragments after Cu has been successfully removed (fig. 15). The largest width of the microbreccia vein shown in the photomicrograph is 4 mm. Therefore, microscopy combined with microprobe analysis is used to determine that Cu is effectively leached from fracture and breccia-hosted chrysocolla mineralization and that similarly mineralized chrysocolla ores will effectively transmit leach fluid through residual amorphous silica during ISLM. Another example of fracture-hosted ore mineralization is four Au grains located in an open fracture of Au-Ag ore (fig. 2).

Figure 15.—Photomicrograph of leached microbreccia cemented by chrysocolla. *A* is a quartz grain; *B* is a potassium feldspar grain. Breccia fragments are rimmed by amorphous silica residue (*C*) that remains after leaching Cu from Santa Cruz deposit.

Example 2: Matrix-Hosted Mineralization

Clay-altered plagioclase grains, from a granite within an oxidized porphyry Cu deposit in Santa Cruz, Pinal County, AZ, consist of a complex, intimate mixture of many different alteration minerals (fig. 16). According to XRD data, microprobe analysis, and petrographic observations, these minerals include kaolinite, illite-hydromica, chrysocolla, and Fe oxides (mainly as goethite). Additionally, some Cu may be sorbed onto some of these minerals. The success of leaching disseminated Cu from these intimate mixtures of alteration products is assessed by petrographic examination using reflected and transmitted light microscopy, microprobe analysis profiles across relict feldspar grains, and X-ray maps for Cu, Fe, Si, Al, Na, K, Mg, and Ca of Cu-enriched, altered feldspar grains from preleached and postleached samples. Results verify that Cu is successfully removed when the altered grains have been exposed to an adequate amount of leach solution. Thus, successful

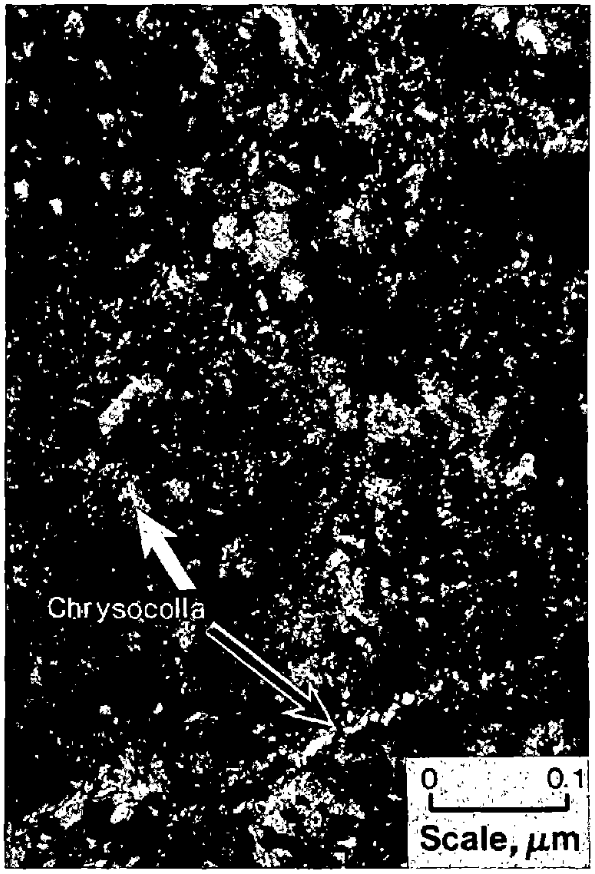

Figure 16.—Photomicrograph of chrysocolla intimately mixed with clay minerals from altered plagioclase from Santa Cruz deposit. Using cross-polarized light, chrysocolla appears as mottled black and light blue patches and veinlets; illite paths are bright orange-yellow; and aggregates of clay minerals occur in green areas.

leaching of disseminated Cu-mineralization in clay-altered plagioclase grains is dependent upon the proximity of grains to a leach fluid channelway; i.e., permeable fracture or vein.

Example 3: Encapsulation of Ore Minerals by Gangue

In granitic rocks from the Santa Cruz deposit, Pinal County, AZ, studied for the Cu ISLM project, goethite often lines the walls of Cu-mineralized fractures and also encapsulates atacamite and chrysocolla (fig. 17). Petrographic observations of preleached and postleached samples indicate that goethite, which physically encapsulates chrysocolla or atacamite (fig. 18), generally does not prevent leach solution access (*7*). For example, figure 18 shows that Cu has been leached from the center of a goethite atoll structure, which originally had been filled with chrysocolla. The previous presence of chrysocolla is indicated by an amorphous silica residue that is now left in the center of the atoll. However, microprobe analysis of goethite shows that it can contain small, but significant amounts of Cu in its crystal structure.

Figure 17.—Photomicrograph showing goethite lining and occluding patches of chrysocolla in step fracture in potassium feldspar grain from Santa Cruz deposit. Average width of fracture is 0.2 mm.

Small amounts of quartz-encapsulated copper sulfides are present in granitic samples from the leached zone of an oxidized porphyry Cu deposit. These sulfide ore minerals were effectively sealed off from natural oxidizing leach solutions, as well as from experimental leach solutions. A photomicrograph of a precious metals-bearing ore from the Coeur-Rochester Au mine, near Lovelock, NV, shows a small grain of Au encapsulated by pyrite (fig. 19), which itself was later encapsulated by quartz. Because of its location within the rock, this grain of Au will not be reached by leach solutions during ISLM (*11*). Mineralization that is predominately encapsulated by unreactive gangue minerals will not be viable ores for ISLM.

Point-Counting Method of Modal Analysis

Standard point-counting methods of polished thin sections using an automatic point counter may be used to

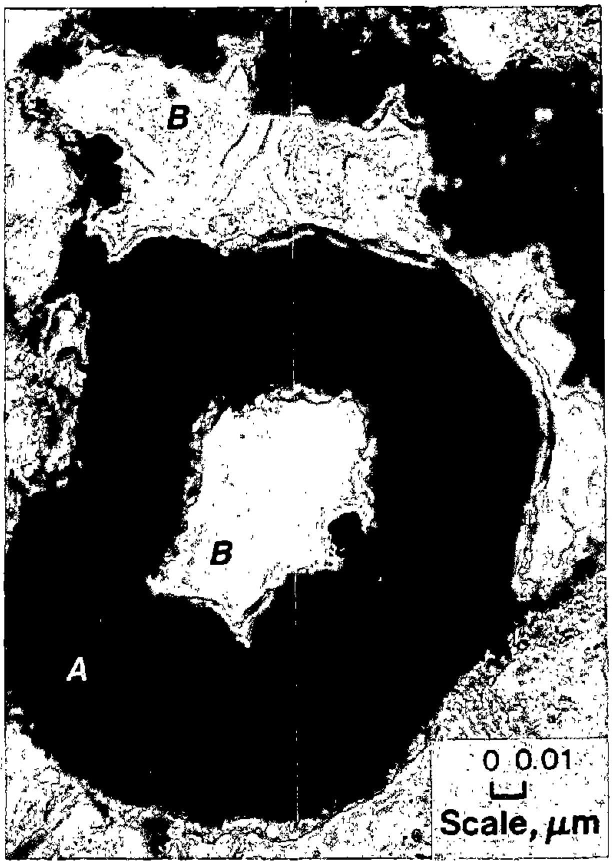

Figure 18.—Photomicrograph of successfully leached chrysocolla encapsulated in goethitic atoll structure (0.16 mm across) (*A*). Remnants of amorphous silica (clear, with slight relief) (*B*) can be seen in voids that were once filled with chrysocolla.

determine the mineral mode of the rock. Variable translation distances can be chosen between points in the east-west direction and counting lines in the north-south direction. Optimally, a counting line should pass through at least three crystals of any one mineral species (*12*). Also, the area to be counted for a thin section should contain five or more of the largest phenocrysts of any one mineral. Typically, 1,200 to 1,600 points are counted for each thin section, and the average of all thin sections is used to determine the mineral modes of the rock.

Automated point-counting methods using the electron microprobe with computer diagnostics or diagnostic computer-video imaging are other methods of acquiring modal data. The resolution of these methods, however, is not as good as that of a trained microscopist, especially for altered rocks.

SAMPLING STRATEGY AND SAMPLE ORIENTATION

Representative Thin-Section Orientations

Thin-section modal analysis is most representative of the true mode of a rock when the specimen is equigranular, nonporphyritic, and lacks coarse layering. Most natural rocks do not meet these requirements, especially those that contain veined mineralization. Therefore, a larger, more representative area of an inhomogeneous rock is obtained by averaging the modal analyses of many thin sections taken from a length of drill core. It is good petrographic practice to carefully observe the rock fabric and structural features of a rock specimen before cutting it into thin-section billets. A series of orthogonal thin-section orientations should be used to best represent the inhomogeneity of the rock. Figure 20 shows an example of how thin sections were made from a 30-cm length of drill core. Section I of the drill core was cut off before leaching the remainder of the core. It was used to make horizontal (relative to the core axis) thin sections of unleached rock. These thin sections were labeled A, B, C, and D. After leaching, the leached portion of drill core was cut into sections II, III, IV, and V. By this method, a mineral-bearing fracture, which runs through both sections I and II of the drill core, may be analyzed both before and after leaching, although not at the same location along the fracture. Thin sections that are oriented vertically were made from sections II and IV and are labeled E, F, G, and H, and M, N, O, and P, respectively. Thin sections that were oriented horizontally to the core axis were made from the remaining leached sections.

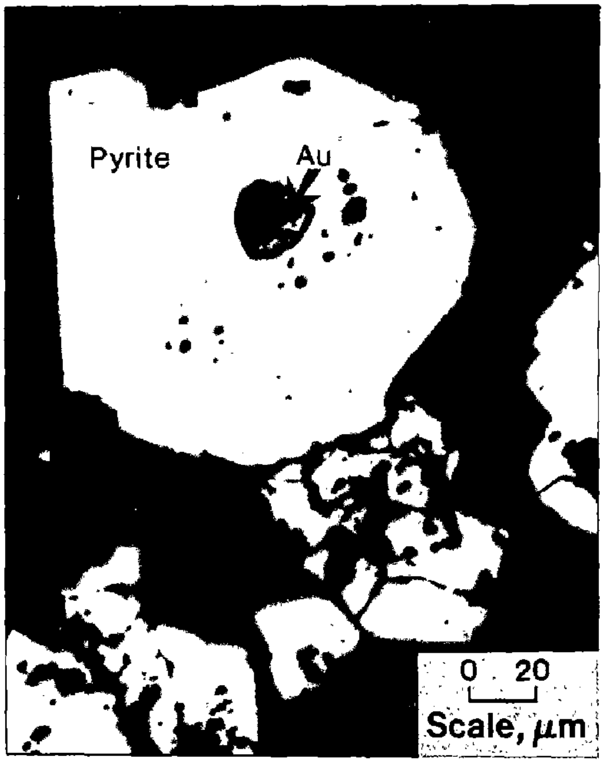

Figure 19.—Photomicrograph of Au encapsulated in pyrite occurring in quartz vein in Coeur-Rochester Au mine. Gold grain is 20 μm in diameter.

Preparation of Rock Samples

It is important to avoid contamination and grain plucking during preparation of ore samples, especially those that have very low concentrations of the target metal; i.e.,

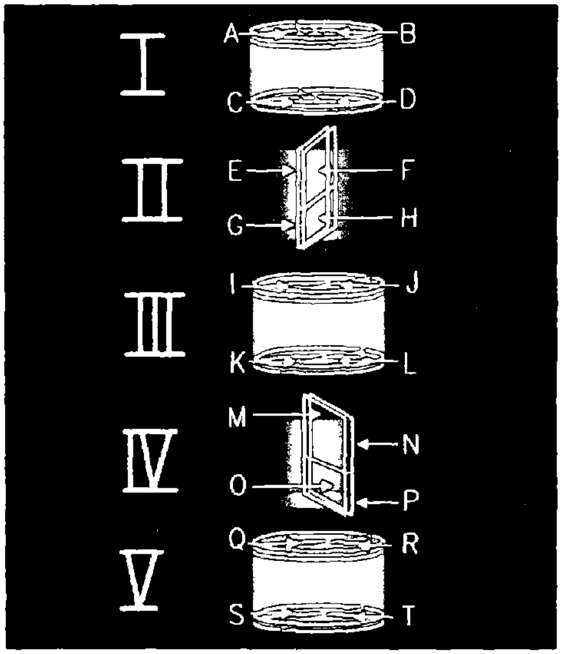

Figure 20.—Schematic of sampling scheme for leached drill core.

precious metals. The sawn surfaces of both preleached and postleached rock samples are usually damaged and show evidence of grain plucking. Therefore, it may be necessary to first impregnate the sample with low-viscosity epoxy, prior to thin-section preparation, by one of the methods described below. When the epoxy has cured, the billet for the thin section can be cut and ground down past the disturbed layer with a diamond wheel before proceeding with the usual grinding and polishing stages used in thin-section preparation. The thickness of the disturbed layer will depend on the grain size and friability of the rock. Leached core is often so friable that it will disintegrate if sawn. It is important to maintain the integrity of the sample in order to observe and quantify the effects of leaching on the texture and mineralogy of the rock.

Epoxy Impregnation

Two methods of epoxy impregnation were developed to strengthen rock samples before thin-section preparation. The first step is to cut the length of leached drill core into cylindrical sections approximately 6 cm in length (see figure 20). These rock cylinders are impregnated with a clear, low-viscosity epoxy by drawing a vacuum through the bottom of the sample and pooling the epoxy on the top surface of the sample, which is open to the atmosphere. This is accomplished by inserting the sample into a rubber jacket that is also attached to a concave clear acrylic disk that has been fitted to a vacuum hose. The rubber jacket is secured around the circumference of the sample and the disk by hose clamps. As epoxy is drawn through the leached fractures of the sample, the pool of epoxy on top of the sample is continually replenished until epoxy can be seen running out of the bottom of the sample. The section of drill core is then allowed to cure (1 to 3 days) before being cut into billets for thin sections.

A different epoxy impregnation method is used for slab-shaped billets and cylindrical core sections with low permeability. In this method, the sample is placed in a bell jar vacuum and low-viscosity epoxy is pooled on the rock surfaces. The denser epoxy will displace air from the pores in the surface of the sample. Over a period of 15 min, the vacuum is released three or four times. This procedure breaks bubbles that may have formed on the sample's surface and redistributes the epoxy. Each time the vacuum is released, air attempts to reenter the sample, forcing the epoxy further into the sample. Unfortunately, penetration depth is very shallow by this method, ranging only from 1 to 5 mm. In an effort to increase penetration depth by further reducing the epoxy's viscosity, the sample can be heated (60° C) prior to impregnation. Some epoxies, however, may lose some volatility with heating and may not cure properly. The typical vacuum gauge pressure used in both of these methods is -70 kPa.

Biotite Sample Mounting

The solid products from the biotite experiments are fragile and require special sample preparation before optical, electron microprobe, and TEM analysis. For analysis parallel to the c-axis, thin biotite flakes are mounted on glass slides with epoxy, or TEM sample grids are mounted on the flake itself and peeled off as very thin foils. The smooth cleavage surfaces do not need polishing prior to electron microprobe analysis. For analyzing biotite specimen cross sections that are normal to the c-axis, the mica flakes were first treated with a silane bonding agent and then embedded in a 3M Concise[3] ceramic amalgam composed of quartz and acrylic. The silane forms a more durable bond with the ceramic than the biotite cleavage surface alone. The ceramic blocks are then cut with a diamond gem saw across the cleavage planes of the micas. One face of the ceramic-mica billet is ground on glass with alumina (Al_2O_3) grits and then mounted face down on a glass slide with an Aremco Crystalbond 509 thermoplastic cement. When the section has reached a thickness of approximately 100 μm during the thin-sectioning process, best results are obtained when the rock section is released from the glass slide, flipped over and reglued, and then thinned to 30 μm. Immersing the specimen in ice water during grinding also helps maintain the integrity of the embedded mica flake because the cement is heat sensitive. If ordinary epoxy is used, the embedded biotite mount is usually plucked out well before it is thinned to 30 μm. The quartz in the embedding material makes it easy to determine when the thin section is 30 μm using the birefringence of quartz. When the section is 30 μm thick, it is ready for optical and electron microprobe analysis. Additional steps are required for TEM analysis, but the ceramic embedding preparation procedure makes TEM sample preparation relatively easy. Thermoplastic cement is used so that a TEM mount (a circular silver grid 3 mm in diameter) can be epoxied onto the specimen after preparing a standard 30-μm microscope thin section. The TEM mount is then removed by scoring the matrix around the grid with a diamond etching tool, heating the sample, and lifting the specimen with attached grid off of the glass thin section. The quartz-acrylic matrix holds up well under the electron beam, in contrast to epoxy, which must be removed from the sample prior to the final thinning stages of TEM sample preparation in order to avoid contamination of the vacuum chamber of the transmission electron microscope.

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[3] Reference to specific products does not imply endorsement by the U.S. Bureau of Mines.

CONCLUSIONS

Evaluation of an ore's potential for successful ISLM is dependent on understanding host-rock mineralogy, texture, and hydrology. Proper sampling and sample preparation, examination by light and electron microscopy, mineral analysis by electron microprobe and XRD, and determination of hydrologic parameters must be combined with whole-rock chemical analysis to estimate the potential for economic recovery of the target mineral.

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