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ELECTROPOLISHING OF PYRITE

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Although a considerable body of literature exists on the electropolishing of metals (see references in Faust 1948 and Tegart 1959) apparently no investigations have been made on sulfides. The technique will probably prove to be a useful one for mineralogists, as crystals or aggregates with either naturally-occurring faces or artificially-produced surfaces may be used. With the proper choice of reagents, crystalline materials can be made to polish essentially uniformly on all surfaces, as is done with most metals. Alternately, using other reagents, crystalline materials may be made to polish or etch differentially on different surfaces; this would yield information on orientation, differential solubility, and distribution of defects, etc. The minerals to be examined by this technique must have a moderate to high conductivity. The electropolishing technique was developed for pyrite during a study of oxidation processes.

A simplified sketch of the electropolishing technique is shown in Fig. 1. The crystal is immersed in a solution of appropriate composition. An electrical potential is placed across the crystal and solution such that the crystal acts as an anode, and a metal plate or cylinder in the solution a cathode.

If the crystal can be electropolished in the solution, the general relationship between applied voltage and current density will somewhat resemble the curve in Fig. 2 (Tegart, 1959, p. 3). Current density is defined as the number of amperes per unit area of reactant surface.

Those portions of the curve characterized by a change in current den-



FIG. 1. Generalized sketch of electropolishing arrangement.

sity with respect to voltage result in etching or pitting of the crystal, whereas the areas where current density remains constant during voltage change, result in electrically polishing the crystal. During polishing, a thin film of either gas or viscous liquid is formed about the crystal. The film is thinner over raised portions of the crystal surface; thus these portions are acted upon more strongly by the surrounding liquid, and the crystal surface becomes leveled. It can thus be seen that viscosity, tem-



FIG. 2. Typical voltage current density curve for a material which is capable of being electropolished.



FIG. 3. Voltage current density curves for pyrite.

- A) Concentrated phosphoric acid saturated with chromic acid.
- B) $4\frac{1}{2}$ parts of concentrated phosphoric acid saturated with chromic acid and $\frac{1}{2}$ part of concentrated hydrochloric acid.
- C) 4 parts of concentrated phosphoric acid saturated with chromic acid and 1 part of concentrated hydrochloric acid.
- D) $3\frac{1}{2}$ parts of concentrated phosphoric acid saturated with chromic acid and $1\frac{1}{2}$ parts of concentrated hydrochloric acid.

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FIG. 4. The effects of electropolishing pyrite. Magnification 125 times.

- A) Original cube surface.
- B) Etched surface after treatment with less than critical voltage.
- C) Polished surface after treatment in the critical voltage range.
- D) Pitted surface after treatment above critical voltage.

perature, concentration and agitation of the solutions tend to vary the polishing characteristics.

Only a limited amount of knowledge is available on the theory of choosing a polishing solution; the choice in general is based upon empirical techniques. A few examples have been examined in some detail (Tegart and Vines, 1951; Casey and Bergeron, 1953).

A variety of the more common polishing solutions were tried in an attempt to find one whose current density-voltage curve shows the characteristic polishing plateau. The solution which produced the best polish consisted of four parts (by volume) of concentrated phosphoric acid (85.9% by weight) saturated with chromic acid, to one part of concentrated hydrochloric acid (36% by weight). Curve C of Fig. 3 shows the electrical characteristics of this solution. Curves A, B and D show the variation in shape and position of the curves with respect to varying concentrations of hydrochloric acid. Within the polishing region of the curve (about 0.375 ampere/cm²) the viscous film formed rapidly; this caused a large drop in current and decrease in polishing rate. This tendency could probably be decreased with either use of higher temperatures or rapid stirring. It was found that the crystal could be removed from the solution, washed, and dried several times during the run with satisfactory results.

The effects of electropolishing under conditions depicted in curve C, Fig. 3, are shown in Fig. 4. Part A shows the original pyrite cube surface. Part B shows a surface etch pattern developed at voltages below the ideal range. Part C shows the surface developed in the polishing range and Part D shows the effect of gaseous pitting at excessive voltages. All photographs are portions of the same surface. Polishing on surfaces other than cube faces resulted in a poorer development of polishing.

The mechanism of the reaction was not examined in detail, but it seems likely that the viscous layer developed at the crystal surface probably consists of $Fe(PO_4)_2$, the most insoluble of the three iron salts. The sulfur from the pyrite probably goes into solution as the sulfate ion as a result of oxidation by the chromic acid.

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