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Effects of Turbomilling Parameters on the Simultaneous Grinding and Ferric Sulfate Leaching of Chalcopyrite

By D. A. Rice, J. R. Cobble, and D. R. Brooks

UNITED STATES DEPARTMENT OF THE INTERIOR



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**UNITED STATES DEPARTMENT OF THE INTERIOR
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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	mL	milliliter
cm	centimeter	μm	micrometer
g	gram	oz/st	ounce per short ton
h	hour	pct	percent
kg	kilogram	rpm	revolution per minute
kW·h	kilowatt hour	st	short ton
kW·h/st	kilowatt hour per short ton	vol pct	volume percent
L	liter	wt pct	weight percent
min	minute		

EFFECTS OF TURBOMILLING PARAMETERS ON THE SIMULTANEOUS GRINDING AND FERRIC SULFATE LEACHING OF CHALCOPYRITE

By D. A. Rice,¹ J. R. Cobble,² and D. R. Brooks³

ABSTRACT

As part of its mission, the U.S. Bureau of Mines continues to develop technologies that will enable cost-effective compliance with environmental requirements. To this end, investigations to improve the kinetics of chalcopyrite dissolution by simultaneous grinding-leaching in ferric sulfate solution were conducted using the Bureau-designed turbomill. The development of such a hydrometallurgical process would provide a nonpolluting alternative to conventional smelter technology. The effects of mill rotational speed and volume percent solids on the leaching time and energy required for copper extraction from chalcopyrite were evaluated. The results of this laboratory study have shown that the simultaneous grinding-leaching of chalcopyrite concentrate using the turbomill is technically feasible with essentially 100 pct extraction of copper being possible at 90° C. An optimal energy consumption of 1,153 kW·h/st of copper leached was found in batch testing with the mill operating at 400 rpm with a slurry containing 35 vol pct chalcopyrite. This energy consumption, while higher than other proposed leaching processes (about 600 kW·h/st), is of the same order of magnitude. Further refinement of the energy requirements would require additional testing using a continuous circuit. Leaching times of less than 1 h, for 80 pct copper extraction, are achievable using the turbomill grind-leach process.

¹Group supervisor, Salt Lake City Research Center, U.S. Bureau of Mines, Salt Lake City, UT.

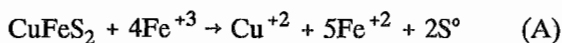
²Research chemist, Tuscaloosa Research Center, U.S. Bureau of Mines, Tuscaloosa, AL.

³Metallurgist, Tuscaloosa Research Center.

INTRODUCTION

Chalcopyrite concentrates are traditionally smelted with resultant sulfur dioxide emissions. As part of its mission, the U.S. Bureau of Mines continues to develop technologies that will enable cost-effective compliance with environmental requirements, including methods to minimize or eliminate environmentally objectionable processes or wastes. To this end, investigations to improve the kinetics of copper dissolution from chalcopyrite by simultaneous grinding-leaching in ferric sulfate solution were conducted by the Bureau. The development of new hydrometallurgical processes such as this could offer industry low-cost, low-pollution alternatives to traditional pyrometallurgical practices.

The hydrometallurgical leaching of chalcopyrite with acid ferric sulfate solution as an alternative process to smelting has been studied extensively (3-4, 9-15, 23-24).⁴ The overall leaching process may be represented by the following reaction.



Munos, Miller, and Wadsworth (11) have suggested that the rate limiting step is the transport process of electrons through the dense, tenacious layer of elemental sulfur deposited on the unreacted chalcopyrite particles as a reaction product.

Several proposed processes (13) for leaching chalcopyrite with ferric sulfate incorporate a grinding step to enhance the leaching kinetics. The University of Utah-Martin Marietta process (13) attrition grinds the concentrates to a mean particle size of 2 to 3 μm and then employs a two-step leach at 90° C with an intermediate

oxidation step to remove the elemental sulfur coating from the particle surface so that the second leach step can proceed at a faster rate. Total leaching time is reported to be 15 h. The ElectroSlurry⁵ process attrition mills the concentrate to about 2 μm and then leaches part (39 wt pct) of the concentrate at 90° C for 5 h using sparged oxygen to generate ferric iron in situ. The leach liquor is combined with the rest of the ground concentrate (61 wt pct) to convert the solids to covellite and digenite by reacting the slurry with sulfur dioxide. Copper is electrowon from the slurry, with ferric iron being generated at the anode instead of oxygen, as is the case in conventional electrowinning. This eliminates the oxygen over potential—the major cost factor in conventional electrowinning.

The direct electrical energy required for both the University of Utah and the ElectroSlurry processes has been estimated from reference 13 to be on the order of 600 kW·h/st of copper leached.

The Bureau turbomill (also known as an attrition grinder) has been used in the past to produce a wide variety of ultrafine minerals (1-2, 5-8, 16-22, 25). The present work was initiated on the premise that it might be possible to use the turbomill for simultaneously grinding and leaching chalcopyrite in a single step. If under suitable operating conditions, the elemental sulfur barrier could be attrited from the chalcopyrite during grinding and leaching in the turbomill, then a fresh chalcopyrite surface would be exposed and leaching rates might be enhanced. Further, this concept of attrition scrubbing the rate-controlling diffusion layer might also be applied to other high-value mineral systems whose leaching involves a diffusion controlled reaction.

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D. Dahlstrom and J. D. Miller, University of Utah, and Professor G. Warren, University of Alabama.

MATERIALS

The use of the Bureau patented (5) turbomill for attrition grinding to ultrafine sizes is well documented, and the general design and operation of the mill has been described previously (25). The turbomill (fig. 1) was constructed of stainless steel. The rotor (4.75 cm diam) was composed of vertical bars set in the slots of upper and

lower disks, which were rigidly attached to the shaft. The cage-like stator (24.75 cm height) was composed of vertical bars attached to rings at the top and bottom. The rotor and stator were positioned inside a stainless steel water-jacketed container. The torque on the mill rotor was monitored with an Eaton Lebow Model 7540 strain gage

⁴Italic numbers in parentheses refer to items in the list of references at the end of this report.

⁵Reference to specific products does not imply endorsement by the U.S. Bureau of Mines.

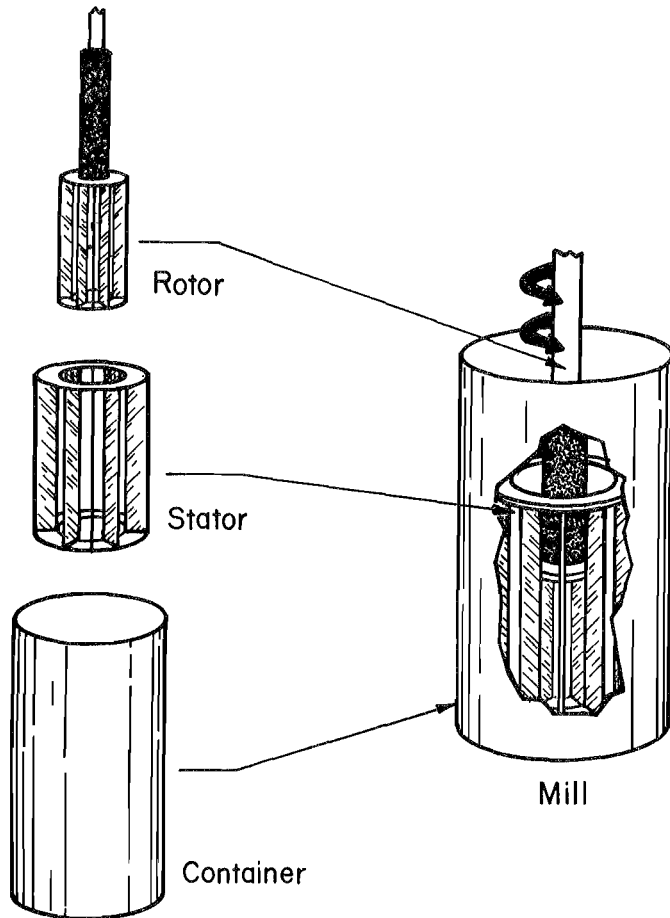


Figure 1.—Stainless steel turbomill.

indicator. The temperature was monitored using a metal-jacketed thermocouple inserted into the slurry, and was controlled within 2° C by circulating heating or cooling water through the water jacket of the container.

The stainless steel mill had a working volume of 2,250 mL, and was used for all tests in this investigation, except test 1. Test 1 was carried out using a similarly designed 4.1-L ultra-high molecular weight (UHMW) polyethylene mill. This mill structurally distorted at the elevated 90° C temperature.

The chalcopyrite concentrate used in this study was obtained from Cyprus Bagdad Copper Co., Bagdad, AZ. The as-received sample was split into smaller representative batches and stored in a moist state in sealed plastic

bags. The moisture content of the concentrate was 7.6 pct. The concentrate analyzed, on a dry weight percent basis, 28.0 Cu, 26.0 Fe, 4.7 SiO₂, 1.5 Al₂O₃, and 0.08 Mo. It was also analyzed for precious metals, and contained 0.9 oz/st Ag and no gold. Mineralogical examination by X-ray diffraction identified chalcopyrite as the major constituent, with minor amounts of pyrite, and trace amounts of quartz, plagioclase, and microcline feldspars also being present.

Ottawa sand, 20 to 30 mesh, with American Society for Testing and Materials (ASTM) designation C-190, was used as grinding media in all tests. Typical chemical analysis of ferric sulfate obtained from Tennessee Chemical Co., Atlanta, GA is given in table 1.

Table 1.—Chemical analysis of ferric sulfate

	<i>Analysis, pct</i>
Water soluble iron, expressed as Fe	20.9
Water soluble Fe ⁺³	19.4
Water soluble Fe ⁺²	1.5
Insolubles, total	5.5
Free acid	2.7
Moisture	3.5

A ferric sulfate stock solution was prepared by mixing large batches in the following proportion: 1 kg ferric sulfate, 1 L deionized water, and 10 mL concentrated sulfuric acid. After mixing, the solution was allowed to settle overnight, and the solution was decanted from the residual solids. The decanted solution had a specific gravity of 1.57 and the following analysis, in grams per liter: Fe_{total}, 167.5; Fe⁺³, 159.8; and Fe⁺², 7.7.

This ferric sulfate solution concentration was chosen to provide the stoichiometry required by reaction A for the ferric sulfate leaching of chalcopyrite, consistent with the desired volume percent solids and liquid volume required for operating the mill (2,250 mL operating volume), without precipitation of the solubilized copper. Earlier beaker tests with ferrous and copper sulfate mixtures had indicated that the Fe⁺² and Cu⁺² reaction products from the leaching reaction could exceed solution solubility to form a precipitate if higher ferric sulfate concentrations were used. The copper-iron precipitate was essentially insoluble in water.

EXPERIMENTAL PROCEDURE

The following procedure was developed to evaluate mill operating parameters on the leaching rate and energy requirements for leaching of chalcopyrite with ferric sulfate solution in batch testing.

Quantities of Ottawa sand, ferric sulfate solution, and chalcopyrite were calculated to give the proper proportions required to achieve (1) the desired volume percent solids in the mill, (2) the mole ratio of ferric ion to cupric ion

desired for leaching, and (3) the total volume required to fill the mill to its operating volume of 2,250 mL. From stoichiometric considerations, the concentration of the ferric sulfate solution fixes the maximal amount of chalcopryrite and sand addition possible for a given solids loading.

For this study, the solids loading (grinding media and chalcopryrite) in the turbomill is most appropriately expressed in percent of the mill working volume. Consequently, the term "volume percent solids" used in this report is defined as:

$$\text{volume percent solids} = \frac{\text{volume (sand + chalcopryrite)}}{\text{volume (sand + chalcopryrite + solution)}}$$

For each test, the mill was preheated to 90° C by circulating hot water through the water jacket and then started. Preheated ferric solution and sand were then added to the mill, and the temperature was readjusted. Chalcopryrite was then added, and the leaching tests were run for approximately 5 h.

The torque and horsepower were continuously monitored during each test using the digital output from the strain gage sensor on the mill drive shaft. The percent copper extraction as a function of time was determined using the following technique: Slurry samples were withdrawn using a 20-mL pipette at intervals of 30, 60, 120, 180, 240, and 300 min. The 20-mL samples were screened at 50 mesh to remove the coarse sand and then vacuum filtered. The filtered residue was washed four times with 25 mL of 1 pct sulfuric acid and then four times with similar volumes of deionized water to remove all soluble copper. The filtrate and wash water containing the leached copper were combined. The washed residue (filter cake) containing the unleached copper was dried at 90° C, weighed, and then a weighed portion was digested in aqua regia. The digested sample and copper-containing filtrate were analyzed using standard atomic-absorption techniques. From the resulting copper assays of the components of each slurry sample, the ratio of soluble copper to total copper in the sample was determined and reported as percent copper extracted.

EVALUATION OF OPERATING PARAMETERS

Turbomill grinding-leaching studies were conducted at 90° C to establish the effect of mill operating parameters on the leaching of chalcopryrite with ferric sulfate solution. The major parameters investigated were (1) mill rotational

speed and (2) solids loading in the mill. The work performed in each area is discussed separately. A summary of the test operating conditions and results is provided in table 2.

Table 2.—Summary of test conditions and results of grinding-leaching of chalcopryrite in ferric sulfate solution

Test	Mill feed, pct solids	Mill speed, rpm	Temp, °C	Ottawa sand, g	Ferric sulfate		Chalcopryrite, g	Time, ² min	Energy consumed kW·h/st Cu
					Mole ratio, Fe ⁺³ /Cu ⁺²	Vol, mL			
1 ¹	22.6	1,571	86.9	2,173	4.96	3,149	425.2	135	14,544
2	20.0	1,200	90.3	994	3.92	1,800	298.1	110	11,751
3	20.0	1,600	89.8	994	3.92	1,800	298.1	95	21,223
4	20.0	800	81.6	994	3.92	1,800	298.1	206	6,749
5	20.0	800	89.0	994	3.92	1,800	298.1	160	5,706
6	20.0	400	88.6	994	3.92	1,800	298.1	350	1,328
7	25.0	1,200	89.8	1,305	3.93	1,690	279.4	90	10,257
8	25.0	800	90.5	1,305	3.93	1,690	279.4	112	4,112
9	15.0	1,200	90.2	728	5.36	1,910	231.2	138	18,026
10	15.0	800	88.8	728	5.36	1,910	231.2	208	9,020
11	25.0	400	89.1	1,305	3.92	1,688	279.4	313	1,335
12	35.0	1,200	90.7	1,956	5.21	1,463	182.4	54	10,719
13	40.0	800	89.9	2,236	3.92	1,350	223.5	111	4,599
14	40.0	400	89.7	2,236	3.92	1,350	223.5	178	1,230
15	35.0	400	90.0	1,947	3.91	1,460	242.26	200	1,153
16	50.0	400	90.2	2,847	3.94	1,130	186.37	130	1,310
17	50.0	800	90.2	2,847	3.94	1,130	186.37	59	3,075
18	60.0	400	90.2	3,470	3.92	900	149.07	97	2,250

¹Test 1 was run using a UHMW polyethylene mill. All others were conducted using a stainless-steel mill.

²Time required for 80 pct copper extraction.

PRELIMINARY STUDIES

Initial testing indicated that only about 11 pct of the copper contained in the chalcopyrite could be extracted in a 1 h grind-leach time at 25° C. These results are in general agreement with the literature (17), which indicates that the leaching reaction proceeds very slowly even with finely ground chalcopyrite at ambient temperatures. To maximize reaction rates in an unpressurized reactor, subsequent testing was carried out at 90° C.

The results of the first test at elevated temperature, test 1, which was carried out using the UHMW polyethylene mill, are summarized in table 2 and in figure 2.

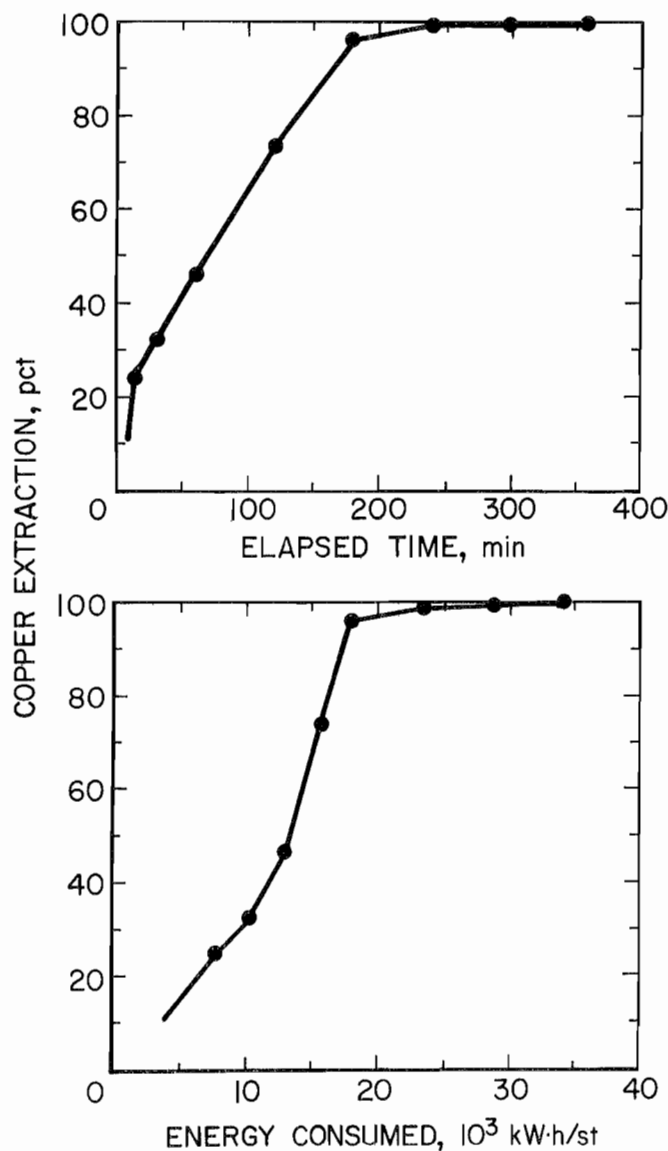


Figure 2.—Time and energy requirements for copper extraction—test 1. Test conditions: 22.6 vol pct total solids; 1,571 rpm; temperature, 86.9 C; 4.1 L UHMW Mill; $Fe^{+3}/Cu^{+2} = 4.96$.

As shown in figure 2, high copper extractions were obtained, and the rate of copper extraction was nearly linear. In a 1 h leach time, 47 pct Cu extraction was achieved at 90° C, at a volume percent solids of 22.6 pct, and a mill operating speed of 1,570 rpm. In a 180-min leaching period, the extraction increased from 95 to 97 pct. For comparative purposes, a review of the literature (13) indicates that the University of Utah-Martin Marietta ferric sulfate acid leach process, which incorporated a ferric sulfate leaching step after attrition grinding, required leaching times of about 12 h to achieve 92-pct Cu recovery (15 h for 99-pct recovery). The same reference states that the Envirotech Corp. ElectroSlurry process required about 5 h leaching with oxygen sparging to achieve 97 pct Cu recovery after attrition grinding of the chalcopyrite.

Figure 2 also shows that the energy required for leaching, expressed as kilowatt-hours per short ton of copper leached, was very high in this preliminary test. The energy consumption was linear with respect to percent copper extraction. To achieve 95 to 97 pct Cu extraction, corresponding to a 180-min leach time, the energy required was 20,000 kW·h/st of copper leached. For comparison, the energy required for the University of Utah-Martin Marietta ferric sulfate acid leach process has been estimated from the cited reference 13 to be approximately 630 kW·h/st of copper leached. Similarly, the ElectroSlurry process for grinding, leaching, and converting of chalcopyrite to covellite, digenite, and ferrous sulfate has been estimated at 560 kW·h/st of copper leached. In the ElectroSlurry process, the copper is subsequently electrowon from the copper sulfide-ferrous sulfate slurry.

Because of the high energy consumption, efforts were undertaken to examine the major mill operating variables in an attempt to reduce the energy requirements. Also, as discussed previously, the rest of the investigation was carried out using a stainless steel mill, which could withstand the elevated 90° C temperature.

EFFECT OF MILL SPEED

A series of tests was run using the stainless steel mill to determine the effect of mill rotational speeds from 400 to 1,600 rpm, with volume percent solids held at 20. Temperature was controlled at 90° C. The ratio of ferric to cupric ion was 3.92, slightly below the stoichiometric requirement of 4.0 indicated in reaction A.

The time and energy requirements as a function of mill operation speed for testing carried out at 20 pct solids are shown in figure 3. The rate of copper extraction is directly dependent on increasing mill operating speed—at 400 rpm, about 6 h are required to achieve 80-pct Cu extraction, compared with only 1.5 h at 1,600 rpm.

In these tests, slightly less than the stoichiometric amount of ferric sulfate was actually used (3.92 mole ratio). This was due to a head analysis for copper that was subsequently revised. The decline in copper recovery, beyond the maximum, however, is not an experimental artifact. The decline may be related to the conversion of

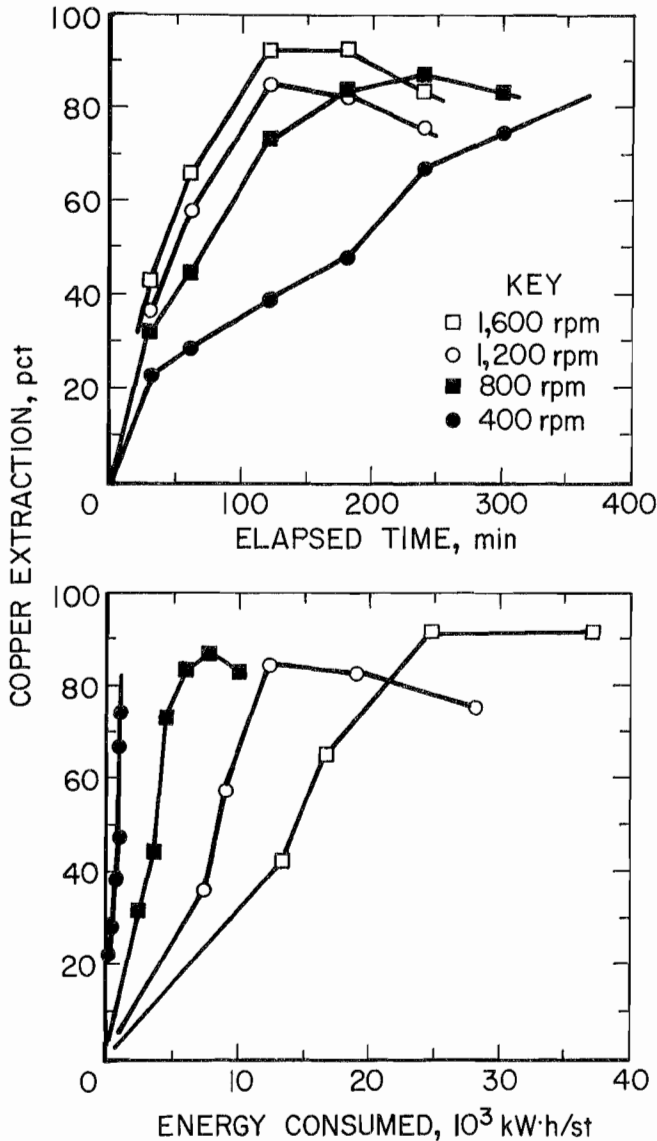


Figure 3.—Effect of mill speed on copper extraction at 20 vol pct solids.

the soluble copper to covellite or digenite as is intentionally done in the ElectroSlurry process. The practical implication of the decline in recovery is, that excessive leaching times may adversely affect overall copper extraction as well as consume additional energy.

Figure 3 also shows that the energy requirements decreased sharply with decreasing mill speed. At 1,600 rpm over 20,000 kW·h is required to leach 1 st of copper, compared with 1,300 kW·h at 400 rpm and 20 pct solids. Thus, as contrasted to typical speeds of 1,200 to 1,600 rpm where the turbomill has been traditionally operated as an attrition mill alone, the optimal speed on the basis of energy consumption for grinding-leaching of chalcopyrite appears to be considerably lower, about 400 rpm.

EFFECT OF SOLIDS LOADING

The effect of solids loading in the mill, expressed as volume percent solids for the mill operating speed of 400 rpm is shown in figure 4. The rate of extraction is seen to increase in a regular manner with increasing volume percent solids in the mill. At 25 vol pct solids, approximately 6 h are required for 80-pct copper extraction, whereas only 1.5 h are required at 60 vol pct solids, test 18. The effect of mill loading on energy consumption (fig. 5) is not quite as obvious as its effect on the rate of extraction.

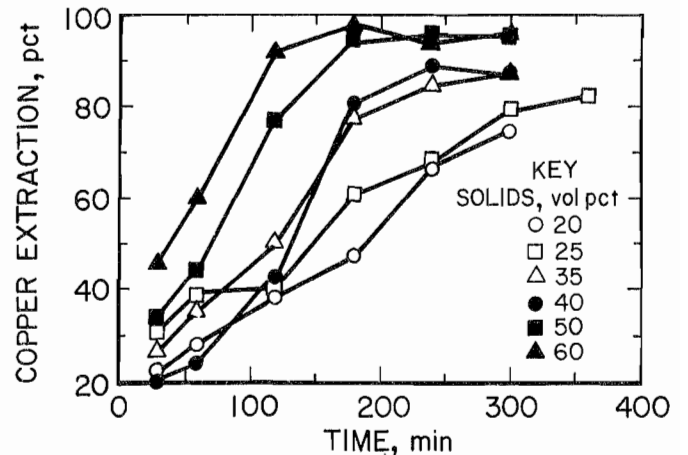


Figure 4.—Effect of volume percent solids on copper extraction at 400 rpm.

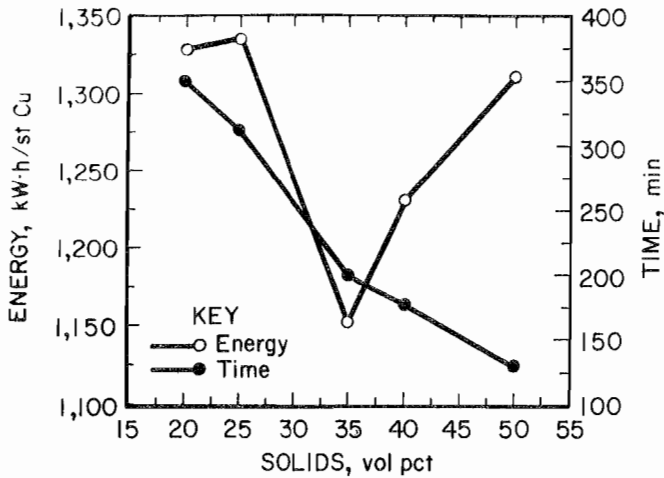


Figure 5.—Effect of volume percent solids on time and energy requirements for 80 pct copper extraction at 400 rpm.

While the time required to achieve 80-pct Cu extraction was found to decrease in a regular fashion with increasing volume percent solids, the energy was found to pass through a minimum level as volume percent solids increased. At 20 vol pct solids, about 1,325 kW·h/st of copper was required to extract 80 pct of the copper. As the solids were increased, the energy required decreased and reached a minimum value of approximately 1,153 kW·h/st of copper leached at 35 vol pct solids. Further increase in volume percent solids resulted in a rapid increase in energy requirements. At low volume percent solids, and consequently higher chalcopyrite to sand ratio, there was apparently insufficient grinding media to facilitate grinding-leaching, whereas above 35 vol pct solids, overloading of the mill with sand and chalcopyrite probably occurs which consumes power, but does not further facilitate leaching.

The energy consumption of 1,153 kW·h/st of copper leached obtained in this investigation appears high when compared with 630 kW·h/st for the University of Utah-Martin Marietta ferric sulfate acid leach process and the 560 kW·h/st copper leached for the ElectroSlurry process. However, a direct comparison of the energy

requirements should not be made from the batch-leaching tests at this stage of development. Rather, the data suggest that the turbomill energy consumption is on the same order of magnitude as the other proposed leaching processes. Further refinement of the energy requirements would require additional testing using a continuous circuit.

The results of all tests conducted using the stainless steel mill are summarized in figures 6 and 7. Figure 6 shows the leaching time required to achieve 80 pct Cu extraction from chalcopyrite as a function of volume percent solids loading and mill operating speed. Figure 7 shows the corresponding energy required to achieve 80-pct Cu extraction as a function of the same variables. The program used to produce figures 6 and 7 extrapolates beyond the region of experimental data, and the extrapolations may not be reliable, particularly in the region of high mill speed along with high fraction solid.

From figure 6, it is clear that the time required for leaching decreases with increasing solids loading and mill speed. The general shape of the surface plot suggests that leaching times as short as 40 min should be possible at solids loading in the 40 to 60 pct range with mill speeds of 1,600 rpm. These short leaching times, compared with the 5 h for the ElectroSlurry and the 12 h for the University of Utah processes, on preatritted chalcopyrite, strongly suggests that leaching rate is enhanced by attrition of the surface during leaching. Enhanced leaching rates, however, must be obtained at the expense of energy required for attrition.

Figure 7 shows the general trend that energy requirements decrease substantially with decreasing mill speed. At 400 rpm, a minimal energy level is indicated for the 20 to 50 pct solids range. This range, while broader due to data smoothing, is in general agreement with the minimum shown at 35 pct solids loading in figure 5. The actual economically optimal levels for solids loading and mill speed for a specific grinding-leaching operation would have to be determined through detailed process evaluation after additional testing, preferably under continuous operating conditions, to further define the operating range of interest.

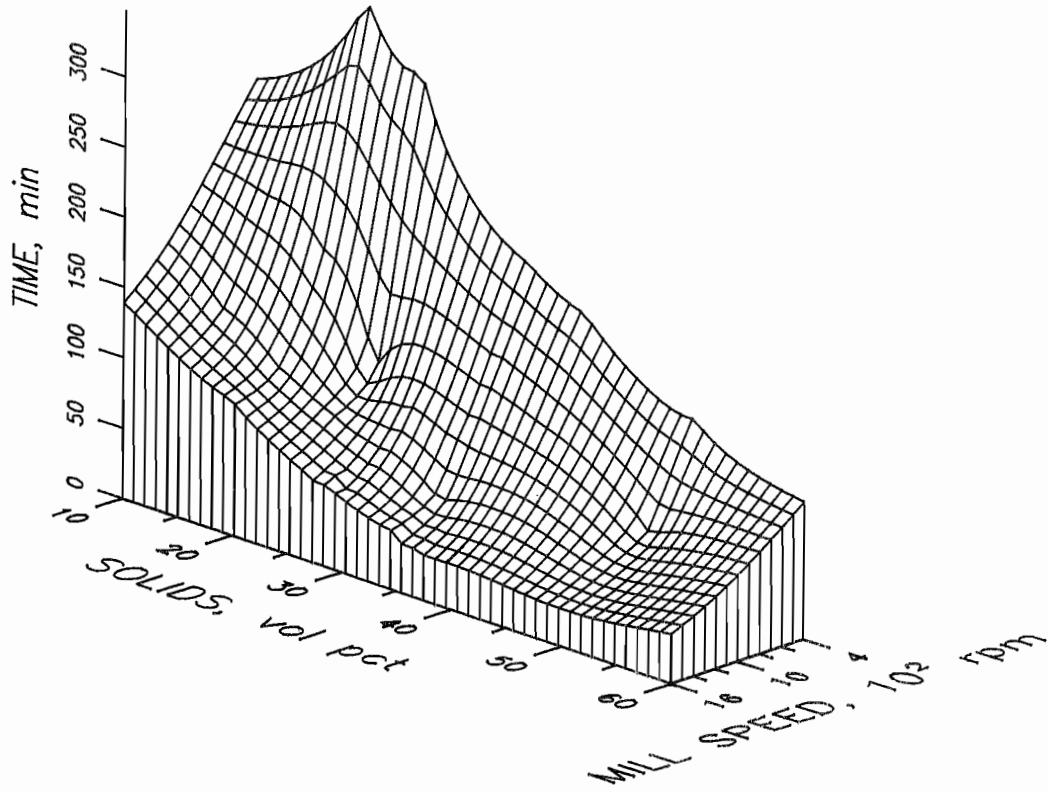


Figure 6.—Time required for 80 pct copper extraction as function of mill speed and volume percent solids.

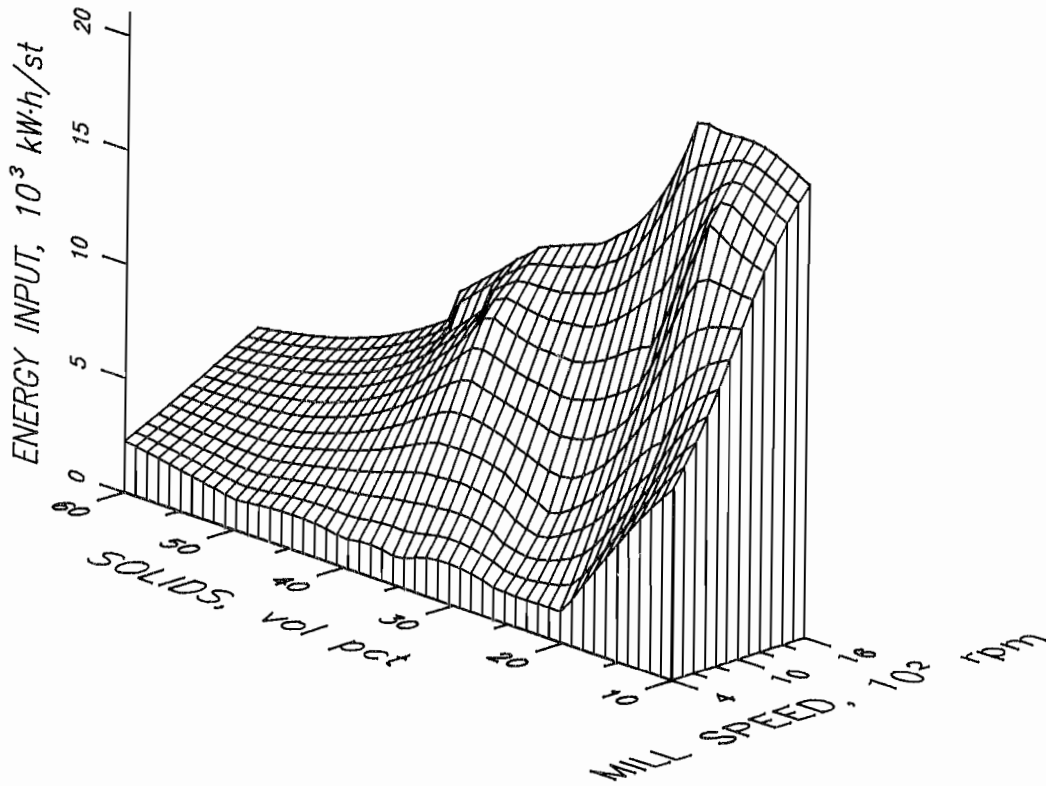


Figure 7.—Energy required for 80 pct copper extraction as function of mill speed and volume percent solids.

CONCLUSIONS

The results of this laboratory study have shown that simultaneous grinding-leaching of chalcopyrite concentrate in ferric sulfate solution using the turbomill is technically feasible. Over the test conditions studied, leaching times to achieve 80-pct Cu extraction ranged from 54 to 350 min, and energy requirements ranged from 21,223 kW·h/st of copper leached to as low as 1,153 kW·h/st leached. The following conclusions and observations have been made from this investigation.

1. The rate of leaching at 25° C is not significantly enhanced using the turbomill as a grind-leach reactor.
2. Essentially 100-pct extraction of copper is possible using the turbomill as a grind-leach reactor at 90° C.
3. The rate of leaching increases with increasing mill operating speed and with increasing volume percent solids in the mill, thus supporting the premise that scrubbing-attrition of the chalcopyrite surface to expose fresh surfaces would enhance leaching rates.

4. The energy required for leaching decreases sharply with decreasing mill operating speed.

5. The mill operating speed (about 400 rpm) for minimum energy consumption in ferric sulfate grinding-leaching of chalcopyrite is well below the 1,200 to 1,600 rpm speeds where the mill was evaluated as a grinder alone.

6. The energy required for leaching passes through a minimum level as the amount of solids is varied. For the conditions studied, a minimum energy level of 1,153 kW·h/st of copper extracted occurred at 400 rpm and 35 vol pct solids at a recovery of 80 pct.

7. The energy consumption of 1,153 kW·h/st of copper leached obtained in this investigation is on the same order of magnitude as other proposed leaching processes (about 600 kW·h/st). Further refinement of the energy requirements would require additional testing using a continuous circuit.

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