Influence of an Organic Polymer in Ball-Mill Grinding of Quartz, Dolomite, and Copper Ore

By A. R. Rule, A. J. Fergus, and C. B. Daellenbach
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Rule, A. R. (Albert R.)

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<th>Abbreviation</th>
<th>Definition</th>
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<tr>
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<td>°C</td>
<td>degree Celsius</td>
</tr>
<tr>
<td>cP</td>
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<tr>
<td>g</td>
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<td>gram</td>
</tr>
<tr>
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<td>g/L</td>
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</tr>
<tr>
<td>in</td>
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</tr>
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<td>kg/(kW·h)</td>
<td>kilogram per kilowatt hour</td>
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</table>
INFLUENCE OF AN ORGANIC POLYMER IN BALL-MILL GRINDING OF QUARTZ, DOLOMITE, AND COPPER ORE

By A. R. Rule,1 A. J. Fergus,2 and C. B. Daellenbach3

ABSTRACT

Research was done by the Bureau of Mines to determine the effect of a low-molecular-weight polymer on wet grinding of quartz, dolomite, and a copper ore. A 5-in ball mill equipped with a torque sensor for precise measurement of input energy was used for batch grinding tests. Grinding parameters were determined from size distribution data on the feed material and ground product. Zeta potential measurements were made to determine the effect of the polymer on the surface properties of the three materials, and a viscometer was used to measure the effect of the polymer on slurry viscosity.

The addition of the polymer caused a sharp increase in the negative value of the zeta potential for all three materials, indicating that pronounced adsorption was taking place. At a constant slurry percent solids, addition of the polymer caused a similar reduction in slurry viscosity for each material. For quartz, dolomite, and the copper ore, grinding efficiency, as measured by kilogram of product per kilowatt hour passing a given screen size, was not affected by the addition of the polymer. However, the lowering in slurry viscosity caused by addition of the polymer permits grinding at a higher slurry percent solids.

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2Metallurgist.
3Research supervisor.

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INTRODUCTION

Maintaining a domestic supply of mineral commodities necessary for national security and to meet the Nation's present demands and forecasted increased future demands will require greater utilization of low-grade reserves. When lower grade ores are mined, larger tonnages of materials must be processed to achieve the same production level. Thus, costs and energy consumption are significantly increased. Communciation is usually the most energy consuming and costly step in the overall mineral processing operation. U.S. beneficiation mills use approximately 29 billion kwh of electrical energy per year for size reduction (1).

Although commination is expensive, it is perhaps the most important operation in the processing of mineral raw materials. It has a significant role in the performance of separation steps that follow because it directly determines the size distribution of the material being processed, the extent of liberation, and the amount of excessively fine material produced.

Over the past 20 yr much research has been devoted to the development of detailed mathematical models to describe grinding circuit behavior. In the batch grinding model described by Mika, Berlitz, and Fuerstenau (2) the kinetics of disappearance of feed sizes is related to selection and breakage functions. The development and application of this model is thoroughly described in the literature (2-7).

During the period from June 1971 through February 1983, Bureau of Mines research performed by the University of California, Berkeley, studied the simulation of comminution processes and developed procedures for using the population balance grinding model in the scale-up design of ball mills. In the first phase of this study, relationships between the parameters of the grinding model and specific power input to the mill were identified, and a specific-energy-normalized model was developed. Experimental data obtained in three different size mills were used to demonstrate the usefulness of this form of the model in the scale-up design of ball mills (8). In the second phase of this research, a grinding model was proposed to describe size reduction kinetics and material transport in rod mill grinding (9). The third phase of this research dealt with five different topics involved with ball mill and rod mill grinding kinetics. A significant finding was that in ball mill grinding of mineral mixtures, a long time is required for a grinding circuit to attain steady state, and during this time, the composition of the mill contents and, hence, the breakage rate of the components continuously change (10).

The effect of conditions existing in the mill on grinding has been the subject of numerous investigations. As early as 1937, Coghill and DeVaney (11) demonstrated the superiority of wet grinding over dry grinding. The effects of organic and inorganic additives have been investigated by El'Shall, Gorken, and Somasundaran (12). Their research showed that quartz grinding could be improved with addition of dodecylammonium chloride under certain pH conditions. Oleic acid additives produced beneficial effects on hematite grinding under all pH conditions. Addition of common dispersants such as sodium silicate, sodium hexametaphosphate, and sodium tripolyphosphate had a deleterious effect on grinding of quartz and hematite. In another research effort (13), the effect of surface-active agents on hardness, microhardness, crushing strength, and wet grinding of hematite showed that dispersants have a greater effect than flocculants, and that each dispersant has a critical concentration at which grinding efficiency, as determined by reduction ratio at 80 pct passing, is a maximum. Raghavan (14) in his investigation of the grindability of a gabbro ore found that neither petroleum sulfonate nor ammonium lignin sulfonate at a dosage rate of 0.6 to 1.2 lb/ton seems to influence the grindability.
The role of dispersion and slurry rheology in the wet grinding of coal, sulfide ores, and nonsulfide ores has been the subject of several investigations by Klimpel (15-17). His research showed that as more fines are created during the grinding cycle, slurry viscosity increases, and that viscosity is significantly affected by slurry temperature (15). As slurry percent solids is increased, slurry viscosity increases; and at some high value of percent solids, the net production of fines decreases significantly. At this point addition of an organic polymer, Dow XFS 4272, decreases pulp viscosity and causes increases in the net production of fines. Under similar circumstances, more commonly used dispersants such as sodium silicate often do nothing to aid grinding and in some instances are a detriment to grinding (15).

Katzer, Klimpel, and Sewell (16) in further studies evaluated the effects of slurry percent solids and viscosity in the grinding of a taconite ore. A study of the effect of mill slurry volume on average torque for different percent solids showed that maximum torque occurred at a mill load volume equivalent to filling of the void space between the balls. Increasing slurry percent solids increases torque until the slurry becomes too viscous to permit cataracting of the ball-ore load. At this point torque decreases. For the taconite ore maximum torque was reached at 80 pct solids. When XFS 4272 was used the maximum torque was attained with the 83-pct-solids slurry. In a further study (17) it was shown that on "a maximum throughput basis, tumbling media mills need to operate on a slurry basis that is as thick as possible, yet still offers a low enough viscosity to keep grinding in a first order manner." When comparing net production of minus 325-mesh taconite to slurry percent solids, Klimpel (17) showed that maximum production of fines occurred at a mill load volume equivalent to filling of the void spaces between the balls, the same volume at which maximum torque occurs. Maximum production of minus 325-mesh taconite was obtained at 80 pct solids; with addition of XFS 4272, maximum production of minus 325-mesh taconite occurred at 83 pct solids. Since the highest production of fines occurs under conditions of highest torque, one would suspect that net energy consumption would also be highest at these conditions. Klimpel (17) reasons that because the total energy input in large-scale continuous tests was relatively constant with or without addition of rheology control chemicals, less energy is required per unit of throughput to a given size when XFS 4272 is added to the slurry. This is because addition of XFS 4272 permits grinding at a higher percent solids. Based on Klimpel's observations on large-scale tests, one could conclude that using the organic polymer would result in a net energy saving.

In this investigation, research was directed toward study of the physical and chemical properties of mineral slurries and the effect of these properties on grinding dolomite, quartz, and a copper ore. Addition of an organic polymer, XFS 4272, an experimental product of the Dow Chemical Co., and its effect on grinding were investigated in batch ball mill grinding studies.

MATERIALS

Dolomite, quartz, and a hard copper ore were selected for testing to represent soft and hard minerals and a multimineral ore, respectively. The dolomite, which came from a massive deposit in northeastern Washington, had a specific gravity of 2.85 and contained the following major constituents in percent: 30.0 CaO, 21.1 MgO, and 48.8 CO₂.

The silica, from a massive quartzite deposit in Rogue River, OR, had minor fracturing in sizes over 1/2 in, had a specific gravity of 2.64, and contained
the following major constituents, in percent: 96.8 SiO₂, 1.6 Fe₂O₃, 1.3 CaO, and 0.3 Al₂O₃.

The copper ore was obtained from Anaconda Company's Butte, MT, operation. It was termed "hard" southeast Berkeley ore and represented fresh to lightly argillized Butte quartz monzonite. The chief copper mineral was chalcopyrite. The ore had a specific gravity of 2.70 and contained the following, in percent:

<table>
<thead>
<tr>
<th>Component</th>
<th>Percent</th>
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<tbody>
<tr>
<td>SiO₂</td>
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<tr>
<td>Al₂O₃</td>
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<tr>
<td>Cu</td>
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</tr>
<tr>
<td>CaO</td>
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</tr>
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<td>K₂O</td>
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<td>1.8</td>
</tr>
<tr>
<td>Na₂O</td>
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Accurately sized 8-by-10-mesh (2.36-by-1.70-mm) fractions of the three materials were prepared for grinding studies by stage crushing in jaw, gyratory, and hammer mills combined with repeated sieve sizing.

EQUIPMENT AND PROCEDURES

GRINDING

In assembling the ball mill grinding unit for this investigation, the design used by Fuerstenau (8) was followed. Figure 1 shows the 5-in ball mill and digital readout controller for the torque sensor used to measure input energy to the ball mill. A detailed description of the grinding equipment is presented in the appendix.

FIGURE 1. - Grinding mill and torque measuring equipment.
For grinding tests the ball charge was maintained at 60 pct of the mill volume. To ensure a well-mixed starting feed, dry material and balls were layer-loaded into the mill, and the water was added. Slurry volume was kept constant to provide for 100-pct filling of the void space, and the liquid-to-solid ratio was adjusted to arrive at the desired slurry percent solids. Hence, when slurry percent solids was increased, the pulp volume remained the same, but the weight of feed material was increased. The weight of solids used for each material to achieve the desired slurry solids content is shown in table 1.

<table>
<thead>
<tr>
<th>Slurry solids content, pct</th>
<th>Dolomite</th>
<th>Quartz</th>
<th>Copper ore</th>
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</thead>
<tbody>
<tr>
<td>64</td>
<td>572</td>
<td>536</td>
<td>548</td>
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</tr>
<tr>
<td>84</td>
<td>964</td>
<td>886</td>
<td>912</td>
</tr>
</tbody>
</table>

The mill was stopped after running at the desired speed for a predetermined time, its contents were discharged, and the ground product was separated from the balls and wet-screened on a 400-mesh sieve. Both undersize (after filtering) and oversize products were dried. The oversize was then dry-screened for 30 min on an appropriate deck of Tyler standard sieves (1/2 series) using the Ro-Tap sifter.

**VISCOITY**

Viscosity measurements were made using either a Brookfield LVT or HVT rotational viscometer mounted on a Helipath stand. Slurry temperature was held at 23° to 25° C. Spindles and rotational speeds for the viscometers were selected to suit the viscosity range for each material. The viscosity as determined on mineral slurries is apparent viscosity and can be used to indicate trends for each mineral slurry. However, these values are not suitable for direct comparison between different mineral slurries when different spindles and speeds are used.

**ZETA POTENTIAL**

Zeta potential measurements were made using a Komline Sanderson ZR-10 Zeta Reader. In this device a dilute slurry (2 g/L solid) is continuously circulated through a conductivity cell, and samples are automatically transferred to the electrophoretic cell in 30-s intervals. A video camera focused on the electrophoretic cell transmits a magnified image to a video monitor. The speed of a moving grid on the display monitor is adjusted to match the average speed of the particles moving in the cell. Zeta potential, solution conductivity, and temperature are shown on a digital display. The Zeta Reader is calibrated with Minusil, a commercially available form of pure silica that has a standard zeta potential of minus 30 mV in distilled water.

**EXPERIMENTAL RESULTS**

**ZETA POTENTIAL AND VISCOSITY**

Zeta potential measurements were made on dolomite, quartz, and the copper ore in the presence of Dow's XFS 4272 short-chain organic polymer. Results of these measurements, shown in figure 2, indicate that addition of the polymer increases the negative value of the zeta potential for all three materials with additions of only 100 or 200 mg/L. The increase in the negative value of zeta potential indicates that the polymer exhibits strong specific adsorption at the mineral surfaces. This condition should favor pulp dispersion.
A study was made of the relationship between the level of addition of the organic polymer, slurry percent solids, and the viscosity of ground dolomite, quartz, and copper ore slurries. For dolomite results from viscosity studies showed that viscosity increases as slurry percent solids is increased. However, the highest percent solids that could be studied was for slurries at 82 pct solids because at higher percent solids the slurries were too viscous for accurate measurement with the available equipment. For the experiments carried out, the organic polymer had its greatest effect in lowering slurry viscosity at 82 pct solids. These results, obtained with a Brookfield T-bar spindle B at 12 rpm and given in figure 3, show that addition of the polymer up to 4 lb/ton of solids caused a significant decrease in slurry viscosity. At lower slurry percent solids, the polymer was not as effective in modifying viscosity.

A Brookfield No. 4 spindle at 6 rpm was used to measure viscosity of quartz and copper ore slurries because these slurries were too viscous for measurement with the T-B spindle. Viscosity measurements on quartz slurries showed that the organic polymer tended to lower viscosity. Beyond 76 pct solids, quartz pulps became too viscous for measurement with the available equipment. Therefore, the maximum observed lowering of viscosity was at 76 pct solids. Results in figure 4 show that slurry viscosity decreases sharply at a reagent level between 0.5 and 2.0 lb/ton of solids.

The copper ore slurries were extremely viscous, and viscosity measurements on slurries containing more than 70 pct solids were not possible with the available equipment. Data plotted in figure 5 show that a significant decrease in viscosity occurs at reagent levels ranging from 0.25 to 2.0 lb/ton of solids.

Both the increased negative value of the zeta potential and the lowering of viscosity by the additions of the organic polymer demonstrate that the reagent is an effective dispersant. These results are in general agreement with those of
previous investigators (13, 15-17), who found that this reagent tended to act as a dispersant and lowered viscosity for coal, taconite, and copper ore slurries.

**GRINDING**

Wet-grinding tests were carried out in the 5-in ball mill to study the effects of slurry percent solids and additions of an organic polymer on grinding dolomite, quartz, and the copper ore. In batch grinding, slurry viscosity increases as the grinding time is increased and more fines are created. An example of this phenomenon was reported by Tucker (18). For our study, grind times of 30 min for dolomite and 45 min for quartz and the copper ore were selected to assure that grinding had progressed sufficiently to observe the effects caused by slurry percent solids and viscosity. The slurry percent solids where optimum grinding occurred was different for each material.

**Dolomite**

A series of wet-grinding tests was done on 8- by 10-mesh dolomite using a 30-min grind time and slurry percent solids ranging from 70 to 84 pct. The results, plotted in figure 6, show that the amount of material ground finer than 212 μm reaches a maximum at 75 pct solids and decreases rapidly as the slurry solids content is increased above 78 pct. The shapes of the size distribution curves in figure 7 show that the products from grinding tests at 82 and 84 pct solids are different from those obtained at lower percent solids. It would appear that these slurries are too viscous for efficient grinding and that addition of the organic polymer might lower viscosity sufficiently to improve grinding. This did not happen. The data in figures 6 and 8 show that the polymer had no effect on the amount of fines created during grinding.
Grinding efficiency, calculated from grinding input energy measured with the torque sensor, is plotted in figure 9 as a function of slurry density at different levels of polymer addition. These results show that, without polymer addition, grinding efficiency increased as slurry percent solids was increased up to 80 percent solids. Above 80 percent solids, grinding efficiency decreased rapidly in both cases. At 70 to 76 percent solids the polymer had no effect on efficiency. For slurries of 78 to 84 percent solids, addition of the organic polymer actually caused a decrease in efficiency.

Quartz

Batch ball mill grinding tests were done on 8- by 10-mesh quartz slurries ranging from 64 to 82 percent solids. Size distribution curves for these tests are shown in figure 10. The leveling out of the slope of these curves for solids content of 74 percent solids and above indicates a decrease in the rate of generation of fines during grinding because of the increased solids loading. Figure 11 shows the grams of material ground finer than 212 μm for the same series of grinding tests. These data show that the amount of material ground finer than 212 μm decreased rapidly as percent solids was increased above 72 percent, and that addition of the organic polymer had no effect on the production of material finer than 212 μm. Grinding efficiency is plotted in figure 12 as a function of slurry percent solids and polymer level. These results indicate a gradual increase in grinding efficiency as percent solids is increased from 64 to 72 percent. Grinding efficiency begins to drop at 72 percent solids and decreases quite rapidly above 74 percent solids. Grinding becomes quite ineffective at 80 and 82 percent solids. Beyond 82 percent solids, slurries were too viscous for grinding. Previous investigators (17) indicated that the decrease in efficiency, after passing the maximum point on the efficiency-percent solids curve, results from high slurry viscosity. It is
in this area that they found that the use of viscosity control chemicals allows for higher net production. The results given in figures 11 and 12 show that, for quartz, addition of the polymer up to a level of 4 lb/ton had no effect in improving fineness of the ground product and did not contribute any improvement in grinding efficiency.

Copper Ore

The copper ore was selected for this study to represent a hard-to-grind multi-mineral ore. Viscosity data described earlier showed that slurries of this copper ore were considerably more viscous than similar slurries of either dolomite or quartz. For batch ball mill grinding tests on the copper ore, slurry percent solids was varied from 66 to 80 and grind time was held constant at 45 min. Size distribution curves for these tests are shown in figure 13. The decrease in the slope of these curves, as slurry percent solids increases, indicates a decrease in the percent of fine material in the product being produced during grinding. At 80 pct solids, the material discharged from the mill was extremely viscous, and as the size distribution curve (fig. 13) shows, grinding was poor. About 45 pct of the mill product was coarser than 1 mm. The data in figure 14, for the same series of tests, shows that without the polymer the weight of material ground finer than 212 \( \mu m \) decreases steadily as percent solids is increased from 70 to 78 pct and drops off sharply at 80 pct solids. Also these results show that addition of the polymer caused an increase in the weight of material ground finer than 212 \( \mu m \). The greatest difference occurs in the region from 72 to 80 pct solids. These results indicate that addition of the polymer in the grinding of the copper ore tended to increase the fineness of the ground product.

The data for grinding efficiency (fig. 15) show that without the polymer, there
is a slight increase in grinding efficiency up to about 72 pct solids. Efficiency then begins to decrease gradually and drops sharply at 80 pct solids. Furthermore, these results show that addition of the polymer had little or no effect on grinding efficiency at solids content lower than 74 pct. At 74 through 80 pct solids, addition of the polymer at the 4-lb/ton level resulted in a slight increase in efficiency. However, the efficiency was not increased sufficiently to exceed that achieved at 72 pct solids without addition of the polymer. Addition of the polymer at levels higher than 4 lb/ton probably would not be economically attractive.

SUMMARY AND CONCLUSIONS

Zeta potential, viscosity, and grinding studies were made on dolomite, quartz, and a copper ore to determine the effect of a commercial polymer (Dow XFS 4272) on ball mill grinding efficiency. Zeta potentials for the three materials showed a sharp increase in their negative value with addition of the reagent, indicating strong adsorption of the organic polymer. This condition should favor pulp dispersion.

Viscosity measurements for dolomite slurries showed that viscosity continuously increased as the slurry percent solids was increased, and that the polymer had its greatest effect in lowering slurry viscosity at 82 pct solids. For quartz the polymer also tended to lower slurry viscosity, with the maximum observed lowering occurring at 76 pct solids. Copper ore slurries were extremely viscous, and a significant decrease in slurry viscosity resulted with addition of the polymer to slurries even at 70 pct solids. Grinding studies were made using a 5-in. batch ball mill equipped to precisely measure input energy. Grinding efficiency was established by determining the particle size distribution of the ground product and relating it to energy input. The results of studies on dolomite show that maximum efficiency in grinding occurred at 78 to 80 pct solids. For slurries higher than 80 pct solids, grinding efficiency decreased rapidly, and there was a significant decrease in the fineness of the ground product. For dolomite, addition of the polymer had an adverse effect on grinding efficiency but no effect on the amount of fines created during grinding (figs. 6 and 9).

Grinding studies on quartz showed that grinding efficiency varied with slurry percent solids and that maximum efficiency occurred at about 70 pct solids (fig. 12). Data also show that the amount of material ground finer than 212 μm decreased as the slurry percent solids was increased above 72 pct. Grinding was
very ineffective at 80 and 82 pct solids. Addition of the polymer up to a level of 4 lb/ton had no effect on the amount of fines produced in grinding, and there was no improvement in grinding efficiency.

Data for grinding studies on the copper ore showed that the weight of material ground finer than 212 μm decreased as the slurry percent solids was increased. Addition of the polymer caused an increase in the weight of material ground finer than 212 μm. These results showed that, for the copper ore, addition of the polymer tended to increase the fineness of the ground product, and that there was a slight increase in efficiency at 74 pct solids and above with addition of the polymer at a level of 4 lb/ton. With or without addition of the polymer, grinding efficiency decreases as slurry percent solids is increased above 74 pct and drops off sharply at 80 pct solids.

This study has shown that for dolomite and quartz the polymer is adsorbed and lowers slurry viscosity, but has no effect on grinding efficiency or on the rate of production of fines. However, for the copper ore, addition of the polymer lowered viscosity and also tended to improve the fineness of the ground product and the grinding efficiency. However, even at a polymer level of 4 lb/ton, improvements in fineness of the grind and efficiency were not sufficient to exceed results achieved at 72 pct solids without addition of the polymer.

REFERENCES


The 5-inch-diam by 5.75-inch-long (inside measurements) ball mill was fabricated using 304 stainless steel. The mill was equipped with eight 1/8-inch-high lifters. Each lifter was 3/8 inch wide at the base and tapered to 1/8 inch wide at the top. Two stainless steel tires, 7.25-inch-diam by 1-inch-wide, were fitted on the outside of the mill to provide clearance for the lid fasteners and to support the mill during rotation. The mill was supported by two 2.5-inch-diam by 15-inch-long urethane-covered rolls with a 1-inch steel core shaft mounted in single-row, self-aligning sealed ball-bearing pillow blocks.

The center shaft of the direct-drive mill was connected by two flexible couplings to a S. Himmelstein and Co. zero speed, 0 to 200 lb·in, noncontact, torque sensor. The main shaft was driven by a Boston Gear 1-hp permanent magnet gear motor. Output from the torque sensor was transmitted to a Himmelstein model 6-201 processing unit with digital display of instantaneous values of mill speed as rpm, torque as lb·in, and calculated instantaneous horsepower. The processing unit provides a signal to a linear, three-pen recorder for recording of the three instantaneous values as a function of time.

Total energy input for a grind was calculated by estimating the area under the horsepower-time curve. Mill speed was regulated with a Ratiopax motor controller. Grind duration was determined by preset countdown counter set to shut off the mill drive motor when the number of revolutions required for the designated time period was completed. Variation between set time and actual grinding time was less than 0.5 pct on a 45-min grind.

The ball charge for the mill consisted of 68 1-inch-diam grade 440 stainless steel balls, heat-treated to Rockwell hardness 58C. Mill speed was set at 90 rpm, 68 pct of the calculated critical speed for the mill, ignoring the lifter effect. This speed was selected to operate the mill in the region of maximum breakage (19).1

1 Underlined numbers in parentheses refer to items in the list of references preceding this appendix.