Comminution behaviour of microwave heated two sulphide copper ores

Erol Kaya
Dokuz Eylul University, Department of Mining Engineering, Buca, Izmir, Turkey
Email: erol.kaya@deu.edu.tr

Received 16 March 2010; revised 5 August 2010

The effect of microwave irradiation on the comminution of two sulphide copper ores with different mineralogy were investigated to enhance the milling processes. Each ore was subjected to microwave radiation for varying time periods to determine their heating profiles. Changes in breakage characteristics with the microwave exposure time were quantified by sieving breakage, standard Bond work index grindability and single particle drop weight comminution tests. The results of the sieving breakage tests with the untreated and microwaved samples indicated significant improvements in the size reduction due to microwave energy induced macro fracturing. Differing ore mineralogy has been shown to affect the effectiveness of microwave assisted size reduction. The results of the standard Bond work index grindability tests performed with both the microwave treated copper ores, on the other hand, suggested no significant improvements in grindability. The single particle fracture experiments performed with an Ultrafast Load Cell (UFLC) indicated similar results of no significant improvements in the fracture energies of the microwave treated ore samples.

Keywords: Microwave irradiation, Comminution, Copper ore

Comminution is an important unit operation in the mining industry to reduce the particle size and liberate the valuable minerals. However, mechanical size reduction of ores is an energy intensive and highly inefficient process with less than 1% efficiency during grinding to generate new surfaces. Up to 70% of energy in any mineral processing plant goes to comminution. Clearly there is much to be gained from improving the efficiency of a breakage process. Microwave pretreatment of ores, as an alternative potential technology to bring the comminution cost down, has attracted the attention of many researchers.

Microwave assisted milling is the process of selectively heating minerals in ores prior to mechanical size reduction to reduce the cost of the comminution energy. In general, most metal sulphides and oxides get heated readily when subjected to a microwave irradiation, on the other hand, common gangue minerals containing silicates and carbonates are nearly transparent to microwaves and get heated slightly. This differential heating promotes grain boundary cracks or fractures between the different minerals due to differences in their adsorption behaviours and the thermal expansion coefficients.

Over the past three decades, significant amounts of research work have been carried out to investigate the impact of microwave pretreatment on the breakage behaviour of ores and minerals. Much of the work done in the field of microwave assisted comminution has been conducted by the former U.S. Bureau of Mines who recognized the potential of using microwaves to weaken ores prior to comminution and by the University of Utah’s Comminution Center who quantified the improvements in impact fracture energies of single particles subjected to microwave pretreatment using Ultrafast Load Cell. Various other researchers have also reported several aspects of the effect of microwaves on grindability and downstream recovery of minerals. In general, the effect of the microwave pretreatment was assessed by comparing the energy savings in comminution of the untreated to that of the microwaved ore. Reductions in the grinding energy (as kWh/t) up to 90% were reported for certain minerals and were strongly influenced by the ore type, mineralogy, particle size, microwave power level and exposure time.

Previous studies imply that, although researchers have attained much knowledge and experience about the fact that microwaves can thermally fracture materials, current status of this research work is still
fundamental. In general, these studies have been exploratory in nature and have also been performed on a laboratory scale. The results published to date, however, indicate that the future for the applications of microwave energy within the mineral industries is encouraging. Further research is needed to advance the application of the microwave energy in the mineral industry. The objective of this study is to evaluate the comminution behaviour of microwave pretreated two sulphide copper ores containing different mineralogy.

**Experimental Procedure**

**Ore characterization**

Two different complex sulphide copper ores with differing mineralogies were selected in this study. The ore samples, collected from two copper concentrator plant feeds, were crushed to below 25.4 mm in a jaw crusher and screened into size fractions. Minus 3.4 mm (6 U.S. mesh) material was discarded in this investigation. Plus 3.4 mm samples were divided into 1 kg samples and bagged. The characterization work, completed on plus 3.4 mm fractions only, included optical microscopy analysis of polished sections, quantitative X-ray diffraction (XRD) analysis and chemical analysis.

The first copper ore is a medium hard monzonite porphyry copper ore. Its main gangue minerals are quartz (45.7%), K-feldspar (18.9%), plagioclase (1.4%), muscovite (11.5%), pyrite (2.9%), kaolinite (4.4%) and iron oxides (2.3%). Copper bearing phases are predominantly mixed sulphide minerals. They are relatively coarse grained and associated with iron sulphides and copper-iron sulphides. Copper occurs as chalcopyrite, covellite, chalcocite and digenite. The ore contains 0.62% total copper and 0.045% oxide copper with 0.011% of molybdenite and 0.04 g/t of gold. The ore’s Bond work index is measured as 11.4 kWh/t.

The second copper ore is a medium hard andesite-hosted mineralization with veins, tabular or lens like breccia bodies. Its gangue mineralogy is dominated by magnetite (18%), K-feldspar (33%), quartz (11%), biotite (8%), plagioclase (5.1%), kaolinite (1.6%). Sulphide gangue consists of varying proportions of pyrite (7%), pyrrhotite and trace sphalerite. Copper mineralogy is overwhelmingly dominated by chalcopyrite with very little secondary speciation. Chalcopyrite textures are present in fine and coarse disseminations mostly in veins. The ore contains 1.39% total copper, 0.014% oxide copper and 0.21 g/t gold. The ore’s Bond work index is measured as 16.7 kWh/t.

**Microwave treatment**

A batch type microwave oven (Microdry, Inc., Crestwood, KY, USA) with a variable power output range of 1-6 kW, a volume of 1.3 m³ applicator and a frequency of 2.45 GHz was used. The 1 kg sample batches with plus 3.4 mm size were irradiated in the microwave oven at a power of 6 kW for varying durations of time. Samples were placed on a 0.09 m² ceramic surface and positioned in the centre of the oven.

Average bulk surface temperatures of the ore particles were measured using a Model Raytec Ranger 3I infrared thermometer after switching off the microwave power. It is desirable to heat the sulphide minerals contained within the ore quickly and to a high temperature. Since it is not feasible to measure the temperature of each sulphide mineral grain, a bulk surface temperature of the ore is recorded as an indication of the extent of sulphide mineral grain heating. Temperature readings within a given sample varied from particle to particle and decreased rapidly with time, therefore, the average surface bulk temperatures of each sample were recorded for the duration of approximately two seconds. It is assumed that the bulk surface temperature is a measure of the coupling between the microwave energy and the sulphide minerals and will, therefore, be an indication of the effect on milling properties. In simpler words, heating the samples to a higher temperature may increase the fracturing.

**Size reduction tests**

Microwave heating curves of both the copper ore samples were determined at varying microwave durations of time in the microwave oven, using 1 kg sample batches with 25.4 × 19.0 mm size fractions. The comminution benefits of the microwave pretreatment were determined by the sieving breakage, standard Bond work index grindability and single particle drop weight tests. All the sieving and comminution tests were performed in dry conditions.

**Sieving breakage tests**

The samples of 1 kg batches with a size fraction of 25.4 × 19.0 mm were exposed to microwave
irradiation for varying time periods. The microwaved and untreated samples were sieved using 19.0 mm screen for 3 min. The size reduction by the sieving breakage was defined as the weight percentage of the ore sample passing 19.0 mm screen size after 3 min. of sieving with a ro-tap shaker. Here, the sieving motion, caused by shaking and vertical tapping, generated the breakage or disintegration of particles.

**Bond work index grindability tests**

The standard Bond work index grindability tests were carried out to assess the energy savings resulting from the microwave pretreatment of both the copper ore samples. The work index is the comminution parameter, which expresses the resistance of the material to grinding and it corresponds to energy consumption as the kWh per short ton of ore to reduce the material from a theoretically infinite size to 80% passing a closing screen size. Because different minerals respond differently to microwave radiation, the influence of microwave radiation on the relative work index of different ore types will not be of the same magnitude. The samples of plus 3.4 mm material were irradiated in the microwave oven for varying time durations and stage crushed to minus 3.4 mm screen size in a roll crusher for the standard Bond work index grindability testing.

The grindability of the samples was determined according to the standard procedure developed by Bond. The feed was packed into a 700 ml graduated cylinder and placed in the mill and ground dry. The mill is 30.5 × 30.5 cm with rounded corners and a smooth lining. It runs at 70 rpm with a charge of 285 iron balls ranging from 1.55 to 3.68 cm in diameter and weighing 20.125 kg. Tests were performed at a closing size of 208 µm (65 mesh). The locked-cycle grinding test continued until the net grams of sieve undersize produced per mill revolution reached equilibrium at a circulating load of 250%. Then, the undersize products of last three cycles were screen analyzed, and the average of the last three net grams per revolution (Gbp) was the ball mill grindability. The ball mill work index, Wi (kWh/st) was calculated from the following equation:

\[
W_i = \frac{44.5}{P_i^{0.23} x G_{hp}^{0.82} x \left( \frac{10}{\sqrt{P}} - \frac{10}{\sqrt{F}} \right)} \quad \ldots \quad (1)
\]

where, \(P_i\) is the closing screen size in microns, and \(F\) and \(P\) are 80% cumulative passing sizes in microns for the feed and product, respectively. The tests were replicated and the average values were used.

**Single particle drop weight breakage tests**

The impact fracture energies of the untreated and microwaved samples were measured with single particle experiments using the Ultrafast Load Cell (UFLC) which is an improved drop weight tester developed at the Utah Comminution Center. It can measure the fraction of the input energy that is actually used in particle breakage.

The UFLC consists of a long vertical steel rod, equipped with strain gauges on which a single particle (or a bed of particles) is placed and impacted by a falling steel ball. The compressive wave resulting from the impact travels down the rod and is sensed by the solid-state strain gauges. This results in a voltage change in the wheatstone bridge, which is then recorded as a function of time using a digital oscilloscope. Compression experienced by a particle sitting on top of the anvil is calculated from the momentum balance of the falling steel ball and the deformation of the steel rod.

The single-particle fracture studies with the UFLC were conducted at the Utah Comminution Center, Utah, USA. The ore 1 samples of 1 kg batches with the size fractions of 25.4 × 19.0 mm were irradiated at different heating conditions in the microwave oven. Water quenching tests were performed at 25ºC.

**Results and Discussion**

**Microwave heating profiles**

Microwave heating curves of both the copper ore samples tested are presented in Fig. 1. As seen, the vein type copper ores heat better than the porphyry copper ores. This may be attributed to the amount of magnetite and pyrite contained in this ore. An important point needs to be mentioned here is that in several instances, as the porphyry copper ore samples were heated, portions of the samples absorbed enough microwave energy to become incandescent. This
Concept is known as thermal runaway, thus, once a localized hot spot forms due to increased exposure to microwave radiation, it consumes even more energy, while the surrounding material heats only slightly. During the recording of the average bulk surface temperatures of the samples with the infrared thermometer, the hot spots, reaching the temperatures of 1200°C, were excluded. The same phenomenon, i.e., thermal runaway, was not observed with the vein type copper ore.

**Sieving breakage tests**

The size reductions produced by sieving the microwaved samples as a function of average bulk surface temperatures are presented in Fig. 2. It is evident from the figure that the size reductions up to 50% can be achieved for both the ore types if heated sufficiently. The results pointed out that a differing threshold temperature existed for each type of copper ore and this threshold temperature must be exceeded for a significant size reduction to take place. These threshold temperatures occured at approximately 300°C and 500°C for the copper ores 1 and 2, respectively.

Degradation of the particles showing localized cracks and fractures after the microwave heat treatment was visually observed. During the screening, the fractures continue to propagate, resulting in size reduction. Two mechanisms can contribute to the improved breakage of the microwave irradiated ores. First, unequal thermal expansion of microwave energy absorbing minerals in a non-heating gangue matrix may cause micro fractures at the mineral grain boundaries by overcoming the tensile strength of the ore. Second, internal pressure created from super heated water vapor and gases (for example, SO₂ forms from the reactions of metal sulphides converting to metal oxides) may also cause fissures or cracks in the host rock. The sulphide minerals in the ore act as high-loss materials, i.e., heating readily, while the gangue minerals are virtually transparent to the microwaves. Sulfur dioxide (SO₂) produced from microwaving the sulphide minerals may ionize to the plasma, called outgassing. Outgassing, which was observed in this study during the microwave heating, was believed to create the cracks or fractures.

**Bond work index grindability tests**

The results of the Bond work index grindability tests with the microwaved and untreated copper ore samples are presented in Table 1, along with the irradiation times and temperatures. As seen in Table 1, there is no indication of improved grindability for both the microwaved copper ore samples heated up to 400°C. Essentially, the grindability work index values of the microwaved samples remained the same below 400°C.

Above 400°C irradiation temperatures, the Bond work index values increased very slightly about 7% for the porphyry type copper ore (ore 1) attributed to
KAYA: COMMINUTION BEHAVIOUR OF MICROWAVE HEATED TWO SULPHIDE COPPER ORES

459

the fusing of some of the particles observed at high
temperatures in this study. The Bond work index
grindability values decreased for the vein type copper ore (ore 2) in which fusing of particles was not observed. The largest decrease, i.e. improvement, in the work index (by 13%) occurred for the ore 2, which was heated up to 608ºC as seen in Table 1. It should be noted here is that the reproducibility of the Bond grindability work index testing was reported about 10%, i.e., the variations in the Bond index values below this value should not be evaluated\(^\text{27}\).

To show the fusing of the copper minerals observed in this study, a research grade specimen of chalcopyrite obtained from Ward’s Natural Science Establishment, Inc. (Rochester, NY, USA) was irradiated at 1 kW power for 30 s. An average bulk surface temperature of 240°C was measured and it reached in excess of 1000°C in certain areas. Figure 3 illustrates a significant fusing of the chalcopyrite mineral took place on the particle surface. It appears that fusion causes particles to become harder, thus overheating of localized areas may be responsible for the poor grinding results at higher irradiation temperatures.

The poor grinding results obtained in this study could be an indication that there were no new cracks formed at fine sizes and the benefits were at coarse sizes. Similar results of no improvement in the Bond work index values, performed on refractory gold, copper and complex sulphide ores, were also reported in the literature\(^\text{18,28,29}\).

**Table 1—Average surface bulk temperatures and measured Bond work index grindability values of the untreated and microwaved copper ore samples**

<table>
<thead>
<tr>
<th>Ores</th>
<th>Irradiation time (s)</th>
<th>Average surface temp. (ºC)</th>
<th>Work index (kWh/t)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ore 1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Untreated</td>
<td>12</td>
<td>58</td>
<td>11.4</td>
</tr>
<tr>
<td>Treated 1</td>
<td>30</td>
<td>87</td>
<td>11.7</td>
</tr>
<tr>
<td>Treated 2</td>
<td>180</td>
<td>321</td>
<td>11.5</td>
</tr>
<tr>
<td>Treated 3</td>
<td>360</td>
<td>420</td>
<td>11.6</td>
</tr>
<tr>
<td>Treated 4</td>
<td>600</td>
<td>588</td>
<td>12.2</td>
</tr>
<tr>
<td>Ore 2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Untreated</td>
<td>30</td>
<td>163</td>
<td>16.7</td>
</tr>
<tr>
<td>Treated 1</td>
<td>120</td>
<td>356</td>
<td>16.6</td>
</tr>
<tr>
<td>Treated 2</td>
<td>180</td>
<td>608</td>
<td>14.5</td>
</tr>
</tbody>
</table>

**Table 2—Fracture energies of 25.4x19.0 mm size particles measured by the ultrafast load cell (UFLC) for the untreated and microwaved ore 1 samples**

<table>
<thead>
<tr>
<th>Samples</th>
<th>Micro-wave heating (s)</th>
<th>Avg. surface temp. (ºC)</th>
<th>Fracture energy J/kg</th>
<th>Stand. dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Untreated</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Replicate 1</td>
<td>25</td>
<td>21.8</td>
<td>0.172</td>
<td></td>
</tr>
<tr>
<td>Replicate 2</td>
<td></td>
<td></td>
<td>24.5</td>
<td>0.253</td>
</tr>
<tr>
<td>Replicate 3</td>
<td></td>
<td></td>
<td>22.5</td>
<td>0.333</td>
</tr>
<tr>
<td>Treated</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Test 1</td>
<td>10</td>
<td>26.5</td>
<td>0.403</td>
<td></td>
</tr>
<tr>
<td>Test 2*</td>
<td>10</td>
<td>23.2</td>
<td>0.142</td>
<td></td>
</tr>
<tr>
<td>Test 3</td>
<td>60</td>
<td>22.2</td>
<td>0.231</td>
<td></td>
</tr>
</tbody>
</table>

* Water quenched at 25ºC.

**Single particle breakage tests**

About 50 particles from the untreated and microwaved Ore 1 samples was fractured using UFLC and the fracture energies of the samples were summarized in Table 2. In general, the fracture energies measured suggest no improvement for the samples subjected to microwave energy treatment. The results fluctuate within the experimental error, calculated from three replicates of untreated particles with 50 particles each. The results support the findings of the Bond work index grinding test, which suggested that there were no gains during the grinding of the heat treated samples. Water quenching, also, did not improve the grinding, although, the fracture energies are expected to be lower for the particles expanding and contracting elastically during heating and cooling.

Fig. 3—Scanning electron microscopy (SEM) micrograph of fused copper minerals on the surface of an irradiated chalcopyrite sample. White scale bar = 1000 micron.
Conclusion

The aim of this study was to quantitatively evaluate the effect of microwave pre-treatment on the comminution characteristics of two sulphide copper ores to improve grindability. The results of the sieving breakage tests performed at a relatively coarse size indicated that the microwave pretreatment of sulphide copper ores improved the efficiency of the size reduction due to macro fracturing. The size reductions up to 50% were achieved for both the copper ores when heated sufficiently. Differing copper ore mineralogy has been shown to affect the effectiveness of microwave irradiation. The results pointed out that a differing threshold temperature existed for each type of copper ore and this threshold temperature must be exceeded for a significant size reduction to take place. These threshold temperatures occurred at approximately 300°C and 500°C for porphyry and vein type sulphide copper ores, respectively.

The results obtained from the grindability studies, on the other hand, did not show significant improvements on the breakage characteristics of the two copper ores irradiated with microwaves. The Bond grindability work index values remained essentially the same at temperatures up to 400°C for the microwave treated porphyry copper ore samples, but, increased about 7% at a higher temperature of 588°C. On the other hand, although the grindability work index value has not changed up to a temperature of 356°C for vein type copper ore, it improved about 13% at a higher temperature of 608°C. The single particle drop weight tests with the porphyry copper samples indicated the similar results that the microwave heat treatment did not improve the fracture energies of copper ore samples.

In general, the results of this study with two sulphide copper ore samples indicate that microwave heat treatment improves macro fracturing, i.e., the benefits are at coarse sizes. On the other hand, there is less pronounced energy saving when the size reduction is carried out below a certain size. The existence of the fusion of the chalcopyrite particles caused by the excessive microwave heating was shown in this study. It appears that fusion causes particles to become harder, thus, overheating may be deleterious to improve grinding.

Acknowledgements

The author thanks the Dokuz Eylul University, Scientific Research Projects (BAP) Coordination Council, for partially funding this research. The author would also like to thank D. H. Flinn for his helpful discussions and contributions in this study.

References