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High powered microwave treatment of carbonate copper ore

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ABSTRACT

The effect of microwave radiation on the grindability of Palabora ore has been investigated. Significant reductions in comparative Bond work index (kWh/t) were exhibited in microwave treated samples. The results were sensitive to applied power level and the mode of application of energy. Exposure at 2.6kW in a multimode cavity was found to reduce the relative work index but the energy expended to get this change was high. Experiments were also carried out using a Sairem 15kW industrial monomode microwave unit. With a microwave exposure time of 0.28 second at 10kW, a 61.3% reduction in Bond work index was obtained, compared to 0.78kWh/t of energy expended due to microwave pretreatment, which gives a more favourable energy balance. Finally, Quantitative Evaluation of Materials using Scanning Electron Microscopy (QEM*SEM) was employed to determine if any liberation at the grain boundaries and evidence of intergranular fracture could be observed. There was evidence of an increase in liberation of copper sulphide and iron oxide in the coarser size fractions for Palabora ore.

Keywords: Microwave energy; Copper ore; Grindability; Bond work index

1. INTRODUCTION

Microwave energy has been used in industrial processing for many years. The most peculiar characteristic of microwave heating derives from the fact that the electromagnetic field penetrates the material thus heating it from the inside to outside. The main advantages of microwave heating are (Bonometti *et al.*, 1998):

1) quick start up and stopping
2) material selective heating
3) uniform heat distribution
4) rapid heating rates
5) volumetric heating

Comminution is a very energy intensive operation. Thermally assisted comminution reduces the energy required for subsequent grinding by generating thermal stresses that can cause weaknesses in the mineral matrix (Fitzgibbon and Veasey, 1990).

Grindability is a measure of a material's inherent resistance to comminution. In ore investigations it is often necessary to determine the grinding characteristics of a material so that a mill of the correct size, with adequate power, can be selected for plant operation (Deniz *et al.*, 1996). Bond's third law is used extensively in mineral processing to determine the power requirement of a given comminution device thus is useful for the design and selection of crushing and grinding equipment (Bond, 1961).

2. MATERIAL AND METHOD

Palabora is situated in the north east part of South Africa in close proximity to the Kruger National Park and was formed in 1956 as a joint venture between Rio Tinto and Newmont Mining Corporation.

The Palabora Igneous Complex can be described as a "figure of eight" layout approximately 8 kilometres from north to south and 3.2 kilometres from east to west (PMC Ltd, 1998).

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Approximately 80,000 tonnes of ore is processed daily, 26,000 tonnes being milled in the autogenous circuit and the balance in the conventional circuit. The primary crushers crush the ore to 100% passing 180mm which is fed directly to the autogenous mills at an average rate of 600t/h and discharged at 80% passing 300μm. In the conventional circuit the primary crushing product passes through secondary and tertiary crushers before being fed to the rod and ball mills, with the final product being 80% passing 300μm. The products from the two grinding circuits are pumped to the flotation plant where a copper concentrate is produced ready for the smelting process.

Slimes are removed from the flotation tailings by hydroseparators prior to the magnetic fraction being removed by drum separators. The magnetic concentrate is then upgraded and sold for use as a dense medium. The non-magnetic fraction is cycloned, the overflow is pumped to tailings and the underflow is the feed to the heavy minerals plant. The heavy minerals, containing uranium and zirconium, are separated from the tailings by gravity separation. The concentrate is upgraded chemically to produce pure oxides of uranium and zirconium. The low phosphate tailings that remain are pumped into the tailings dams. The high phosphate tailings are pumped to a nearby phosphate recovery plant, for the recovery of phosphate concentrates (Henderson, 1990).

The mineralogical investigation, which was carried out in conjunction with Rio Tinto Technology Development, Ltd (1998), showed that the head sample contained approximately 25% magnetite and 1% copper sulphide such as chalcocite (Cu_2S), bornite (Cu_5FeS_4), chalcopyrite ($CuFeS_2$), cubanite ($CuFe_2S_3$), valleriite ($4(Fe,Cu)S.3(Mg,Al)(OH)_2$) and pyrite (FeS_2).

The head sample is dominated by the presence of significant amounts of transparent gangue. Qualitative SEM analysis of the transparent gangue minerals showed that they consist mainly of calcite ($CaCO_3$), dolomite ($CaMg(CO_3)_2$), apatite ($Ca_5(PO_4)_3(F, Cl, OH)$), olivine ($(Fe, Mg)_2SiO_4$), phlogopite ($KMg_3AlSi_3O_{10}(F, OH)_2$), serpentine ($Mg_3Si_2O_5(OH)_4$), pyroxene and plagioclase feldspar. Minor amounts of brucite ($Mg(OH)_2$) were also present.

3. EXPERIMENTAL

Kingman (1999) determined the Bond work index of Palabora ore to be 13.06kWh/t. The method suggested by Berry and Bruce (1996) was used to determine the relative grindability. The comparative method is used for estimating the work index for a material of the unknown ore based on the work index of the reference ore. In order to determine the work index of an unknown ore it must be assumed that the work required (W) will be the same. Using Bond's third theory (Bond, 1952), the comparative method of determining grindability can be written as follows (Berry and Bruce, 1966) and is shown in Equation 1:

$$W_{iu}\left(\frac{10}{\sqrt{P_u}}-\frac{10}{\sqrt{F_u}}\right)=W_{ir}\left(\frac{10}{\sqrt{P_r}}-\frac{10}{\sqrt{F_r}}\right) \tag{1}$$

where
W_i is the work index (kWh/t).
u and r refer to the unknown and reference ores respectively.
P and F represent the 80% passing size of the product and the feed streams receptively.

3.1. Procedure for comparative tests using a microwave oven

A Panasonic II 2600 multimode microwave oven was used. The microwave reactor could be operated at power levels of 0.65kW, 1.3kW and 2.6kW and at a frequency of 2.45GHz. Representative 1kg samples of ore having an identical size distribution were irradiated at a fixed power level of 2.6kW. Irradiation times of 10, 30 and 60 seconds were used. All samples were irradiated in a 14.7×8.5cm fused silica dish, the position of which was kept constant in the microwave cavity. All microwave treated samples were quenched in water and were then dried overnight at a temperature of 65°C. A non-treated sample was also subjected to grinding to provide base line data.

Size distributions were obtained for each sample before and after treatment to provide evidence of any possible effect of microwave treatment. All size distributions were obtained by using a set of sieves ranging from +19mm down to -45μm by root two progression. Ore is ground at Palabora to approximately 80% passing 300μm for further processing.

The 1kg samples of ore were ground in a steel rod mill with dimensions of 28×15.8cm. Five steel rods mills with dimensions of 26.5×2.5cm were contained in the mill. The mill was run at 80% of the critical speed, which is in this case 90rpm.

This was found to take 35min total grinding in the above mill. All grinding tests were therefore carried for this time period with size distribution being determined after 5, 15 and 35 minutes. The 80% passing size obtained after 5 minutes grinding was used for comparison purposes as the differential grinding action of the rod gave similar size distributions after the total grinding period. Comparative Bond work indices were calculated using Equation 1. Comparative Bond work indices of samples were calculated using the 80% passing sizes obtained at 0 and 5 minutes milling time.

3.2. Procedure for comparative tests using an industrial monomode microwave unit

A Sairem 15kW industrial monomode microwave unit was used for two varied tests. The microwave reactor could be operated at power levels from 3 - 15kW and at a frequency of 2.45GHz.

In the first instance dolerite was removed from the representative 1kg samples of ore having an identical size distribution in order to see how sensitive the Palabora ore is to microwave treatment. Samples were irradiated in a monomode cavity at power levels of 10, 13, 14 and 15kW. A fixed irradiation time of 0.5 second was used for each power level. In a monomode system the microwave energy is applied in a more direct and efficient manner. The results obtained gave the baseline data for the second set of tests of ore with dolerite present, which represents the real effect of the actual material on the plant.

In the second instance the representative 1kg samples of ore having an identical size distribution were irradiated at power levels of 5 and 10kW. Irradiation times of 0.28, 0.54, and 1.38 seconds were used for each power level.

All samples were irradiated in a silica receptacle. All microwave treated samples were quenched in water and were then dried overnight at a temperature of 65°C. A non-treated sample was also subjected to grinding to provide base line data. The same grinding procedure as described previously was then carried out.

4. RESULTS AND DISCUSSION

Figure 1 shows the change in comparative Bond work index versus microwave exposure time for ore treated at 2.6kW.

It can be observed that microwave treatment has had a significant effect on the comparative Bond work index after 5 minutes milling. It can be seen that there is a reduction from 13.06kWh/t to 6.94kWh/t after 10 seconds irradiation. As exposure time was increased to 30 seconds treatment a further decrease in the comparative Bond work index from 13.06kWh/t to 4.56kWh/t was noted. Samples exposed to microwave treatment of 60 seconds and above showed some evidence of oxidation. Even though at an exposure time of 60 seconds a decrease in the comparative Bond work index from 13.06kWh/t to 5.6kWh/t was noted, however there was a slight increase in the comparative Bond work index compared to the 30 seconds. This may be due to experimental error or some sintering of sulphide particles due to overheating at hot spots.

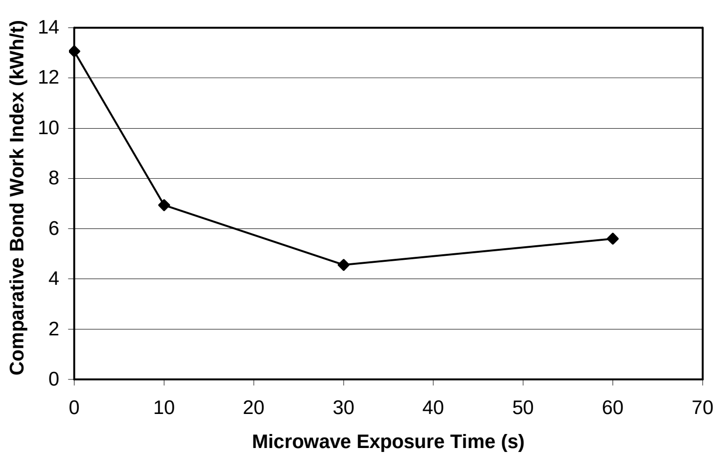

Figure 1. Plot of comparative Bond work index (kWh/t) *versus* microwave exposure time (s) 2.6kW (multimode)

The reduction in the comparative Bond work index after 5 minutes milling for the microwave treated samples is due to the thermal stresses generated that can cause weaknesses in the mineral matrix. All minerals respond differently to the application of the dielectric fields. Some mineral phases will absorb microwave energy and heat rapidly whereas others will be transparent to microwave energy, which will cause differential expansion within the mineral matrix. This expansion causes fracture around the grain boundaries, which will help reduce the grinding energy and give rise to an increase in recovery due to the liberation of the whole mineral grains.

Table 1 shows the energy balance required to obtain a reduction in Bond work index. For example, for material treated for 30 seconds at 2.6kW an energy reduction in comparative Bond work index of 8.5kWh/t is obtained. However, the energy expended (kWh/t) due to microwave pretreatment is 21.6kWh/t, therefore the energy balance does not work for this particular process.

The energy expended is greater than the energy reduction in comparative Bond work index that is obtained due to the inefficient multimode microwave heating at a power level of 2.6kW. However, at higher power levels using a monomodal unit better heating rates would be expected therefore experiments were carried out using a Sairem 15kW industrial monomode microwave unit.

Table 1
Energy balance for microwave heating 2.6kW (multimode unit)

Microwave Exposure Time (s)	Power Level (kW)	% Reduction in Bond Work Index	Reduction in Bond Work Index (kWh/t)	Energy Expended (kWh/t)
0	0	0	0	0
10	2.6	46.9	6.12	7.2
30	2.6	65.1	8.5	21.6
60	2.6	57.2	7.46	43.3

Figure 2 shows the change in comparative Bond work index versus power level for ore irradiated at 0.5 second in the Sairem high power microwave unit.

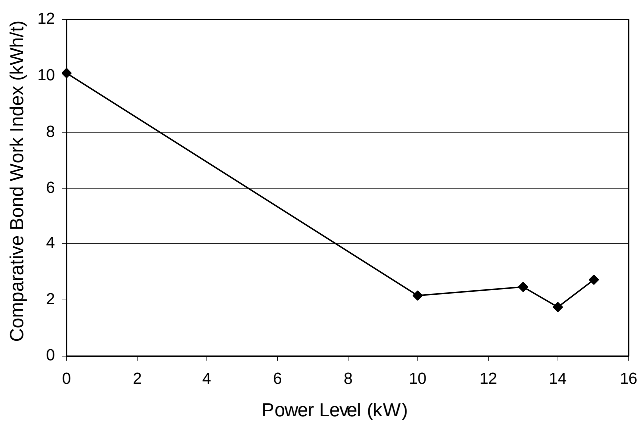

Figure 2. Plot of comparative Bond work index (kWh/t) versus power level (kW) 0.5 second (monomode) for Palabora ore with dolerite removed

Dolerite causes specific problems in the grinding circuit at Palabora. Dolerite, with its high work index of 30kWh/t, does not grind at the same rate as the rest of the ore, which results in building up a high circulating load in the circuit. For the trials on the Palabora ore with dolerite removed, it was not possible to perform a full Bond work index test due to sample quantity limitations. Therefore, based upon plant data (Henderson, 1990) and analysis of the feed material, the dolerite content was determined to be 20%. Upon removal of dolerite a re-calculated work index of 10.1kWh/t was obtained and used in Figure 2.

It can be seen from Figure 2 that there is a significant embrittlement on the ore at all power levels. This is accentuated due to the removal of dolerite from the 1kg samples.

For example, for a non-treated sample a value of work index of 10.1kWh/t is obtained as compared to a value of 2.18kWh/t for material treated for 0.5 second microwave exposure at 10kW.

Table 2 shows the energy balance required to obtain a reduction in Bond work index. It can be seen from Table 2 that, compared to the negligible increase in energy expended (kWh/t) due to microwave pretreatment, there is a significant decrease in the reduction in the comparative Bond work index (kWh/t) at all power levels. For example, for material treated for 0.5 second at 10kW a reduction of 7.9kWh/t in comparative Bond work index is achieved with an energy input due to microwave pretreatment of 1.4kWh/t. This results in a favourable energy balance of 6.5kWh/t.

Table 2
Energy balance for microwave heating 15kW (monomode unit)

Microwave Exposure Time (s)	Power Level (kW)	% Reduction in Bond Work Index	Reduction in Bond Work Index (kWh/t)	Energy Expended (kWh/t)
0	0	0	0	0
0.5	10	78.4	7.92	1.39
0.5	13	75.7	7.65	1.81
0.5	14	82.7	8.35	1.94
0.5	15	73	7.37	2.08

It can be seen from Table 2 that the optimum conditions equate to a microwave exposure time of 0.5 second at 10kW. Even though favourable results were obtained, however this is not a true representation of plant operation because dolerite has been removed by hand.

Figure 3 illustrates the effect of varying the microwave exposure time on the comparative Bond work index at different power levels for the Palabora ore with dolerite present. Compared to Figure 2, a smaller reduction in Bond work index can be seen, as dolerite is not affected by heat treatment.

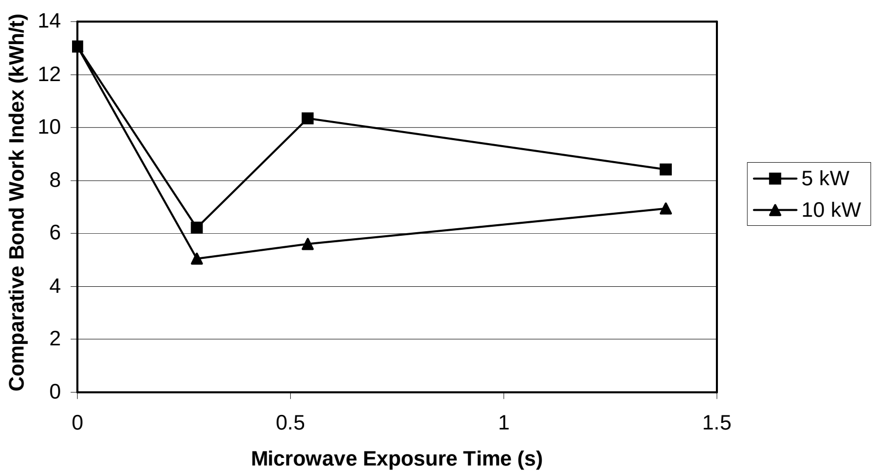

Figure 3. Plot of comparative Bond work index (kWh/t) versus microwave exposure time (s) (monomode) for Palabora ore (run-of-mine)

It can be seen from Figure 3 that for a non-treated sample a value of work index of 13.06kWh/t is obtained as compared to a value of 5.6kWh/t for material treated for 0.54 second at 10kW.
Moreover, the samples treated for 10kW show a more significant decrease in comparative Bond work index than those exposed to radiation of 5kW.

For example, for a sample treated for 0.28 second at 10kW, a value of work index of 5.05kWh/t is achieved. This can be compared to a value of 6.22kWh/t for the material treated at 5kW.

As mentioned previously, the percentage dolerite has a big effect on the overall work index of the ore. It is suggested that the reduction in embrittlement after 0.28 second microwave exposure time was caused by the sample inconsistency due to the dolerite being present. The presence of an extra particle of dolerite, which is a difficult material to process, can significantly affect the comparative bond work index. However the overall trends show significant reduction in the comparative bond index across all heat treated samples thereby making the microwave embrittlement process an attractive commercial proposition.

As energy is being added to the process the duration of microwave exposure must be optimised. Table 3 shows the energy balance for microwave heating. It can be seen from Table 3 that compared to the negligible increase in energy expended (kWh/t) due to microwave pretreatment there is a significant decrease in the reduction in the comparative Bond work index (kWh/t) at all microwave exposure times and power levels. For example, for material treated for 0.28 second at 5kW the energy expended (kWh/t) due to microwave pretreatment is 0.39kWh/t as compared to a reduction in comparative Bond work index of 6.8kWh/t.

It can be seen from Table 3 that the optimum conditions equate to a microwave exposure time of 0.28 second at 10kW. This makes the microwave treatment a potentially commercial viable process providing that it does not have a negative effect on the downstream processes such as flotation.

Table 3
Energy balance for microwave heating 15kW (monomode unit)

Microwave Exposure Time (s)	Power Level (kW)	% Reduction in Bond Work Index	Reduction in Bond Work Index (kWh/t)	Energy Expended (kWh/t)
0	0	0	0	0
0.28	5	52.4	6.84	0.389
0.54	5	20.8	2.71	1.75
1.38	5	35.6	4.65	1.92
0.28	10	61.3	8.01	0.78
0.54	10	57.2	7.46	1.5
1.38	10	46.9	6.12	3.83

5. QEM*SEM ANALYSIS

QEM*SEM is an acronym for "Quantitative Evaluation of Materials using Scanning Electron Microscopy" (Napier-Munn *et al.*, 1999). Two 1kg samples were manufactured to the same size distribution, with the particle size ranging from +500 down to -38 microns. One sample of ore was microwave treated in a Sairem 15kW industrial microwave unit. The sample was irradiated at a power level of 15kW for 0.1 second. Immediately after microwave treatment the sample was quenched in water at room temperature. The other sample was not treated to provide baseline data.

Upon their arrival at the Rio Tinto Technical Services Laboratory, each of the samples was screened into four size fractions (+500, -500+150, -150+38, and -38μm). A small amount was kept unscreened as a head sample. A small representative portion of each size fraction was mounted in chlorinated epoxy resin, polished and carbon coated for analysis by QEM*SEM.

Particle settling in the epoxy is an issue for heavy minerals especially in the coarser size fractions. Generally it is recommended that 10 particles layers be removed from the sample block in order to eliminate settling issues. Obviously this is not practical for fractions where the particle size is of the order of 0.5mm. Thus the modal abundances reported below in Table 4 have been normalised to the chemical assays.

Table 4
Normalised modal abundances

Mineral	% Mineral Per Sample	
Cu-sulphide	1.93	2.66
Pyrite	0.71	0.02
Galena	0.01	0.01
Sphalerite	0.01	0.03
Other sulphides	0.01	0.02
Fe-oxide	13.61	13.86
NSG	77.41	78.88
Other	6.31	4.52
	100	100

The size fractions were also submitted for quantitative mineralogical characterisation. Figures 4 and 5 show the percentage of liberated copper sulphide in each size fraction.

It can be seen from Figure 5 that microwave treatment of Palabora ore improves the liberation of copper sulphides particularly in the +500μm fraction. For a microwave exposure of 0.1 second at 15kW, a value of 68.2% of liberated copper sulphide is obtained in the +500μm fraction. This is compared to a value of 30.8% of liberated copper sulphide for a non-treated sample. This can be seen in Figure 4. Thus there is a significant decrease in the locked and middling material.

Figures 6 and 7 show the percentage of liberated iron oxide in each size fraction.

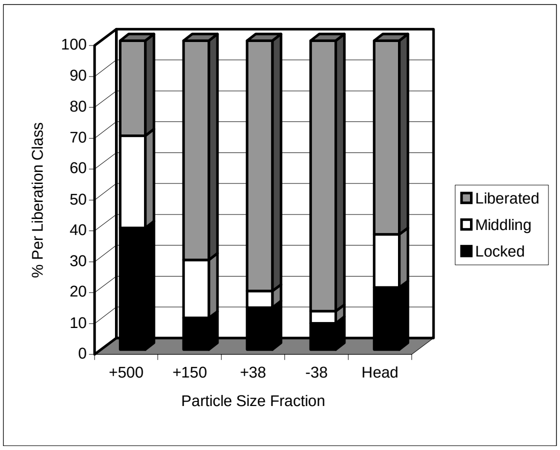

Figure 4. Cu-sulphide distribution of non-treated Palabora ore

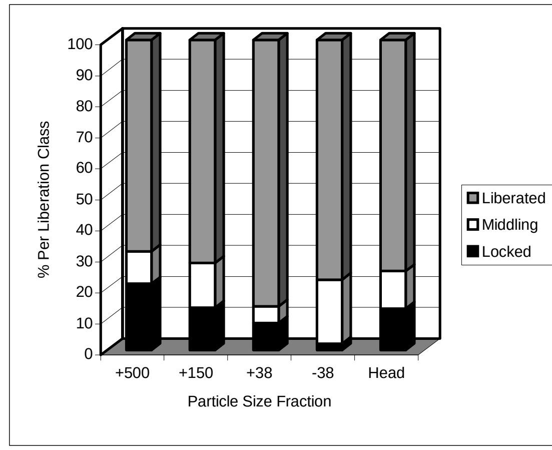

Figure 5. Cu-sulphide distribution of Palabora ore irradiated at a power level of 15kW for 0.1 second

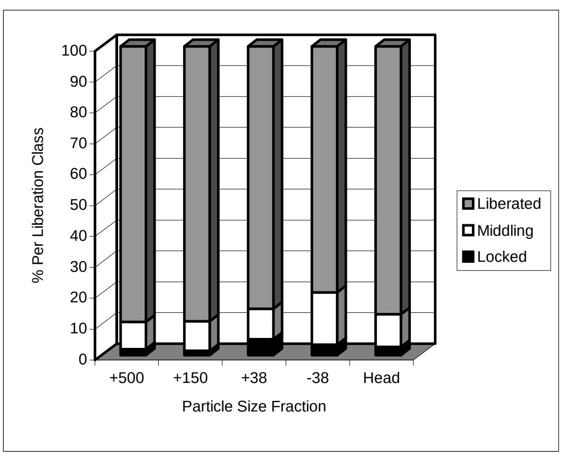

Figure 6. Fe-oxide distribution of non-treated Palabora ore

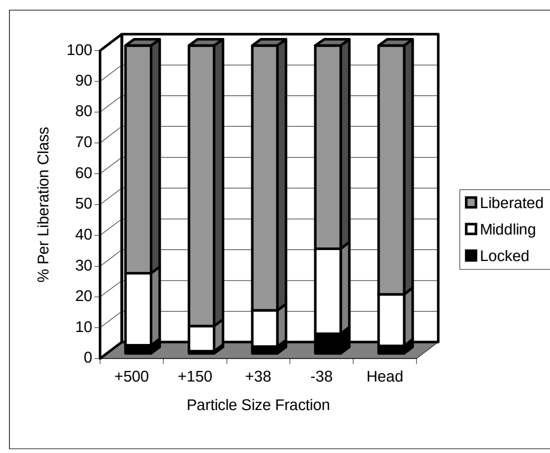

Figure 7. Fe-oxide distribution of Palabora ore irradiated at a power level of 15kW for 0.1 second

It can be seen from Figure 7 that microwave treatment of Palabora ore improves the liberation of iron oxides as a general trend. For a microwave exposure of 0.1 second at 15kW, a value of 91% of liberated iron oxide is obtained in the -500+150µm fraction. This is compared to a value of 89% of liberated iron oxide for a non-treated sample.

6. CONCLUSIONS

Reductions in the comparative Bond work index for the microwave treated samples is mainly due to the thermal stresses generated that can cause weaknesses in the mineral matrix.

Comparative Bond work index tests were carried out at a power level of 2.6kW in a multimode cavity. There was some evidence of embrittlement due to the differential expansion at the mineral lattice. Reductions in the comparative Bond work index in the region of 47-65% were achieved (reductions of 6-8.5kWh/t), however the energy expended to get this change was considerably higher (7-43kWh/t). Because the power absorbed is exponentially proportional to the microwave power level applied, utilising higher power levels and a monomodal unit provides more efficient heating.

Experiments were then carried out using a Sairem 15kW industrial monomode microwave unit. In the first instance dolerite was removed from the ore in order to determine the effect of microwave treatment on the actual sulphide minerals. Significant reductions in comparative Bond work index were obtained in the order of 73-83% at an energy input of 1.3-2kWh/t.

In the second instance as received run-of-mine Palabora ore was used and the optimum conditions equate to a microwave exposure time of 0.28 second at 10kW. A 61.3% reduction in Bond work index was obtained as compared to 0.78kWh/t of energy expended due to microwave pretreatment which gives a favourable energy balance.

Finally QEM*SEM was employed to determine if any liberation at the grain boundaries and evidence of intergranular fracture can be observed. There was evidence of an increase in liberation of copper sulphide and iron oxide in the coarser size fractions for Palabora ore. The quantitative mineralogical characterisation is given in Figures 4 to 7. In the +500 microns size fraction a value of 30% of liberated copper sulphide is obtained for a non-treated sample as compared to a value of 70% of liberated copper sulphide after microwave treatment.

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