The influence of microwave pre-treatment on copper flotation

by C. Sahyoun*, N.A. Rowson*, S.W. Kingman†, L. Groves†, and S.M. Bradshaw‡

Synopsis

The influence of microwave treatment on copper flotation has been investigated. Comparative batch flotation tests carried out on ore treated at 5–12 kW for 0.1–0.5 s showed that improvements in copper recovery of between 6–15 % could be achieved compared with untreated ore. Both recovery and cumulative grade increased with increasing treatment time and power. A preliminary economic analysis showed that microwave treatment will be economically viable if recoveries similar to those determined on laboratory scale can be achieved on a plant.

Keywords: microwave treatment, copper ore, flotation, liberation, recovery

Introduction

The principal objectives of flotation are to achieve as high a grade and recovery of valuable minerals as possible. This is clearly aided by the liberation of minerals at a size which is as coarse as possible within the limits of flotability. It is well accepted that the efficiency of froth flotation processes is significantly reduced at fine sizes (King, 2001). However, as resources of high grade orebodies decrease, the production of finer flotation feeds becomes ever more problematic as increasingly refractory and finely disseminated orebodies are processed.

Unfortunately, standard grinding technologies such as rod, ball, AG and SAG mills have been shown to produce only random fracture in particles of untreated ore. Therefore no control can be exercised over the amount of intergranular and phase boundary fracture (between grain boundaries and phases) compared with the amount of transgranular fracture (fracture across grain boundaries). Phase boundary fracture is highly desirable as it would enable the recovery of whole mineral particles at coarser sizes, thus making the separation processes, typically froth flotation, more efficient. This would increase the grind size required for liberation and therefore reduce the loss of unrecoverable material in slimes. It is therefore likely that if a method could be found to induce fracture at phase boundaries it would improve the recovery of valuable metals in a process flow sheet.

It has been suggested for a number of years that microwave pre-treatment can induce phase boundary fracture (see e.g. Walkiewicz, 1988, Whittles *et al.*, 2003 and Kingman *et al.*, 2004). Significant amounts of work have been carried out to investigate the impact of such pre-treatments on both the comminution behaviour and downstream recovery of minerals. Massive Norwegian ilmenite ores exposed to microwave radiation for varying times showed reductions in Bond Work index of up to 90% (Kingman *et al.* 1999). They quantified the influence of the microwave treatment on downstream magnetic separation and showed increased recovery of ilmenite in comparison with untreated material. High power treatment for short exposure times was found to be most effective because over exposure of the sample led to reductions in downstream processing efficiency.

This work was extended to investigate the reasons for increases in recovery of valuable mineral after microwave treatment (Kingman and Rowson 2000). They suggested that increased recovery was not only due to improved liberation but also to improved magnetic properties of the material. Unfortunately, while this work has shown promising technical benefits, preliminary economic analysis has shown it to be commercially unviable due to the amount of microwave energy required to achieve the process benefits.

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A very recent paper by Kingman *et al.* (2004) has shown, for the very first time, that microwave treatment of ores may be economically possible. This work showed that significant savings in comminution energy could be achieved, but with microwave energy inputs of less than 1 $kWht^{-1}$, by optimization of the method of application of the microwave energy to the sample. It was also suggested that the recovery of valuable minerals may be enhanced due to the occurrence of phase boundary fracture as a result of rapid differential heating and that increases in recovery would provide the real economic driver for the technology. The work concluded that pilot-scale testwork was required to demonstrate the benefits and, more importantly, to assess the economics of microwave assisted ore processing at scale.

The objective of this study was to investigate the first of these issues, namely the improvement in recovery due to microwave treatment. To this end, the influences of microwave treatment time and power on the batch flotation of a copper carbonatite ore were investigated at economic microwave energy inputs. As a secondary objective of the work, a preliminary economic analysis has been performed to assess the economic viability of the process.

Experimental procedure

Ore characterization

A South African copper carbonatite ore was used for all testwork. The mineralogy and texture of the ore had been quantified previously and reported (Kingman *et al.* 2000 and Kingman *et al*, 2004). The principal copper-bearing minerals within the ore were found to be chalcopyrite, bornite, chalcocite and cuprite. The secondary ore mineral was principally magnetite and the major gangue minerals were found to be calcite and dolerite.

Microwave treatment

Nine representative 1 kg samples of plant rod mill feed were used for all tests (100% passing 22 mm). A Sairem 15 kW single mode industrial microwave unit was used for treatment. The microwave unit could be operated at power levels from 3 to 15 kW and at a frequency of 2.45 GHz. Details of the equipment can be found in Kingman *et al.*, 2004. Samples were treated at power levels of 5, 7.5, 10 and 12 kW. Exposure times of 0.1 and 0.5 seconds were used for each power level. These combinations correspond to a range of microwave energy inputs between 0.14 and 1.67 kWh/t.

All samples were treated in the apparatus shown in Figure 1, the residence time in the microwave field being controlled by the retraction stroke of the pneumatic piston. After treatment, the representative 1 kg samples of ore were ground in a steel rod mill, with dimensions of 28 x 15.8 cm, to 80% passing 300 microns. Five steel rods with dimensions of 26.5 x 2.5 cm were contained within the mill.

Flotation procedure

450 g of each sample with a particle size range from –355 +45 microns were used for the flotation experiments. All flotation tests were carried out using a standard 3 litre Denver laboratory flotation cell. The impeller speed was kept

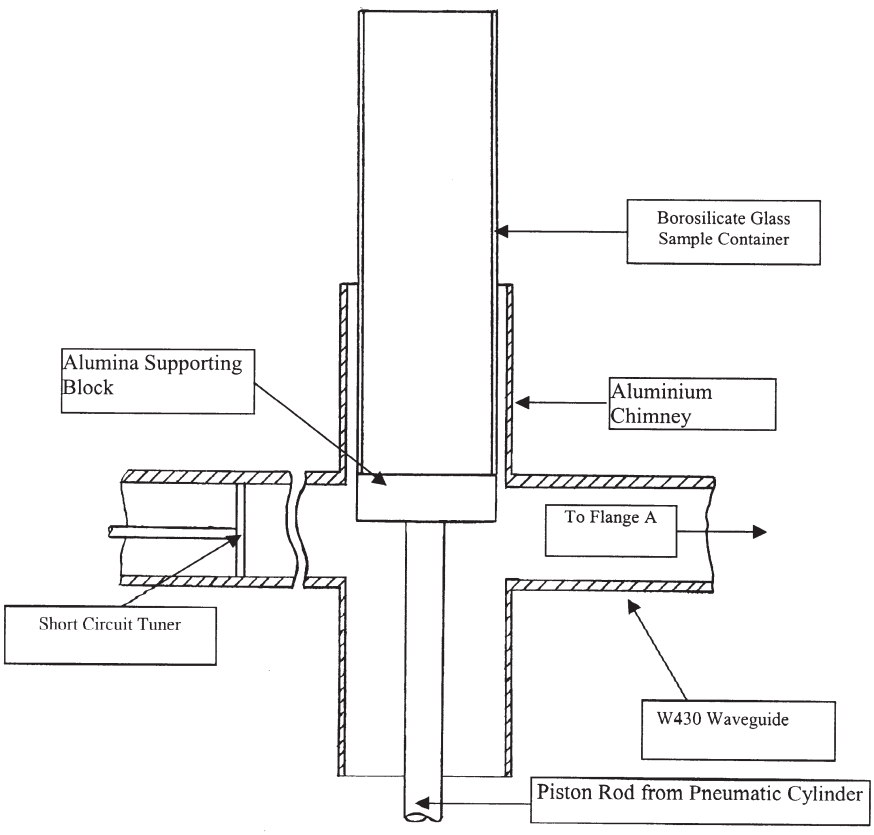

Figure 1—Sketch of arrangement for tests in single mode cavity

constant at 1500 rpm. Sodium iso butyl xanthate (SIBX) was used as a collector and was added at a dosage of 40 g/t. Senfroth® was used to stabilize the froth and two drops were added. The average grade of the samples was found by acid dissolution and then atomic absorption to be 0.35% copper.

The flotation procedure used was as follows:

The ore was added to the 3 litre flotation cell and 2.5 litres of water added. The machine was switched on and the ore mixed without reagent addition for a period of 2 minutes.

The collector was added and a further 2 minutes were allowed for conditioning the pulp. The frother was then added and 30 seconds of further conditioning were allowed. The air was then turned on fully and scraping of the froth was continued for 1 minute. The collecting tray was then changed and flotation continued for a further 1 minute. Again the collecting tray was changed and flotation continued for a further 3 minutes. The air was turned off and 40 g/t of collector was added again for scavenging and a further 2 minutes allowed for conditioning. Further frother was also added and 30 seconds was allowed for frother conditioning. The air was again turned on fully and flotation was then continued for 7 minutes. After flotation, each concentrate was pressure filtered and dried overnight at a temperature of 65°C. Copper analysis was done in duplicate for each concentrate and tails using acid dissolution and atomic absorption spectroscopy, and the mean of the two readings is reported.

Results and discussion

Figures 2–5 show cumulative grade recovery curves for the different treatment conditions. Complete data are given in Table I. Figures 2–5 show that, for all power levels used, the copper recovery at 1 min is higher than for the untreated ore. This result is statistically significant at the 99% confidence level. Figure 2 indicates that for a non-treated sample a

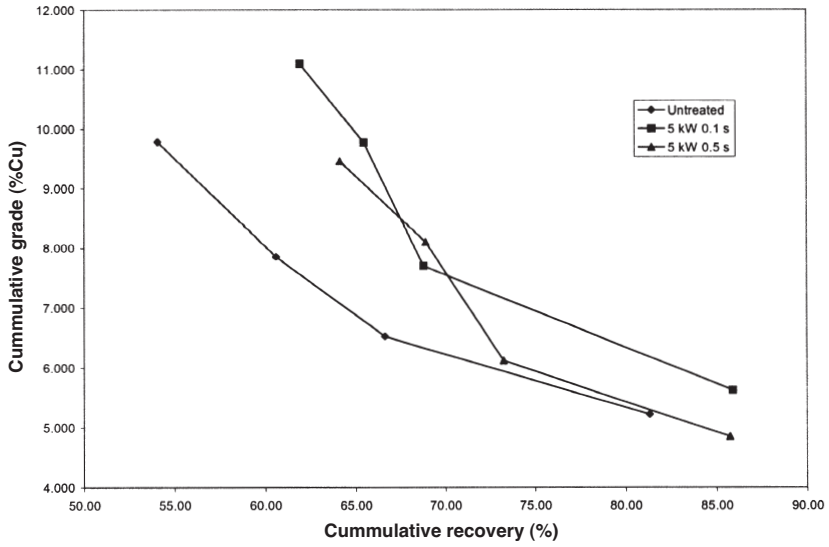

Figure 2—Grade-recovery curves for ore treated at 5 kW

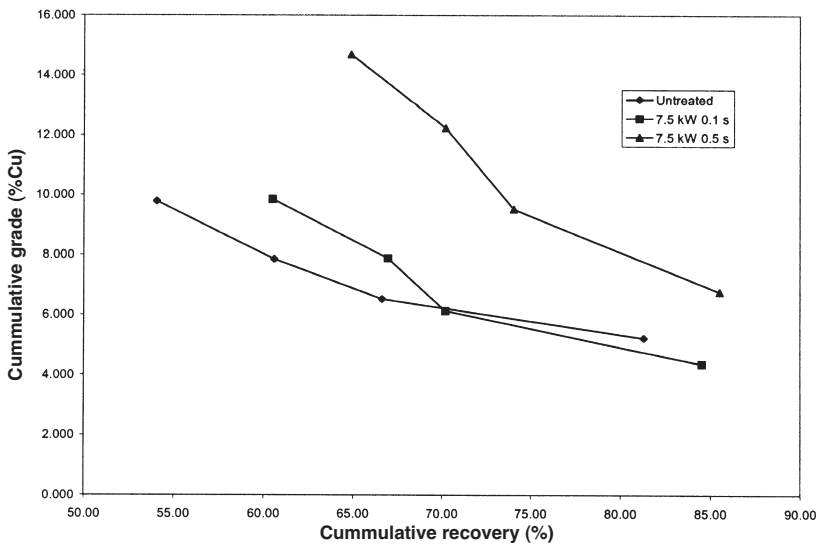

Figure 3—Grade-recovery curves for ore treated at 7.5 kW

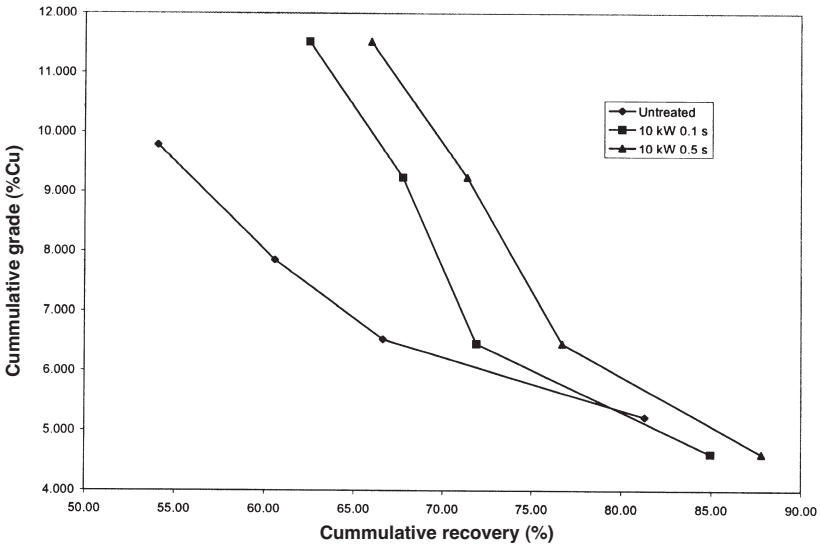

Figure 4—Grade-recovery curves for ore treated at 10 kW

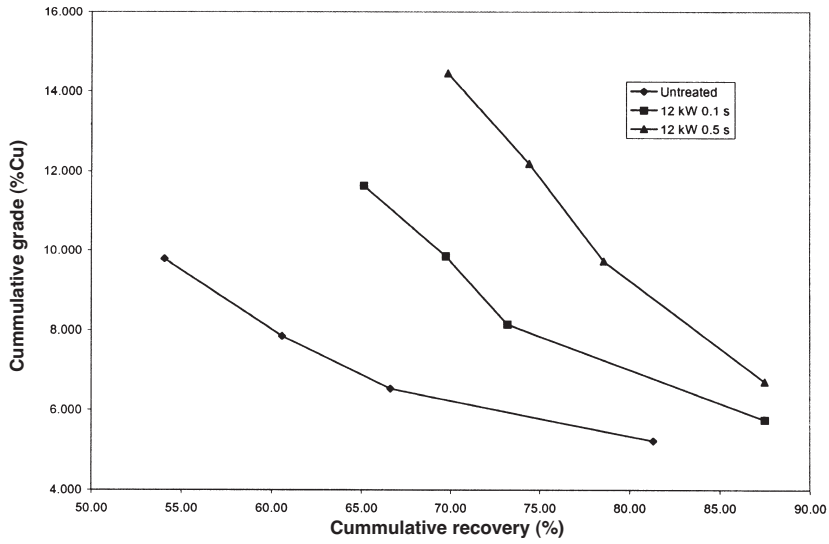

Figure 5—Grade-recovery curves for ore treated at 12 kW

copper recovery of 54% is obtained compared with a copper recovery of 61.9% for ore treated for 0.1 second at 5 kW. After 0.5 second microwave exposure, a copper recovery of 64.1% is achieved. It can be seen that the final cumulative recovery is higher for treated ore, while the final cumulative grade is similar. The same trends can be seen in Figure 3, although the difference between ore treated for 0.1 s and untreated material is small. For treatment at 10 kW, the initial recovery is significantly higher, the final cumulative recovery higher and the final cumulative grade slightly lower than for untreated material. The difference between treatment times is also more marked in this case. Figure 5 shows that copper recovery for the non-treated sample after 1 min flotation was 54% compared with 65.1% for ore treated for 0.1 second at 12 kW. For 0.5 second microwave exposure, a copper recovery of 69.9% is achieved. The final cumulative recoveries and grades for treated material are significantly higher than for untreated material.

Following the development from Figures 2–5 it can be seen that the increasing power and treatment time both improve the grade and recovery achieved, with the best result being that achieved at 12 kW for 0.5 s. When considering the initial recovery, for a given flotation time of 1 min, recovery increases with both increasing microwave power and with increasing microwave treatment time. These results are statistically significant at the 95% level. These results indicate that the microwave exposure has not caused unwanted oxidation products. It is known that SIBX will not form stable products with oxidized copper compounds, which would have caused recovery to fall had oxidation taken place.

The increases in copper recovery and grade are almost certainly due to increases in the amount of liberated material in the coarser size fractions of the treated ore. This makes the flotation process more efficient and thus gives rise to the increases in recovery. An increase in the amount of liberated material can be explained by the microwave induced failure

Table I

Grade-recovery data for batch flotation tests

Treatment	Product	Reagent 0.1% SIBX (ml)	Cumulative float time (min)	Cummulative grade (%Cu)	Cumulative % Cu recovery
Non-microwaved	Conc. 1	18	1	9.785	54.06
	Conc. 2		2	7.852	60.58
	Conc. 3		5	6.527	66.62
	Conc. 4	18	12	5.224	81.31
	Tails			5.290	100.00
5 kW monomode cavity microwaved for 0.1	Conc. 1	18	1	11.092	61.90
	Conc. 2		2	9.773	65.44
	Conc. 3		5	7.703	68.78
	Conc. 4	18	12	5.630	85.87
	Tails			5.694	100.00
5 kW monomode cavity microwaved for 0.5	Conc. 1	18	1	9.455	64.12
	Conc. 2		2	8.096	68.90
	Conc. 3		5	6.120	73.24
	Conc. 4	18	12	4.847	85.73
	Tails			4.899	100.00
7.5 kW monomode cavity microwaved for 0.1	Conc. 1	18	1	9.844	60.48
	Conc. 2		2	7.881	66.95
	Conc. 3		5	6.128	70.19
	Conc. 4	18	12	4.352	84.52
	Tails			4.401	100.00
7.5 kW monomode cavity microwaved for 0.5	Conc. 1	18	1	14.690	64.88
	Conc. 2		2	12.228	70.20
	Conc. 3		5	9.512	74.04
	Conc. 4	18	12	6.764	85.50
	Tails			6.826	100.00
10 kW monomode cavity microwaved for 0.1	Conc. 1	18	1	11.518	62.48
	Conc. 2		2	9.238	67.72
	Conc. 3		5	6.452	71.89
	Conc. 4	18	12	4.617	84.95
	Tails			4.666	100.00
10 kW monomode cavity microwaved for 0.5	Conc. 1	18	1	12.169	65.94
	Conc. 2		2	10.296	71.36
	Conc. 3		5	8.115	76.70
	Conc. 4	18	12	5.617	87.78
	Tails			5.664	100.00
12 kW monomode cavity microwaved for 0.1	Conc. 1	18	1	11.631	65.13
	Conc. 2		2	9.855	69.74
	Conc. 3		5	8.147	73.19
	Conc. 4	18	12	5.755	87.47
	Tails			5.804	100.00
12 kW monomode cavity microwaved for 0.5	Conc. 1	18	1	14.456	69.85
	Conc. 2		2	12.191	74.40
	Conc. 3		5	9.733	78.58
	Conc. 4	18	12	6.712	87.48
	Tails			6.759	100.00

mechanism. Whittles *et al.* (2004) showed that high power densities, such as those produced by single mode cavities, could produce extremely rapid localized heating within microwave responsive phases. If the heated phase were contained within a microwave transparent matrix, the heated phase would be restrained as it expanded. This creates hoop stresses that are tangential to the direction of the initial expansion, i.e. at the phase boundary. This weakens the ore and effectively allows for liberation of the absorbing particles (the mineral values) during subsequent milling. Evidence to support this theory is found in Table II. This shows results of QEM*SEM analysis on the material broken in a drop weight test at an energy input of 1 kWh/t. The original size of the material was -22 +19 mm. From the data in the table it can be seen that +500 μm size fraction showed over 100% more material classified as liberated for the treated sample when compared with the untreated. Further details can be found in Kingman *et al.*, 2003.

Inspection of the data in Table I shows how the grade of concentrate varies with microwave power level and exposure time. Microwaved treated samples show a statistically significant (98% confidence level) increase in the grade of the initial concentrate (i.e. after a flotation time of 1 min) compared with untreated material. Almost certainly the increase in grade after 1 min of flotation seen in the treated samples is due to the promotion of phase boundary fracture within the samples. It is expected that significant amounts of phase boundary fracture give rise to liberation by detachment (King 2001). This occurs when minerals are loosely bound to the ore matrix. During the comminution process (in this case the rod milling) mineral grains become detached, leading to significant and clean liberation of the copper mineral phase. This has the effect of increasing the grade of the concentrate. To examine this further, SEM analyses were done on representative samples of both untreated and treated feed ore. Figures 6a and 6b show SEM images of treated and untreated material. The presence of phase boundary fracture (which could lead to liberation by detachment during milling to flotation feed size) between the sulphide (light grey) and host (darker grey) phases can be clearly seen.

Table II

Liberation data for untreated and treated ore

	Untreated material					Treated material				
	+500 μm	+150 μm	+38 μm	-38 μm	Head	+500 μm	+150 μm	+38 μm	-38 μm	Head
PSD	32.22	31.06	21.37	15.35	100.00	31.91	31.46	21.86	14.77	100.00
Locked	39.40	10.20	13.60	8.50	20.07	21.40	13.70	8.70	2.10	13.35
Middling	29.80	18.80	5.30	3.90	17.17	10.40	14.40	5.40	20.60	12.07
Liberated	30.80	71.00	81.10	87.60	62.75	68.20	71.90	85.90	77.30	74.58
	100	100	100	100	100	100	100	100	100	100

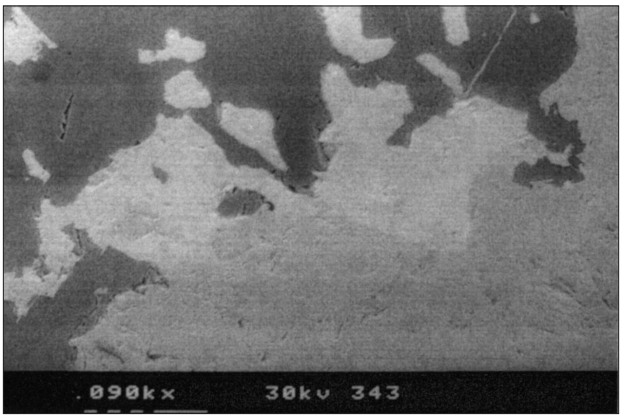

Figure 6a—SEM photomicrographs illustrating nature of untreated ore

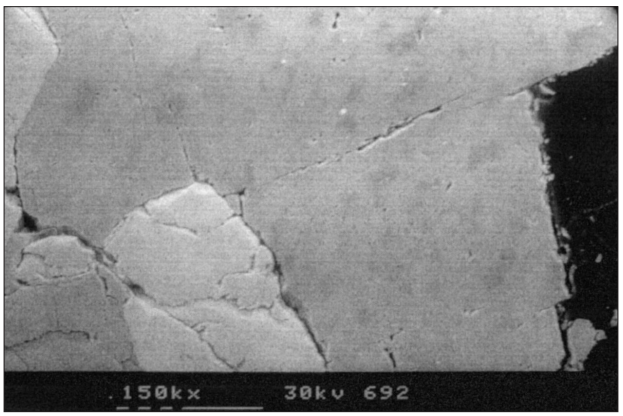

Figure 6b—SEM photomicrographs illustrating phase boundary fracture in microwave treated samples (12kW, 0.5 seconds)

A preliminary economic analysis for a number of copper mines is shown in Table III, based on increased recovery due to microwave treatment. Throughputs reported in the table are based on published operating data for the mines. In order to assess the economic potential for microwave treatment, the increased annual profit for each of the mines was calculated by assuming that the recovery of copper could be increased by microwave treatment and that the increase would lead directly to increased revenue. The increase in annual profit was determined by subtracting an appropriate electricity cost and tube replacement cost. No account was taken of taxes, depreciation, etc. Profitability is given in terms of payback period calculated as the capital cost of the equipment divided by the annual profit as defined above. Note that payback time is a rather crude profitability measure, but one that is sufficiently accurate, given the assumptions made elsewhere in the analysis.

It was assumed that the entire plant throughput would be treated in the microwave unit(s). This is not always the case and is dependent on the grinding circuit of the mine. Microwave treatment would be applied only to the rod or ball mill feed, as the size of the AG/SAG mill feed is too large for reasonable microwave operation. It is also possible that the technology could be applied in regrind circuits. For the purpose of this simplified analysis, this option was not considered.

Based on electromagnetic simulations (unreported here for confidentiality reasons), it was assumed that the applicator would be either a tunnel with moving bed, or a vertically oriented cylindrical applicator. The bed voidage was assumed to be 0.5 and the mass fraction of absorbing phase was assumed to be 10%, which is typical for many of the ores that would be amenable to treatment. It was assumed that the gangue was totally transparent and, although this is not the case, it is a reasonable first approximation. The cross-sectional area of the treatment zone was assumed to be 60% of the tunnel cross-section for the appropriate frequency band, while the length was assumed to be equal to the broad dimension of the waveguide. These dimensions are in accordance with those been found to give acceptable processing uniformity in electromagnetic simulations.

From experiment and thermal stress simulations, it is known that there is a minimum power density for any given ore, below which the strength reduction is small and the process benefits similarly poor. It is has also been established by thermal stress simulations that the degree of damage done to a 2-phase simulated ore is directly related to the energy density in the absorbing phase, once above the critical power density threshold just mentioned. Depending on the degree of damage required to achieve a processing benefit (which will be ore and process specific), this value could be approximated as 10^8 Jm^{-3} of absorbing phase. In the economic analysis this value was used as a threshold for acceptable performance.

In assessing the economic benefits of the technology, it has been assumed that microwave treatment successfully achieves phase boundary fracture at the power and energy densities described above.

The data in Table III show results of the analysis for a number of copper mines. Further assumptions may be found in the table. It has been assumed that operation would be done at 433 MHz, as the lower frequency would allow larger

Table III

Payback times for microwave equipment based on increased recovery

Mine	Number of units required	Increased copper production (tpy)	Increased annual revenue US ($M/y)	Annual operating cost for units ($M/y)	Increased annual profit ($M/y)	Payback time (months)
Large mine A	5	313 147	36.82	0.88	35.93	16.7
Small mine B	1	68 460	8.05	0.15	7.90	15.2
Large mine C	6	715 909	84.17	1.02	83.15	8.7

Notes
(a) Residence time 0.1 s
(b) Recovery increased from 80 to 84%
(c) Increased recovery assumed to give equivalent increased metal production
(d) Operating costs include electricity at $0.035 /kWh (South African cost)
(e) Tube life 10 000 h
(f) Tube replacement cost £30 000
(g) Cost assumed to be $20 000 /kW

equipment and greater throughput. Note that 500 kW magnetrons are not available commercially at 433 MHz. However, development of such equipment is feasible should this be required. Because of this, a high capital cost estimate has been used. To put this value into perspective, 915 MHz equipment costs approximately $1000/kW. Total installed equipment cost, including the microwave applicator can be up to $7000/kW, depending on the nature of the plant. Note that there are no economies of scale associated with microwave generators. Thus a total installed cost of $20 000/kW has been assumed as a likely upper bound for equipment operating at 433 MHz. Throughput has been calculated based on cross-sectional treatment area, power requirement and residence time. Bed speeds have been kept below 1 ms^{-1} to approximate reasonable operating practice.

Based on the experimental data obtained in this work, it has been assumed that a 4% increase in recovery can be achieved, and that plants have an existing 80% recovery. No reduction in costs as a result of decreases in grinding energy requirements were included in this analysis, as the intention in this work was to highlight the benefits of treatment on recovery. Although it is clear from the results obtained to date that reductions in grinding or crushing energy can be significant, this preliminary analysis has focused on the effect of improved recovery. For all 3 mines considered, it can be seen that the payback times are less than 2 years. This emphasizes the tremendous benefits that could be realized through improved recovery as a result of microwave treatment. In a worst case scenario, not shown in Table III, it was assumed that the increase in recovery would be only 0.5%. In this situation the range of payback times for the mines above would be 7–19 years. Note once again that this assumes no reduction in grinding/crushing costs.

Conclusions

Batch flotation tests on a copper carbonatite ore showed that pre-treatment of rod mill feed with microwave energy at economically viable levels has a significant influence on the flotation of the ore. Initial recovery was found to be higher in the treated samples for all power levels and exposure times. At lower power levels, the cumulative grade of the treated material after 12 min of flotation was similar to that of untreated material. At the highest power level used (12 kW for 1 kg of ore) both grade and recovery were significantly improved for microwave treatments time of 0.1 and 0.5 s. The recovery was shown to be sensitive to applied power level and duration of microwave exposure. A simplified economic analysis showed that the recoveries reported in this work are economically attractive.

Acknowledgements

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South Africa celebrates 100 years of safe mine hoisting*

In the face of growing health and safety challenges, stringent government regulations and rapid transformation, the local mining industry has one success record that it can look back on with pride. 2004 marked 100 years of hoist rope testing in South Africa, which was celebrated at a centenary event held at the Cottesloe testing facility of CSIR Mining Technology on Friday 26 November 2004.

Vishnu Pillay, Director of CSIR Mining Technology, explains that as depths increase, along with the demand for bigger and heavier payloads, rope testing is an increasingly important part of mine safety. CSIR Mining Technology currently runs one of only two hoist rope testing facilities approved by the Department of Minerals and Energy (DME) in South Africa, at its Cottesloe laboratory in Johannesburg.

'The records of the Department of Minerals and Energy (DME) show more than 1 000 registered mines in South Africa, of which an estimated 135 are underground operations,' says Pillay. 'The CSIR's own rope database reflects more than 1 150 hoist ropes in use, illustrating the responsibility that rests with our laboratory to provide mine engineers with accurate test data and results.'

The statutory testing of mine hoist ropes was introduced following the failure of a winding rope in No. 2 shaft, Robinson Deep, on 25 April 1904. This accident caused the death of over 40 men and led to the immediate introduction of a government regulation that stipulated the periodic removal of a section of head rope adjacent to the skip, to be tested at the Government Mechanical Laboratory of the Department of Mines.

The Department of Mines relinquished control over the Government Mechanical Laboratory in 1964, when it was taken over by the CSIR and incorporated into its National Mechanical Engineering Research Institute. This laboratory now forms part of CSIR Mining Technology and continues to conduct statutory tests on mine hoist ropes and provide related mechanical testing services.

Hoist rope fact file

- South African mines hoist about 280 000 people up and down mine shafts each day
- The same mines hoist about 10 million tons of ore every month
- An eight-compartment, 2 000 m deep shaft contains more than 16 km of hoist rope
- In the deepest shaft each hoist rope is more than 3 000 m long
- DME regulations stipulate that ropes installed on licensed winders must be tested every six months
- Canada has adopted certain South African standards relating to rope discard criteria
- CSIR Mining Technology's hoisting and mechanical testing laboratory tests between 2 000 and 3 000 ropes every year, including hoist ropes, scraper ropes, dragline ropes and ropes for marine applications
- The biggest specimen the centre has ever tested is a 153 mm diameter steel wire rope, which was locally manufactured, and was to be used as one element of a sling for an engineering application
- The biggest steel wire rope used for mine hoisting in South Africa is 63 mm in diameter, and weighs 17 kg a metre. At a depth of more than 2 000 m, the weight of this rope alone—without any skip or cage attached—is some 34 tons
- Although mines sometimes experience winding accidents, they are rarely due to rope failure. Rope testing allows mines to predict and prevent such failure. Testing also keeps costs down, as it helps mines to avoid premature replacement of expensive steel wire rope
- CSIR Mining Technology maintains the largest database of rope tests in Africa, to ensure the traceability of all specimens tested.

CSIR mining technology's Cottesloe testing facility

The largest tensile test machine in use at Cottesloe is a 15 MN MFL machine that was commissioned in 1989. In addition to testing mine hoist ropes, the MFL machine is able to accommodate large diameter wire ropes (~160 mm) as used in the oil drilling, bridge and shipping industries.

The capacity of the 15 MN machine also caters for tensile tests on conveyor belts up to 1.2 m wide and large-link chains, such as those used on coalmine draglines.

The hoist rope tests evaluate a rope's absolute breaking force, mechanical properties, modes of wire and strand failure, extent of corrosive and abrasive damage, and the state of lubrication. If the results of the test show that the rope does not conform to the code of practice (SABS 0293, 1996), then the recommendation is that the rope be discarded.

The majority of ropes, however, only reach this condition following several years of service. The results of the test, their graphical history, and subsequent visual examination of the test specimen, are recorded on a test certificate supplied to the mine.

The smallest diameter hoist rope that CSIR Mining Technology tests is about 16 mm in diameter, and the largest is currently 63 mm. Peake says that in terms of hoist ropes, the predominant size ranges between 30 mm and 50 mm in diameter.

In addition to the testing of mine hoist ropes, CSIR Mining Technology offers a range of other mechanical tests including tensile, proof and compression tests of a broad range of mining and industrial products.

These include:

- a 4 448 kN Mohr and Federhaff tensile test machine with a 25 m bed
- an 8 896 kN Mohr and Federhaff compression test machine; a feature of this compression test machine is the 'daylight' opening of 4.5 m, permitting the testing of full-scale mine support systems such as packs and elongates and
- three Amsler universal testing machines, with capacities of 100 kN, 500 kN and 1 000 kN.

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