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EFFECTS OF MICROWAVE RADIATION UPON THE MINERALOGY AND MAGNETIC PROCESSING OF A MASSIVE NORWEGIAN ILMENITE ORE

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The effect of microwave radiation upon the mineralogy and magnetic processing of a massive Norwegian ilmenite ore is presented. Short exposure to microwave radiation has been demonstrated to cause fractures within the ore matrix. Increased exposure to microwave radiation is shown to cause localised sample melting. The microwave treated samples have subsequently undergone a multi-stage magnetic separation process which produced concentrates of significantly higher grade and also better recovery of valuable mineral, when compared to those that are nontreated. Conclusions are made regarding further development and implementation of this technology.

Keywords: Microwave radiation; Mineralogy; Magnetic separation; Magnetisation; Ilmenite

INTRODUCTION

Microwaves are a form of electromagnetic energy with associated electric and magnetic fields. When microwave energy is applied to the material, the dipoles align and flip around, since the applied field is alternating. As a consequence, the material will be heated as the stored internal energy is lost to friction. This *in-situ* mode of energy conversion has the advantage of being selective to individual mineral phases within a mass [1]. Conventional heating has the disadvantage that the total

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mass of material is heated and the radiation is absorbed into the material by conduction. Overheating and wasteful heating of insulators can result. These problems are alleviated with microwave radiation since this form of energy selectively heats individual phases within a material lattice, [2] creating differential heating at the grain boundaries which may lead to embrittlement of the material and also better liberation of mineral grains.

Various factors influence the dielectric properties of a material (or the ability of a material to absorb or generate heat). These include the frequency of the applied field, the temperature and the physical properties of the material. The most significant effects arise due to particular physical properties of the material in question, such as the chemical composition, the water content, the particle size and also the crystallography.

A variety of applications for microwave radiation in the mineral processing and extractive metallurgical industries have been proposed [3–9]. These cover a wide area of interest, however, the fundamental principle behind all of these applications remains the ability of microwaves to heat individual phases within a mineral matrix. Coupled with these proposed applications, studies have been conducted concerning the fundamental response of minerals to microwave radiation [10,11]. Both studies concluded that the majority of silicates, carbonates and sulphates were transparent to the microwave radiation, however, most sulphides, arsenides, sulphosalts and sulphoarsenides heated strongly were emitting fumes and fusing. Results of a quantitative study by Walkiewicz [11] are presented in Table I.

Another important observation made during this research was that rapid heating of ore minerals in a nonheating gangue generated thermal stresses which produced flaws at discrete locations within the matrix, effectively causing embrittlment. A further study of the effect of applied

TABLE I Summary of mineral heating rates (after Walkiewicz, 1988)

Mineral	*Chemical composition*	*Max temp. achieved* (°C)	*Time* (min)
Chalcopyrite	$CuFeS_2$	920	1
Galena	PbS	956	7
Magnetite	Fe_3O_4	1258	2.75
Orthoclase	$KAlSiO_3O_8$	67	6
Pyrite	FeS_2	1019	6.75
Quartz	SiO_2	79	7
Sphalerite	ZnS	88	7

power level on mineral heating rate was performed [12]. Similar minerals to those shown in Table I were powdered and treated at various applied power levels from 500 to 2000 W. In general, an increase in power level led to an increase in heating rate (°C/s). Low loss dielectric minerals such as plagioclase feldspar, quartz and orthoclase feldspar exhibited no significant temperature rise at any applied power level.

The ore used for the current study was obtained from Titania A/S, a Mining company in the southern part of Norway. The deposit, which was discovered in 1954 contains 350 million tonnes assaying approximately 18% TiO_2 (or 39% ilmenite) and 2% magnetite. The deposit is mined by the open pit method. All processing being carried out on site. The main valuable mineral from the plant, ilmenite is used to produce titanium dioxide (TiO_2) which in turn is used in the production of paints, plastics paper and rubber. In addition to the main concentrate, magnetite is produced by a two-stage magnetic separation process. Copper, nickel and cobalt are also produced as by-products of the process. The complete plant flow sheet is shown in Fig. 1. The process flowsheet consists of standard mineral processing unit operations. The average through put of the plant is approximately 4.8 million tonnes from which 820 000 tonnes of ilmenite, 40 000 tonnes of magnetite and 12 000 tonnes of sulphides are produced.

EXPERIMENTAL PROCEDURE

(1) Mineralogical Investigation

Three representative samples of ore were prepared by core drilling of the bulk rock material from the mine. Each sample was in the form of a disc with a diameter of 19 mm and was approximately 3.5 mm thick. Two of the discs were irradiated within a variable power Panasonic 2.6 kW microwave source operating at 2.45 GHz. Discs being exposed for 30 and 60 s at both 1.3 and 2.6 kW, respectively. After irradiation, each sample was immediately quenched in water at ambient temperature. A nontreated sample was kept for comparison. After the appropriate treatment, each of the samples was mounted into epoxy resin and polished to 0.25 μm for microscopy and electron microprobe examination. A Buehler Ominmet Advantage image analysis system was used to illustrate textural features and relationships. Each of the polished

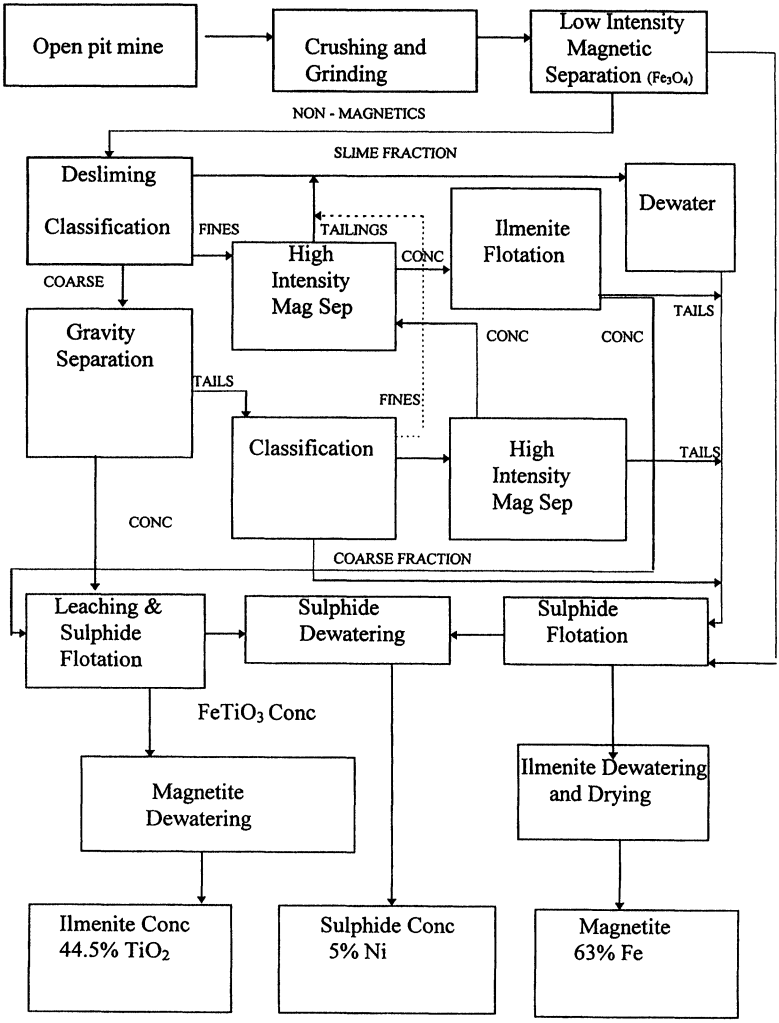

FIGURE 1 Tellnes ore beneficiation flowsheet.

samples were systematically examined and the minerals identified on the basis of their bulk chemistry.

(2) Determination of Magnetisation

Since the minerals contained within the ilmenite ore are separated individually it was decided to determine the effect of microwave

radiation on the individual mineral phases. An Oxford Instruments vibrating sample magnetometer was used to determine the magnetisation of each phase.

(3) Determination of Microwave Effects on Separation

Two hundred gram representative samples of ore were crushed to 100% passing 16 mm. Samples were exposed at the same applied power levels and for the 10, 30 and 60 s respectively. Three treated samples were used to produce a detailed record of the effects of the microwave radiation. After treatment each sample was ground until 100% passing 220 μm; the size used for magnetic separation on plant. A two-stage magnetic separation process was used in order to reproduce the plant flowsheet as accurately as was reasonably possible. A single pass Boxmag Rapid (BHW) high intensity wet magnetic separator was used for all tests, a wedge wire matrix being employed to reduce particle entrainment to a minimum. Magnetite was removed firstly from the sample utilising an applied field strength of 0.045 T. Ilmenite was then removed using an applied field strength of 1 T. This procedure is representative of current plant practice. The percentage of titanium in each sample was determined by colorimetry.

RESULTS

Mineralogical Investigation

Figure 2 shows an image of untreated ore. The untreated sample is holocrystalline and medium grained with the individual crystals ranging from between 0.5 and 3.0 mm in size. The ilmenite grains are disseminated throughout the specimen, however, they are more frequently present in the form of small granular aggregates. Ilmenite (white to lightest grey) grains are present both as polycrystalline aggregates or clusters as mentioned above. The grains are intergrown with larger grains of calcic plagioclase (dominant uniform dark grey areas). Small amounts of olivine are also present with these grains showing evidence of incipient serpentinisation (olivine changing to serpentine) and extensive fracturing (small areas, similar to the more abundant calcic plagioclase, but with numerous fractures). A small degree of fracturing

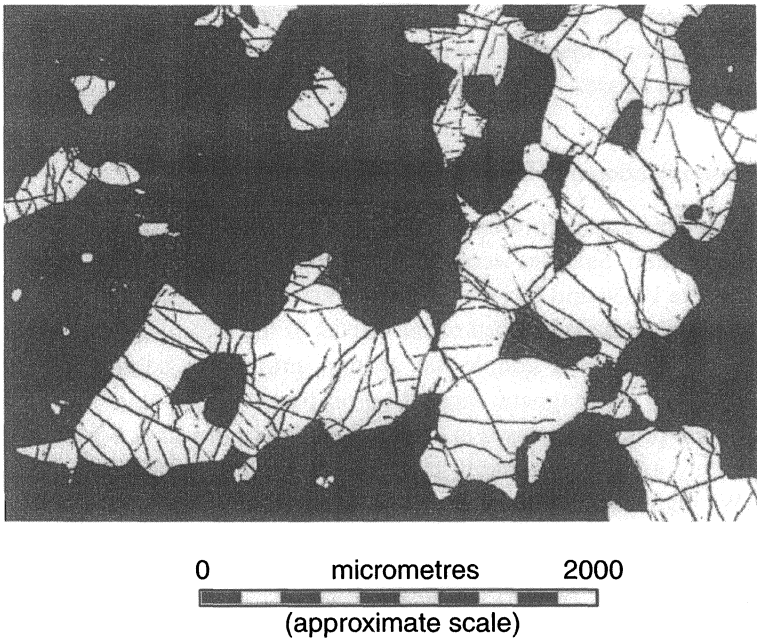

FIGURE 2 Untreated ilmenite ore.

can be observed in the ilmenite (black intersecting lines) and the calcic plagioclase. This fracturing is, however, less extensive than the fracturing in the partially serpentinised olivine and may, at least in part, be a result of volume changes within the olivine which are associated with the serpentinisation process.

Figure 3 shows a false colour computer enhanced electron backscatter image illustrating the nature and appearance of a small complex sulphide grain. The composition of the sulphide grains vary within the sample, but in most cases they consist of a certain amount of pyrite (light grey-brown) that is intergrown with one or more of chalcopyrite, cubanite and or millerite (yellow). Subordinate amounts of a discrete Co–Ni sulphide phase (blue) are also present. The Co : Ni ratios of this phase vary significantly and it may represent the mineral siegenite $(Ni\ Co)_3S_4$. Small amounts of fine grained, Mg-rich chlorite or clinochlore (dark green shades) are also present within the surrounding sulphide aggregates. These sulphide intergrowths are assumed to represent the decomposition products of former primary magmatic sulphide assemblages.

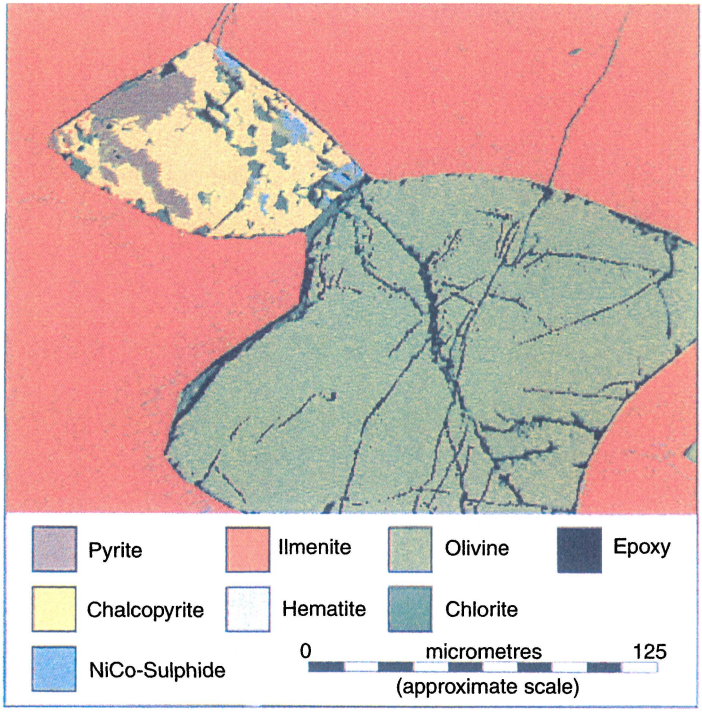

FIGURE 3 False colour electron backscatter image of untreated ore. (See color plate I.)

Figure 4 shows a digitised monochrome reflected light photomicrograph prepared using low magnification (× 60) to illustrate the overall nature and appearance of the sample. The sample shares many features with Fig. 2 but the overall degree of fracturing of both silicate minerals and ilmenite appears to be greater. Some evidence of increased serpentinisation can be observed. Cracking of both a transgranular and intergranular nature is apparent. Fracture in the silicate minerals may be caused by their dehydration as the temperature increases.

Figure 5 shows a digitised monochrome reflected light photomicrograph, again at a modest magnification (× 60) to illustrate the overall nature and appearance of the sample after treatment. Relative to the samples treated at 0 and 30 s, Fig. 5 is characterised by the presence of an increased amount of fracture, particularly in the silicate minerals and in the ilmenite. In addition to the increased amount of fracture, some cracks are wider and more pronounced.

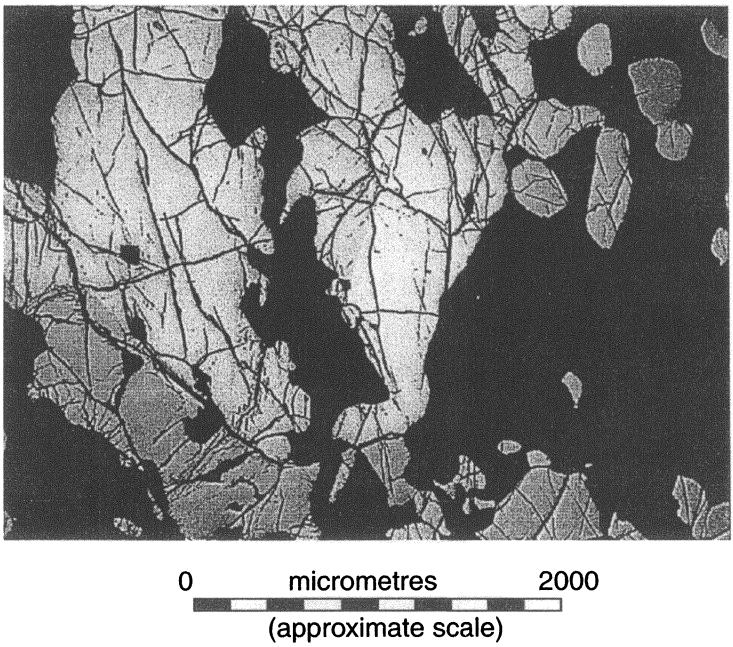

FIGURE 4 Ilmenite ore after 30 s microwave treatment.

Figure 6 shows a digitised monochrome reflected light photomicrograph, prepared using medium power magnification (× 250) to illustrate the overall nature and appearance of the sample in an area where partial melting was initiated. The partial melting has occurred between two ilmenite grains (lightest grey shades). The area of quenched partial melt is characterised by the development of numbers of rounded gas cavities (darkest grey) that are present within an aluminosilicate glass. This glass hosts large numbers of skeletal and elongated crystallites of ferian rutile (medium grey shade). Qualitative energy dispersive electron microprobe analyses of the aluminosilicate glass show that it contains subordinate, but nevertheless significant amounts of calcium and potassium in addition to lesser amounts of titanium and iron.

Figure 7 shows a false colour computer enhanced electron backscatter image illustrating the nature and appearance of a glass-rich area of the partial melt products. In this case an extensively melted grain of calcic plagioclase (dark blue) is separated from a partially melted residual ilmenite grain (large orange grain) by a prevalent area of

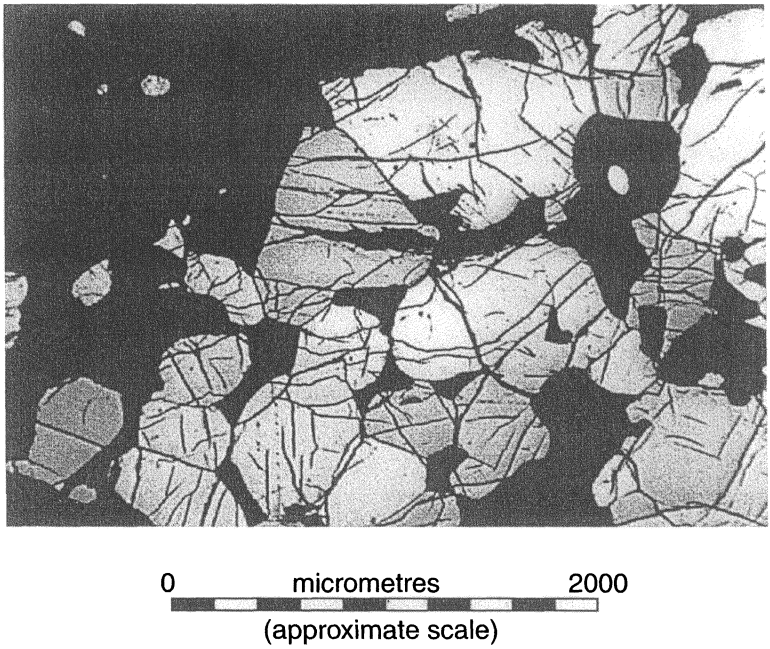

FIGURE 5 Ilmenite ore after 60 s microwave treatment.

aluminosilicate glass (red shades). The glass hosts typical ferrian rutile crystallites (elongated grey phases), but also shows the presence of numbers of smaller more equant grains. Qualitative energy dispersive electron microprobe analyses of these small crystallites show that they consist largely of titanium and iron. This phase is not ilmenite but is more likely to be a member of the pseudobrookite–ferropseudobrookite solid solution series (Fe_2TiO_5–Ti_2FeO_5).

This study has revealed the significant effect of microwave radiation upon the mineralogy of a massive Norwegian ilmenite ore. This is especially true at the longest exposure time of 60 s. However, after 30 s of treatment the effects of the radiation were still pronounced. Considerable fracture can be seen in both the ilmenite and the calcic plagioclase gangue. Exposure for 60 s revealed the most significant results. After being exposed for this time period, increased fracture can be seen in the ilmenite and calcic plagioclase phases compared with samples treated for 0 and 30 s, respectively. The amount of serpentinisation also appears

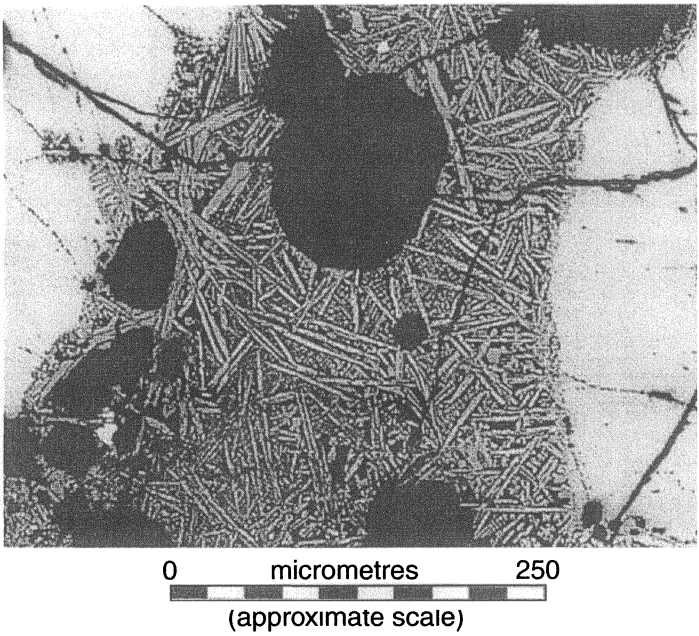

FIGURE 6 Ilmenite ore after 60 s microwave treatment.

to have increased. Further analysis of material treated for 60 s has revealed that partial melting had occurred in certain areas. This indicates that the temperatures reached in the matrix of the ore must have been quite considerable ($\sim$ 1100°C). While the significant increase in fracture after 60 s microwave treatment may give rise to significant reductions in Bond work index, this long exposure time and high temperature may prove to be detrimental to any further processing of the ore. It was observed that aluminosilicate glasses were being formed from the melting of the gangue minerals and that the ilmenite phases were decomposing to form members of the pseudobrookite–ferro-pseudobrookite solid solution series. Previous work [13] has shown that minerals in this category lack the properties that make beneficiation and utilisation an attractive proposition, especially in terms of magnetisation and ease of processing.

Microwave treatment of minerals has two main objectives: firstly to reduce the grinding energy required to mill an appropriate size and secondly to promote the formation of intergranular fracture, thus

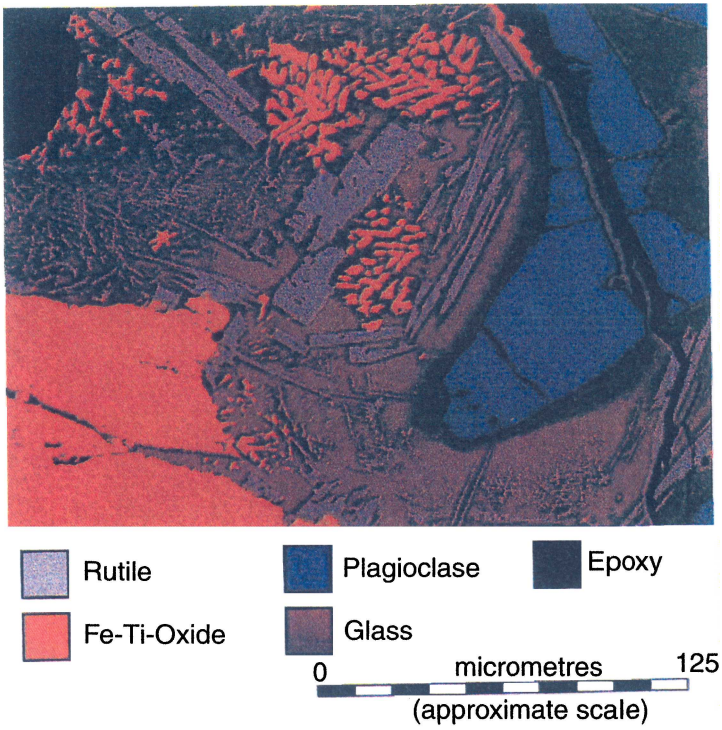

FIGURE 7 False colour electron backscatter image of ore treated 60 s. (See color plate II.)

increasing liberation of the valuable mineral. This study has revealed two likely mechanisms that could produce these objectives.

(1) *Serpentinisation* This is the changing of olivine to serpentine at high temperature. As exposure time increased the amount of fracture that occurred in the olivine increased. This would have the effect of weakening the lattice and reducing the cohesion of the macroscopic sample.

(2) *Differential expansion of minerals* Previous research [13] has indicated that different minerals heat in an applied microwave field at different rates. Different heating rates will necessarily give rise to different levels of volumetric expansion at the grain boundaries. This differential volumetric expansion in the ore matrix will lead to

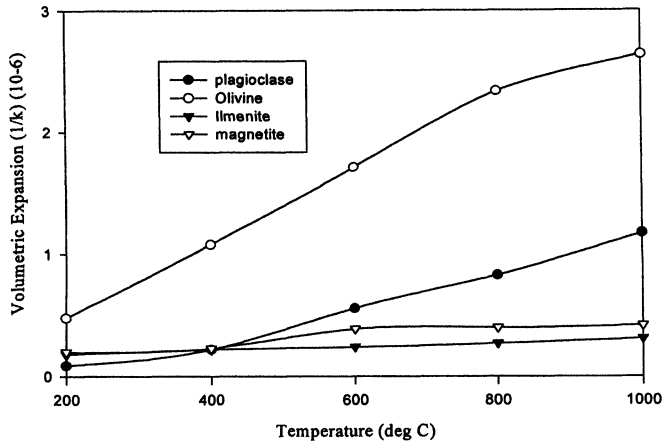

FIGURE 8 Volumetric expansion rates of ilmenite constituents.

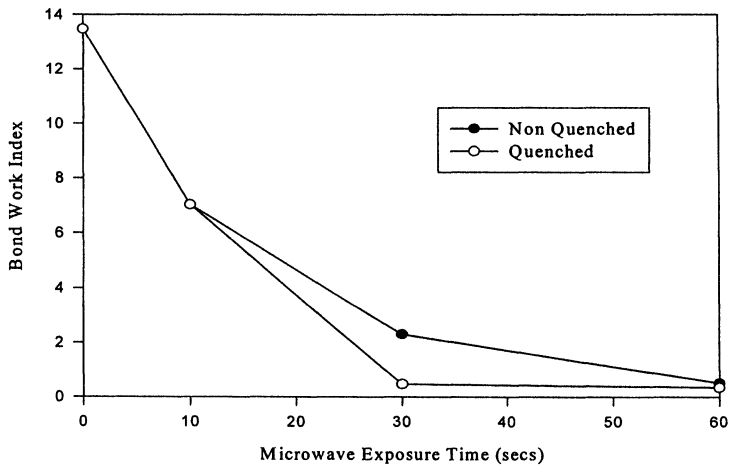

FIGURE 9 Effect of microwave radiation on the Bond work index of massive Norwegian ilmenite ore.

weakening, owing to the formation of intergranular and transgranular cracks and, therefore, possibilities for liberation of more whole mineral particles thus increasing grade of concentrate and recovery of valuable mineral. Figure 8 [14] shows the variation in volumetric expansion with temperature for the main constituents of massive Norwegian ilmenite ore.

It can clearly be seen that when a matrix containing the above minerals is heated large internal stress will occur. Therefore, for this material, significant increases in grade and recovery would be expected after microwave treatment.

Previous studies [15] have quantified the effects of microwave radiation upon the Bond work index of massive Norwegian Ilmenite ore. Samples were exposed to radiation of varying power levels for periods of 10, 30 and 60 s. Figure 9 shows results for material exposed to radiation at 2.6 kW and 2.45 GHz.

It can be clearly seen that microwave pre-treatment has had a significant effect on the Bond work index of this material. Further work, is at present, being carried out to optimise the presentation of the microwaves to the ore samples, therefore, reducing the energy input into the process. It is clear, however, the benefits of microwave treatment even at relatively short exposure times are significant.

Magnetisation

Figures 10 and 11 show the effects of microwave radiation on the valuable constituents of the massive ilmenite ore. It can clearly be seen that ilmenite (Fig. 10) shows an increase in magnetisation especially after 30 s treatment. After 60 s microwave treatment the susceptibility is similar to that of untreated ore. This can be explained by the material

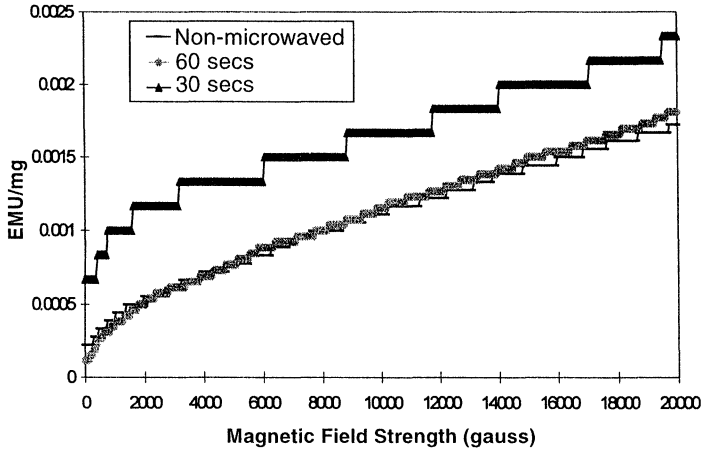

FIGURE 10 Magnetic response of ilmenite to microwave radiation (2.6 kW).

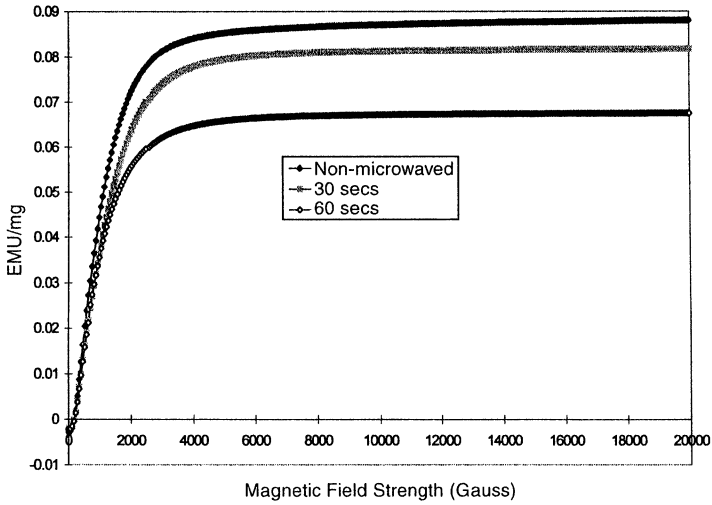

FIGURE 11 Magnetic response of magnetite to microwave radiation (2.6 kW).

treated for 30 s developing remanent magnetisation which shifts the curve up. Material treated for 60 s does not show this increase in magnetisation, this is due to formation of the aluminosilicate glass and parts of the pseudobrookite–ferropseudobrookite solid solution series (Fe_2TiO_5–Ti_2FeO_5). These materials have very poor magnetic properties.

Figure 11 shows significant reductions in the saturation magnetisation of magnetite as the microwave exposure time is increased. These reductions may be explained by the oxidation of magnetite to form hematite as the temperature is increased by the application of more energy. The temperatures required for this reaction are readily obtained within a microwave oven.

While the reductions in saturation magnetisation of magnetite are significant, it is important to note that they are still several orders of magnitude above those of ilmenite. This means that an effective separation can still be achieved. The separation of ilmenite will be enhanced due to the increase in remanent magnetisation.

Determination of Microwave Effects on Process Flowsheet

Figures 12 and 13 show the effect of microwave exposure time on the magnetic separation process for this ore. Each plotted point is the mean

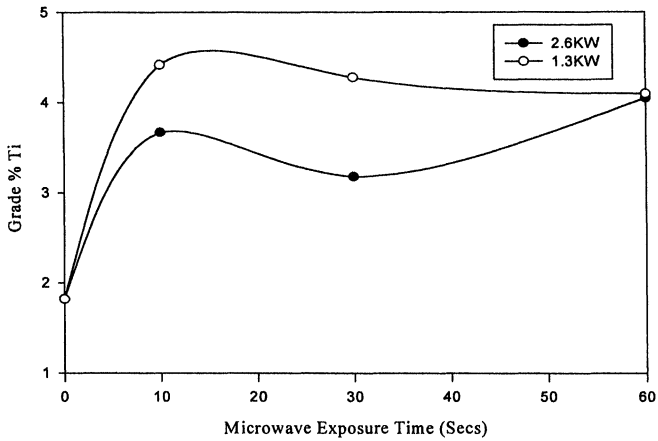

FIGURE 12 Effect of microwave radiation on concentrate grade.

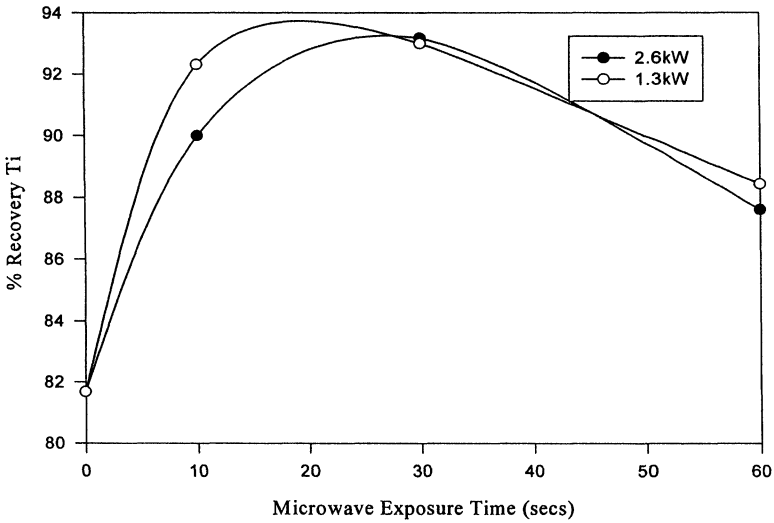

FIGURE 13 Effect of microwave radiation on titanium recovery.

of three experimental determinations. For each determination the percentage of titanium was determined twice.

It can be seen from Fig. 12 that microwave treatment considerably increases the grade of the ilmenite concentrates. This was especially true

for material exposed to radiation at 1.3 kW rather than that exposed to 2.6 kW. The probable explanation for this unexpected result is that the higher power radiation is causing the formation of the partial melt products shown in Figs. 6 and 7. These have a much lower magnetisation than that of the ilmenite and so they pass through the magnetic separator as gangue. This observation is confirmed when Fig. 13 is considered. As microwave exposure time is increased from 0 to 10 s a sharp increase in recovery of titanium is observed, however, as more energy is applied and exposure is increased recovery falls although still remains above that for nontreated material.

From the results obtained for both grade and recovery it is clear that to treat ore for 60 s has little benefit, and the most significant benefits occur during the first 10–20 s. To continue with irradiation after this time serves only to consume energy and to detrimentally effect the process economics.

CONCLUSIONS

The results of this study indicate that microwave radiation has a significant effect upon the mineralogy and magnetic processing of massive Norwegian ilmenite ore. It has been shown that short periods of exposure can cause fracture at grain boundaries which leads to the formation of intergranular fractures. This fracture coupled with an increase in remanent magnetisation of the ilmenite mineral has been demonstrated to give rise to an increase in both concentrate grade and valuable mineral recovery. However, the study has also indicated that process efficiency can be effected with over exposure to microwave radiation.

These benefits coupled with the decrease in Bond work index suggest that further investigation is warranted. As energy is being added to the process a detailed techno-economic analysis is required. This will form the next stage of this research together with further work on reducing the cost controlling factor of microwave exposure times. Points to consider are the use of higher powers for shorter times and pulsed delivery of radiation as both these methods will reduce the input of energy to the system overall and add support to the process economics.

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References

[1] Kelly, R.M. and Rowson, N.A. Microwave reduction of oxidised ilmenite concentrates, *Minerals Engineering*, **18**(11) (1995), 1427–1438.
[2] Mingos, D.M.P. Applications of microwave dielectric heating to problems in synthetic chemistry, *Chemistry Society Review*, No. 20 (1991), pp. 1–47.
[3] Worner, H.K. *et al.* Microwaves in pyrometallurgy, *Proc. First Aus. Symp. On Microwave Power Applications* (February 1989), pp. 179–188.
[4] Woodcock, J.T. Possibilities for use of microwave radiation in the processing of gold, *Proc. First Aus. Symp. On Microwave Power Applications* (February 1989) pp. 139–142.
[5] Haque, K.E. Microwave irradiation treatment of a refractory gold concentrate, *Proc. International Symposium on Gold Metallury*. Winnipeg, Canada (1986), pp. 327–339.
[6] Zavitsanos, P.D. Coal desulphurisation using microwave radiation, *US Patent* No: DOE.PC 30142-71 (1981).
[7] Rowson, N.A. and Rice, N.M. Desulphurisation of coal using low power microwave energy, *Minerals Engineering*, **3**(3/4) (1990) 363–368.
[8] Butcher, D.A. and Rowson, N.A. Microwave desulphurisation of coal, *I. Chem. E Research Event* **1** (1995), 583–585.
[9] Lyttle, J. *et al.* Influence of preheating on the grindability of coal, *Inte. J. Min. Proc.*, **36** (1992), 107–112.
[10] Chen, T.T. *et al.* Relative transparency of minerals to microwave radiation, *Canadian Metallurgical Quarterly* **23**(3) (1984), 349–351.
[11] Walkiewicz, J.W. *et al.* Microwave Heating Characteristics of Minerals and Compounds, *Miner. Metal. Proc.* (February 1988), 39–42.
[12] McGill, S.L. *et al.* The effect of power level on the microwave heating of minerals and chemicals, *Materials Research Society Proceedings* **124** (1988), 247–252.
[13] Harrison, P.C. Microwave processing of minerals and ores, Ph.D. Thesis University of Birmingham (1997).
[14] Clark, P.S. Handbook of physical constants, *Geol. Soc. of America*, No 97 (1966).
[15] Kingman, S.W. and Rowson, N.A. Applications of microwave radiation to enhance performance of mineral separation processes. *Richard Mozely Memorial Symposium*, IMM (1997).

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