THE EFFECT OF MICROWAVE RADIATION ON MINERAL PROCESSING

by

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Synopsis

Between 50% and 70% of the total energy used in the extraction process may be attributed to comminution. Microwave pre-treatment has been suggested as a means to decrease the energy requirements. A variety of mineral ores have been investigated and the effects of microwave radiation quantified in terms of the mineralogy, changes in the Bond Work Index, flotability and magnetic separation. It has been shown that microwave pre-treatment is most effective for coarse grained ores with consistent mineralogy consisting of good microwave absorbers in a transparent gangue (up to a 90% decrease in Bond work index for Palabora copper ore) whereas fine grained ores consisting predominantly of good absorbers are not affected as well (a reduction of only 25% in work index for Mambula ore). Although the mineralogy of minerals are affected by exposure to microwave radiation, flotability and magnetic separation characteristics have been shown not to be adversely affected, unless the microstructure is completely destroyed after prolonged microwave exposure. Computer simulations have shown that significant changes to comminution circuits are possible as a result of microwave induced work index reductions (three mills reduced to one). Purpose-built microwave units may hold the solution for more efficient mineral extraction in the near future.

Dedication

In memory of Mrs AC Booysen

Acknowledgements

- I would like to take this opportunity to thank and acknowledge the Almighty God for His support and help throughout my life and studies. Without Him and His Presence, this thesis would not have been possible.

- Secondly I would like to thank my parents for always being there and the opportunities they have given me.

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NOMENCLATURE

A	ore breakage parameter
A_s	sample area, m^2
b	ore breakage parameter
c	denotes the speed of light $and\ is \approx 3 \times 10^8 \frac{m}{s}$
C_p	is the specific heat capacity in $\frac{J}{kg\ °C}$
C_s	fraction of critical mill speed
D'	mill diameter in ft
$\bar{D}$	average size of particles, m
e	emmisivity of the material
E	is the root mean square intensity of the external electric field in $\frac{V}{m}$
E_0	is the root mean square intensity of the external electric field in $\frac{V}{m}$
E_i	is the root mean square intensity of the electric field inside the mineral in $\frac{V}{m}$
E_{cs}	is the specific comminution energy, kWh/t
f_0	is the resonant frequency, Hz
f	denotes the frequency in Hz
f_c	is the crushing strength, N

G	the grindability in $\frac{g}{rev}$
h_i	is the height from which the mass is dropped, cm
j	is the imaginary number $\sqrt{-1}$
k_R	is Rittinger's constant
k_K	is Kick's constant
k	is a constant
K_{ic}	is the mode I fracture toughness
m	material mass, kg
$\bar{m}$	mean particle mass, kg
M_d	mass of impact plate, kg
M_{-P1}	the mass of -P_1 material, g
N_{cc}	critical mill speed, rpm
N	mill speed, rpm
N_c	the number of revolutions
L	is the load in N
p	is the comminution exponent
P_1	is the recycle sieve size in the Bond test, micron
P_a	is the power absorbed by the material, W
P_d	is the power density dissipated in the heated material in $\frac{W}{m^3}$
P'	Load at primary failure, kg

P, F	is defined as the 80% passing sizes of the product and feed respectively
Q	is the energy in J
r_0	proportion of new oversize mill feed
R	is the reduction ratio, $R = \frac{F}{P}$
R_s,R_d, R_l	is the radius of the support circle, disc and load circle respectively
t	the time in s
t10	is the percentage passing a tenth of the input size
T	is the temperature of the heated material in °C
tan δ	is the loss tangent
V	is the volume in m^3
W	is the energy required, kWh/t
W_i	is the work index of the material.
V_s	sample volume, m^3
z	is the depth at which the field strength needs to be calculated in m
α	is the attenuating factor in m^{-1}
ϵ_0	is the permittivity of free space $and\ is\ = 8.85\ \times 10^{-12}$
$\hat{\epsilon}_r$	is the relative permittivity
$\epsilon_r^{/}$	is the relative dielectric factor
$\epsilon_r^{//}$	is the relative loss factor

$\hat{\epsilon}$ is the complex permittivity in $\frac{F}{m}$

$\epsilon_{eff}^{//}$ Is the effective permittivity, $\frac{F}{m}$

ϵ' is the real part of the complex permittivity and is called the dielectric or dispersion factor in $\frac{F}{m}$

ϵ" is the imaginary part of the complex permittivity and is called the loss factor in $\frac{F}{m}$

λ denotes the wavelength in m

ρ is the density of the material in $\frac{kg}{m^3}$

δX denotes a change in property X

ν Poison's ratio

σ equivalent conductivity, S/m

σ_{sb} Stefan-Bolzman constant, $(5.67051 \pm 0.00019) \times 10^{-8} \frac{W}{m^2K^4}$

σ_r is the stress in the radial direction, MPa

τ is the thickness in m

ω is the angular frequency

Subscripts

r,t refers to the reference and test ores respectively

CHAPTER 1

INTRODUCTION

1.1) Introduction

1.1.1) Prologue

Engineering may be defined as "the science by which the properties of matter and the sources of power in nature are made useful to humans in structure, devices, machines and products" (New York Public Library, 1995). This is especially well illustrated in the field of minerals engineering. Man is dependent on his environment for survival. Our environment supplies us with the basic requirements for life (oxygen, water, food and clothes) as well as the luxuries of civilization (computers, electricity and microwave ovens).

In contrast with the basic requirements, which, to a reasonable extent, are unchanged by human intervention (i.e. oxygen and water), the luxury items follow a longer route before ending up in our homes. Most of these materials have to be mined and undergo extensive physical and chemical modification before they are utilized. As an example, an everyday commodity, steel, is made from iron, which in turn is extracted from iron ore (magnetite/hematite) which requires huge mining operations to extract it from the earth's crust in the quantities required by the earth's growing population. In addition, modern technology requires steel with vastly superior characteristics than would have been satisfactory a few short decades, or even years, ago.

Valuable minerals do not, as a rule, occur freely in nature, but are frequently locked away within gangue particles. As an example, gold (yellow Figure 1.1) usually occurs in extremely low concentrations in nature and are finely dispersed within the host rock (mostly gangue) as is shown in the scanning electron micrograph (Fig 1.1).

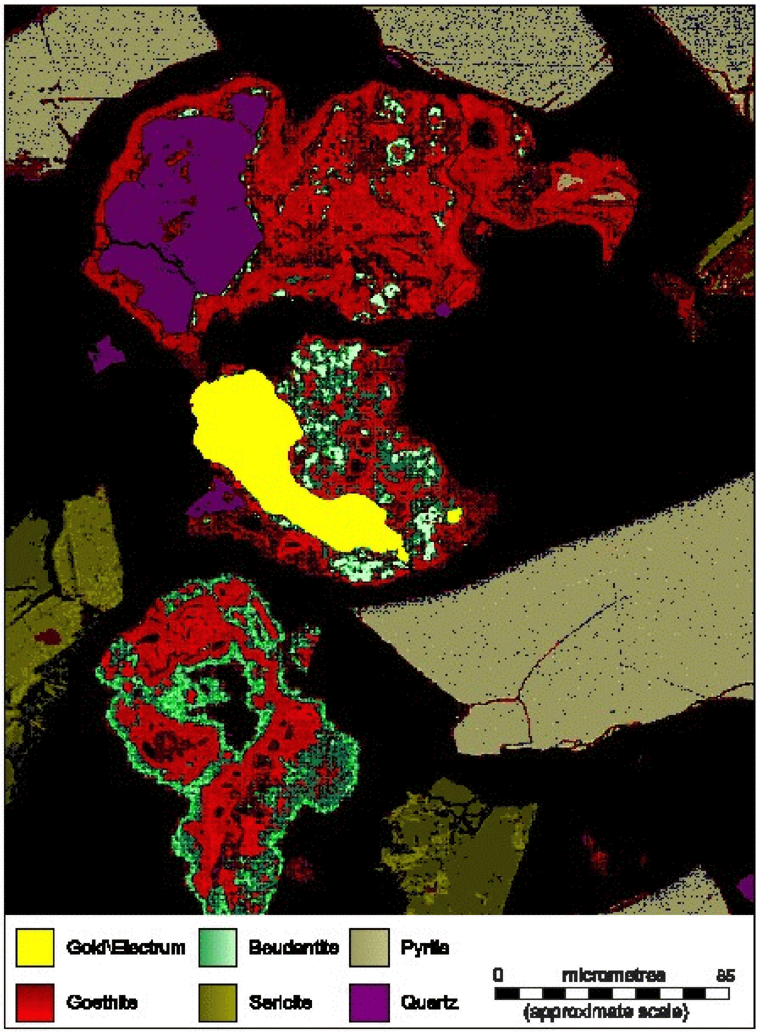


Figure 1.1: Scanning electron micrograph showing a gold particle (yellow) interlocked in gangue

The mineral processing industry is responsible for the production and supply of the raw materials in sufficient quantity and grade required for further processing. In order to accomplish this, the valuable mineral needs to be effectively and efficiently separated from the gangue material before it can be processed further.

1.1.2) Present Mineral Processing Situation

The dramatic increase of industrialization since the 18^{th} century, has caused an exponential rise in metal demand - in particular for the base metals. Copper and aluminium output have increased by a factor of 250 and 900 respectively (Wills, 1997). As a result, most metal reserves will be depleted in approximately 50 or 60 years. Therefore, ever decreasing grades of ores have to be mined to satisfy the needs of the more than six billion people on earth (Cable News Network, 1999). This affects the cost of mineral extraction - causing many mining ventures to become uneconomical.

Furthermore, a closer look at the mineral processing industry reveals that vast amounts of valuable material is wasted daily, because of physical and chemical constraints preventing the complete separation of values from gangue.

A typical mineral beneficiation circuit (or concentrator) would reduce the particle size distribution of the primary crusher product by comminution. The comminution circuit is usually followed by a flotation circuit before smelting (other processes such as magnetic or dense media separation and leaching may also be used).

As an example of the sheer scale of mining operations and the subsequent minerals engineering processes required for the extraction of the valuable metals from ore, some facts from the Palabora Mining Company (the largest producer of copper in South Africa) are given. The mine is an open pit (underground mining operations are due to start in 2002), the dimensions of the approximately ellipsoidal pit being 1800m $\times$1400m with a current depth (1999/2000) of around 650m. Around 80000 tonnes of copper ore are extracted daily, containing less than 0.5% of copper, yielding 113 200 tonnes of refined copper in 1997. The flotation stage in the concentrator consists of more than 900 flotation cells (PMC, 1998).

In general, comminution is extremely important as it is the main step where liberation of the valuable mineral from the gangue would occur. It would be of no use to attempt to separate the valuable mineral from the gangue using only flotation (as is shown in Fig 1.1). The valuable mineral needs to be liberated from the gangue first. This is accomplished by the comminution process - consisting of a series of open and closed circuit mills and screens. However, comminution is energy intensive, with typically only 1% of the energy input available for breakage. This results in energy requirements between 50% and 70% of the total energy used in the extraction process (Wills, 1992).

To illustrate Esk (1986) compared the electrical energy requirements of various stages in a Chilean copper concentrator:

Table 1.1: Energy requirements for the copper sulphide concentrator (Esk, 1986)

Operation	kWh/t	Operation	kWh/t
Crusher	**2.04**	Filtration	0.21
Grinding	**9.80**	Regrinding	**0.26**
Flotation	1.28	Water Pumping	2.80

The comminution stages in the above example account for 62% of the total electrical energy utilized in the concentrator. Esk (1986) also investigated the energy distribution of the whole mineral processing circuit, from the mining stage to the refining stage (Table 1.2).

Table 1.2: Energy distribution in Chilean copper mines (Esk, 1986)

Stage	Energy Consumption Distribution, %		
	1976	*1980*	*1990*
Mining	4.47	4.82	4.95
Concentration	39.42	42.52	43.65
Smelting	29.57	28.83	30.40
Refining	16.40	14.72	12.91
Others	10.14	9.11	8.09

It is therefore clear that any reduction in the amount of energy required to separate the valuable mineral from the gangue material will significantly reduce the cost of mineral extraction.

The Bond Work Index of an ore defines the theoretical amount of energy required to break the rock from an infinite size down to 100% passing 100µm. Typically the Bond Work Index ranges between 10 $kWh.t^{-1}$ and 20 $kWh.^{-1}$. Reducing the work index by 50% would theoretically reduce the amount of energy required in the comminution circuit by 50% and, again theoretically, double the throughput of the comminution circuit (Wills, 1997). However, although processes are available to lower the work index of ores, electrical, radioactive and ultrasonic techniques as well as thermally assisted liberation (Veasey and Wills, 1986), these methods themselves require energy, always in excess of that by which the work index has been lowered - making these processes economically unfeasible.

1.1.3) Problem Statement

In the preceding section the main problems with the current mineral processing situation have been discussed:

- High energy consumption of comminution equipment
- Low efficiency of comminution equipment
- Limited reserves and ever decreasing ore grades
- Limited liberation of valuable minerals and subsequent waste of valuable material

The practical implications if these problems are not addressed are that mineral processing circuits continue to operate inefficiently and that large quantities of valuable mineral are lost to the waste stream.

If these problems are solved, however, then huge economic savings is possible. This will enable higher plant throughout, production rates and subsequent increases in revenue.

The limiting factor in the investigation is of an economic nature. Procedures investigated in the past, although effective in decreasing energy cost of comminution circuits and increases in mineral liberation, have always been uneconomical themselves - requiring energy input in excess of the savings made possible.

1.2) Hypothesis and Objectives

1.2.1) Introduction

Table 1.1 shows that the three-stage comminution process, crushing, grinding and regrinding, contribute to 62% of the total concentrator energy consumption. If the grinding process may be altered such that valuable minerals are liberated more effectively and with 50% less energy required, then the total energy requirements will decrease from 16.39 $kWh.t^{-1}$ to 6.33 $kWh.t^{-1}$. Thus, the total contribution of the grinding circuit is only 44%.

Thermally assisted liberation has been suggested as a means towards reducing costs and increasing efficiency of process of separating the minerals from the gangue material. This method relies on the cyclical heating and cooling of a host rock, introducing thermal stresses within the mineral lattice due to the repeated expansion and contraction of the structure (Wills 1987, Tavares and King, 1995). This results in intergranular and intragranular fractures which increases the ore's grindability and decreases both the amount of energy required and the percentage of minerals in the wasted gangue material (Walkiewicz, 1993). In addition to increases in ore grindability, the following claims have also been made (Wills, 1987; Tavares and King, 1995; Walkiewicz, 1993):

- increased mineral liberation
- reduction in fines in downstream processes
- reduced plant wear is reduced.

The technique proposed in this thesis is a modification of the thermally assisted liberation technique. Instead of using conventional methods to heat the ore, the dielectric properties of the mineral constituents of the ore will be utilised and heating will be by microwave radiation (dielectric heating is further explained in Chapter 2). This allows for extremely short treatment times (as some of the minerals have extremely quick response times), thermally shocking the mineral ore (first by heating and subsequent quenching), producing fractures inside the mineral structure, thereby lowering the amount of grinding energy (Chapter 3). The application of microwave radiation has found a niche in speciality applications in a wide range of industries (as is shown in Chapter 4). Previous work illustrated that microwave thermally assisted liberation has the potential to achieve a reduction in work index of up to 90% (on laboratory scale).

1.2.2) Objective

The purpose of this investigation is to evaluate the viability of microwave energy in the comminution process with reference to the cost of mineral processing, efficiency of mineral extraction and downstream processing in order to optimise the mineral extraction process.

1.2.3) Hypotheses

Hypothesis One

If the application of microwave energy can be optimised then the energy requirements in the comminution circuit will be reduced sufficiently to reduce the cost of mineral processing.

Hypothesis Two

It is hypothesised that if an optimum amount of energy can increase the efficiency of mineral extraction, then the mineral content in the wasted gangue material will be reduced.

Hypothesis Three

If an optimum amount of microwave energy is applied to the grinding process to reduce the total energy consumption of the process, then the downstream processes in the concentrator will not be adversely affected.

1.3) The Importance of the Study

1.3.1) Benefits

This study is concerned with the thermal effects if microwave radiation on minerals, in a modification of thermally assisted liberation. From literature (Walkiewicz, 1991) the benefits of thermally assisted liberation may be summarized as:

- increases in grindability (i.e. reductions in energy consumption)
- reductions in the amount of fines (i.e. more efficient downstream processing)
- increases in mineral liberation (i.e. higher recovery)
- reductions in plant wear (i.e. lower operating cost).

1.3.2) Feasibility

Large scale microwave heaters may be adequate for drying purposes as they have been specifically designed for the dielectric properties for water, but the dielectric properties of minerals differ vastly from that of water. Industrial scale microwave equipment is available that will produce about 75kW in continuous operation. For mineral processing plants such as copper concentrators, many generators would be required and the capital cost of such a system will undoubtedly be significant. It will be vital that energy input to the system be minimized.

The comminution circuit is merely the first in a series of processes aimed at separating the valuable material from the gangue. Flotation, magnetic separation and leaching are some of the processes that may be employed after the comminution stage in the concentrator. In order for the proposed microwave treatment of the ore to be technologically and economically viable, these downstream processes must be either unaffected or affected favourably.

1.4) Conceptual Clarification

Microwave effects have become a highly controversial issue in recent years (Rybakov and Semenov, 1999). The nature of the energy sources is the main difference between so-called conventional processes and microwave processes.

Conventional processes rely on conduction, convection and radiation to heat materials. In contrast, dielectric (or microwave) heating relies on molecular vibration to heat materials from the inside (due to internal friction). Microwave effects should therefore denote only those deviations between conventional and MW processes under exact temperature conditions (Rybakov and Semenov, 1999). However, this study is mainly concerned with the thermal effects of microwaves on minerals.

1.5) Ores Tested in this Research Work

Five different materials have been tested during this research project. These are :

- Mambula ore from Richards Bay in South Africa
- Massive copper ore from Neves Corvo (Somincor) in Portugal
- Massive copper-zinc from Neves Corvo (Somincor) in Portugal
- Vermiculite from Palabora in South Africa
- Copper ore from Palabora in South Africa

Rio Tinto sponsored this research project - therefore all the ores were tested on their request. A query was received by Rio Tinto regarding the magnetic susceptibility of Mambula ore and whether or not microwave radiation will have a beneficial effect upon it. The Neves Corvo material as well as the Palabora material (copper ores) were investigated as part of ongoing research at the University of Birmingham (following on from the work of Dr Kingman). The vermiculite test work started off as a laboratory demonstration (the material was received from Mandoval - the distributors of Palabora vermiculite in the UK) and work continued regarding the alternative method for exfoliation of vermiculite.

The majority of the work consisted of trying to optimise and scale-up treatment of Palabora copper ore. It is of little use to investigate the entire downstream process after microwave radiation if the comminution section is largely unaffected (as this will be the major cost reduction stage). Therefore, most of the work on Palabora material focussed on optimising the reduction in work index (thereafter the downstream processes can be further investigated).

CHAPTER 2

INTRODUCTION TO MICROWAVE HEATING PRINCIPLES

2.1) Introduction

Microwaves is the name for electromagnetic waves arising as radiation from electrical disturbances at high frequencies. Chablinsky (1989) suggested that microwave power is THE technology of the future - with economical benefits including energy conservation and reduced processing time as well as being more environmentally acceptable.

Although this might be an overly optimistic view of the benefits of microwave energy, microwave technology is used successfully in a number of applications including the food and telecommunications industry. After the second world war (during which microwaves were first used for radar applications) intensive research into the application of microwaves in domestic and industrial appliances started (Osepchuk, 1984).

2.2) Microwave Generation

2.2.1) History

In 1888 Hertz produced the first generated microwave radiation with frequencies of up to 500 MHz by oscillatory spark discharges. In 1946, Dr P Spencer, with the Raytheon Corporation experimented with a vacuum tube called a magnetron and noticed a candy bar in his pocket had melted. This led to the first microwave oven patent (Fig 2.1).

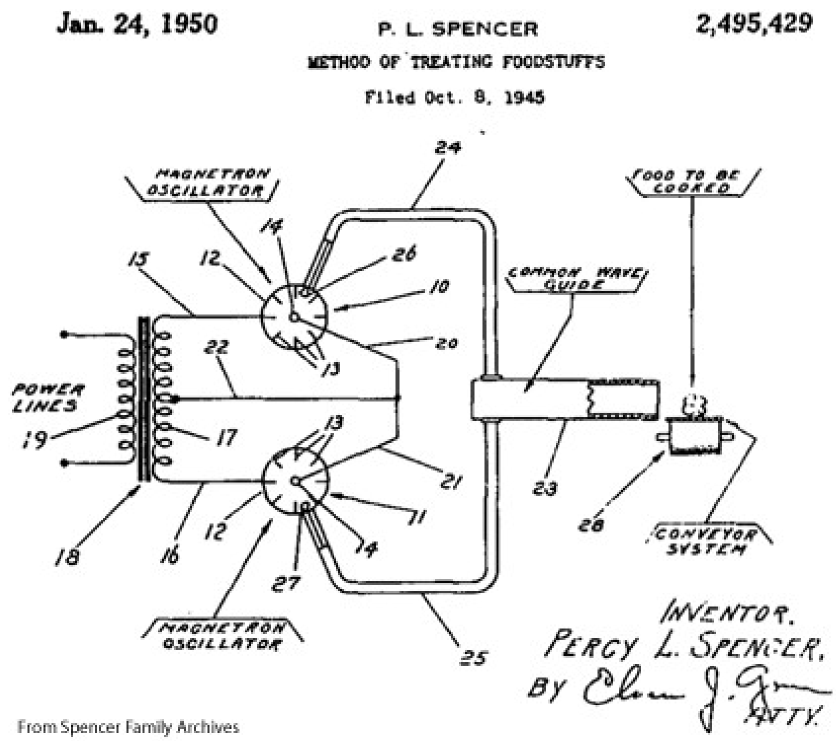


Figure 2.1 : Dr Spencer's patent for a microwave oven (after Gallawa, 2000)

The original microwave units sold by the Raytheon Corporation in 1947 were 1.7 m tall, weighed over 340 kg costing just over $5000 (Gallawa, 2000). Microwaves can be defined as that part of the electromagnetic spectrum between the infra red range and the radio frequency range with wavelengths between 1 m (frequency ~300 MHz) and 1mm (frequency ~ 300 GHz) (Stuchly et al, 1983). Characteristic of all electromagnetic radiation, the wavelength and frequency of microwaves are related by Equation 2.1 (Giancoli, 1988) :

$$c = \lambda \times f \qquad \text{Equation 2.1}$$

Where : c denotes the speed of light, 3×10^8 m.s^{-1}

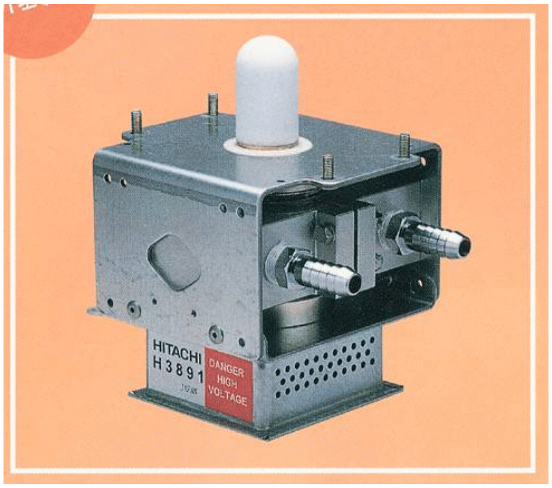
λ denotes the wavelength, m

f denotes the frequency, Hz

Currently microwaves are generated using a variety of different devices including magnetrons, klystrons, cystrons and other solid state devices (Rowson, 1986; Cober Electronics, 1998; Ishii, 1995). As the microwave units used for experimentation in this work used only magnetrons, these are the only devices discussed in more detail.

2.2.2) Magnetrons

The first devices capable of generating high power microwaves were magnetrons, a type of vacuum diodes consisting of circular, resonant cavities around a cathode immersed in a perpendicular magnetic field, which deflects an electron beam, producing the microwave field (Ishii, 1995; Roddy, 1986). A photograph of a Hitachi 3891 magnetron as well as a schematic diagram showing the different sections in the magnetron are shown in Figure 2.2.



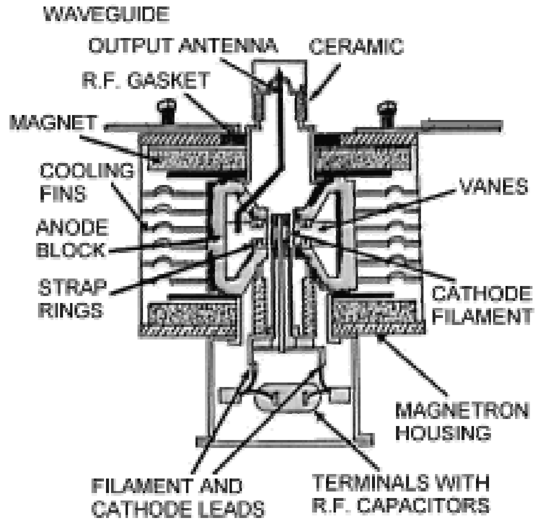


Figure 2.2 : Photograph of a Hitachi 3891 magnetron (water cooled) as well as a diagram showing the different sections within the magnetron.

The cathode (usually constructed of carbide coated tungsten in a helix shape) releases electrons when heated. The applied electric field exerts a force on the electrons such that the electrons are driven from the cathode to the anode - as shown in Fig 2.3A (absence of a magnetic field). The magnetic field (as a result of the magnets added above and below the tube as in Fig 2.3B) is perpendicular to the electric field and the path of the electrons. Figure 2.3C shows the forces and fields around an electron when viewed from the north pole of the magnets. When an electron moves through space, a magnetic field is formed around itself which strengthens the field from the magnets on the one side and weakens the field on the other side of the electron (Fig 2.3C). This causes the electron trajectory to be altered such that the electrons follow a circular route to the anode (Fig 2.3D).

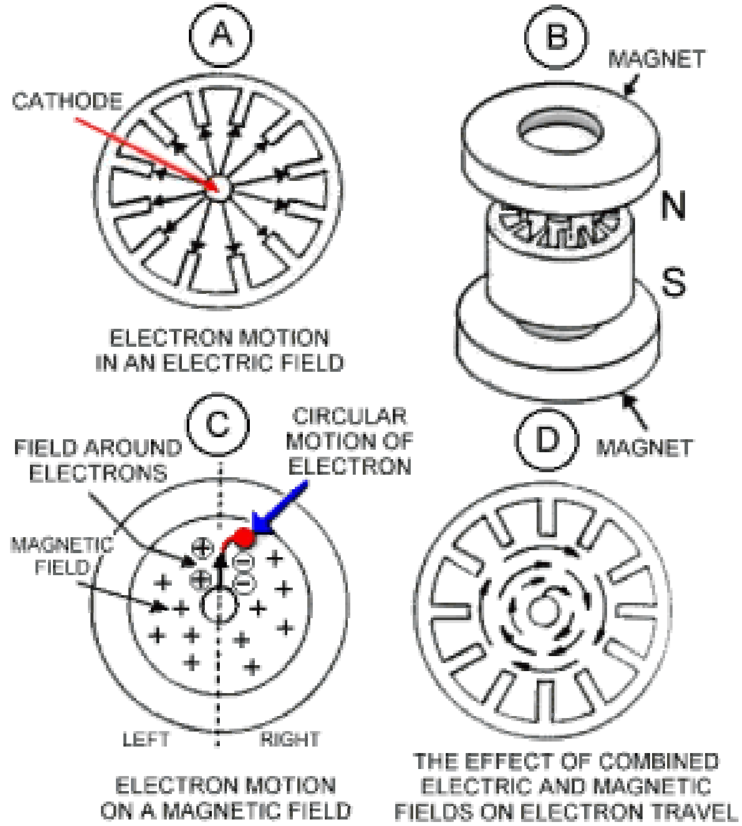


Figure 2.3 : Motion of electrons in a magnetron (after Gallawa, 2000)

The cloud of electrons resembles the spokes in a spinning wheel. When a “spoke” of electrons approach an anode vane (the segment between two cavities in the anode), a positive charge is induced in that segment. As the electrons pass, the positive charge diminishes and a new positive charge is induced in the next vane. An alternating current is induced in the anode which is transmitted through the antenna which extends into the waveguide.

The induced current is responsible for the generation of an electromagnetic field, which propagates through a waveguide as a sinusoidal wave, to an applicator (metallic enclosures containing the materials to be exposed to microwave radiation) (Cober Electronics, 1998; Bradshaw et al, 1998; Gallawa, 2000).

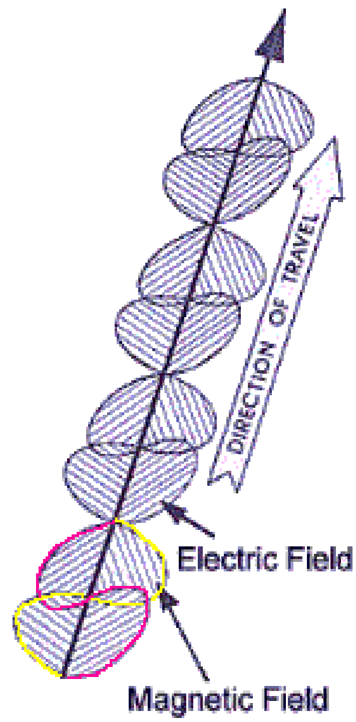


Figure 2.4 : The electromagnetic wave travelling through the wave guide (Gallawa, 2000)

2.2.3) *Cavity Types*

The first of two types of applicators is a multimode cavity, used in most domestic microwave ovens. In this type of oven the microwaves are typically scattered using a mode stirrer and heating uniformity is ensured by placing the load on a turntable.

In the second type, a monomode cavity, the microwaves are "focussed" on the load - allowing for much faster heating rates (Cober Electronics, 1998, Personal Communication : Dr Kingman, 1998).A diagram of a multimode microwave oven is shown in Figure 2.5 :

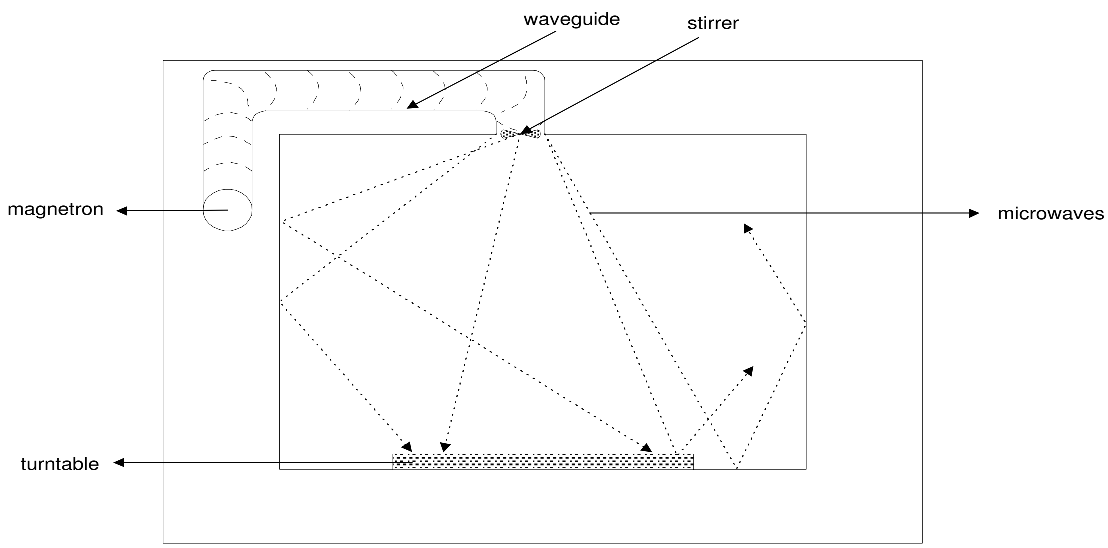


Figure 2.5 : A diagram of a multimode microwave heater

A diagram of a monomode microwave oven is shown in Figure 2.3 :

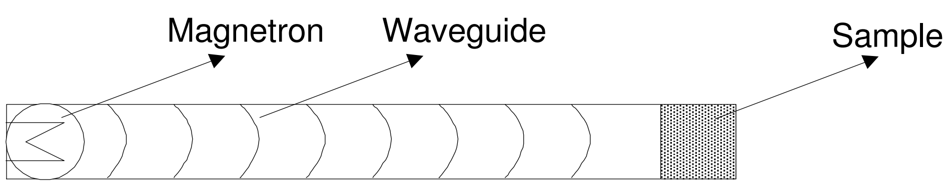


Figure 2.6 : A diagram of a monomode microwave heater

The power output of a magnetron can be adjusted in several ways. These include, pulsing the power output (the most common method), adjusting the anode current, changing the magnetic field and attenuating the microwave energy.

The last method does not affect the magnetron at all, but are purely mechanical in nature (it attenuates the microwave energy between the magnetron and load) and is very expensive (Ishii, 1995).

2.2.4) Waveguides and Cavity design

The life of the magnetron is dependent on the design of the microwave cavity to optimise the load impedance to the magnetron. Microwaves may be directed along waveguides or co-axial cables, in a similar way as water through a pipe. Waveguides are often preferred as they are capable of transmitting higher powers. Waveguides are metallic tubes, either circular or rectangular in cross section, the dimensions determines the frequency of the microwaves. The microwaves propagate through the waveguide, in the form of a sinusoidal wave, until it reaches the load. If the load absorbs the wave completely, then there is no reflection of the wave back down the waveguide. This only occurs when if the resistive load and the source load have equal impedance, and is called a matched load (Ishii, 1995).

In the case of unequal load and source impedances, absorption of the radiation is incomplete and the wave is reflected. The reflected load combines with the transmitted wave, resulting in a standing wave. The ratio of the maximum and minimum amplitudes is called the voltage standing wave ratio (VSWR). A high VSWR means that a significant amount of energy is reflected (the magnetron may be damaged), thus it is important to calculate the VSWR of any load during the design phase (Ishii, 1995).

The VSWR may be calculated from Equation 2.2 (V_i ,V_r are the voltage due the incident and reflected wave respectively).

$$VSWR = \frac{V_{\max}}{V_{\min}} = \frac{(V_i + V_r)}{(V_i - V_r)} \qquad \text{Equation 2.2}$$

The Rieke diagram is a circular diagram (on a polar coordinate system), indicating the output power for fixed input conditions. It also plots the frequency change of the magnetron as a function of the phase and magnitude of the VSWR. A Rieke diagram is shown in Figure 2.6 :

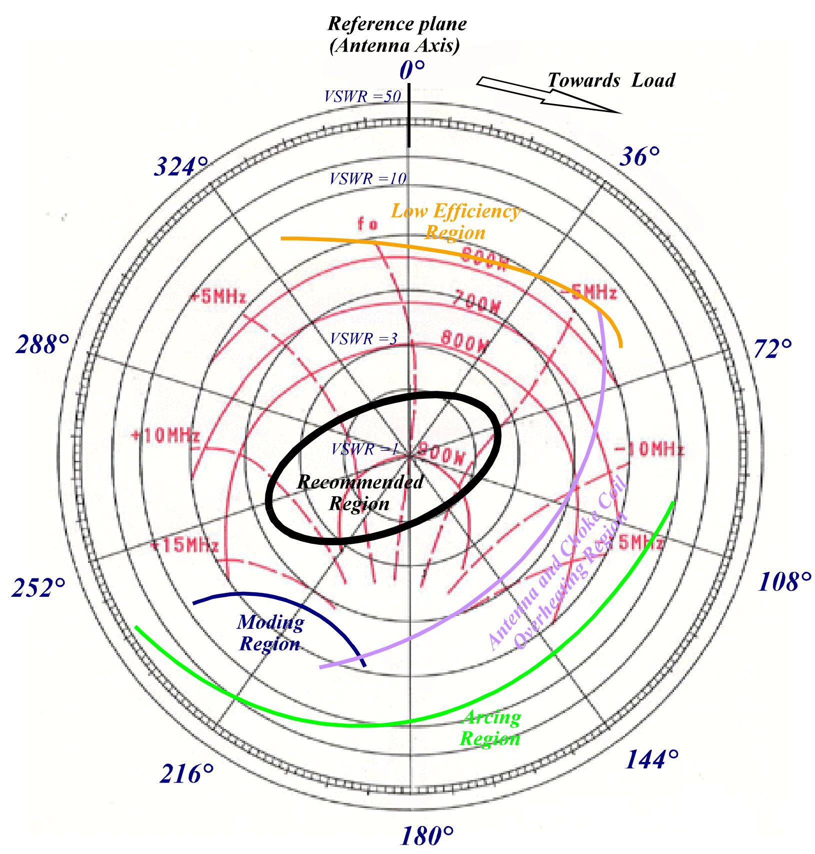


Figure 2.7 : Rieke diagram for a typical domestic magnetron

Sometimes recommended operating regions as well as several restricted regions (such the moding, arcing and overheating regions) are indicated on the Rieke diagram..The moding region is the place where moding may occur if the impedance loci are in the region and if the length of the waveguide for the connecting magnetron to the cavity is short (Ishii, 1995). The arcing region is the region where arcing may occur around the antenna of the magnetron is the load impedance loci are in the region. The arc may occur between the antenna and the inside wall of the waveguide.

The antenna and choke coil parts may be overheated if the impedance loci are in the overheating region. Even if the loci are only near the region, the antenna and choke coil may be considerably overheated. If the magnetron is operated for extended time periods in the low-efficiency region, the output power will be low and the magnetron will overheat. It is recommended that the impedance loci under the load conditions of normal use are in the recommended region, as the highest efficiency and stable oscillation will be obtained.

2.3) The Mechanism of Dielectric Heating

According to Hulls (1992), two frequency bands can be used for dielectric heating - radio frequencies (below 300 MHz) and microwaves (above 300 MHz). In this work, dielectric heating refers to heating induced by microwave radiation only.

Microwave heating occurs at molecular or atomic level and is be due to a combination of 4 methods (Cober Electronics, 1998) :

Dipole Rotation	Electromagnetic Heating
Resistive Heating	Dielectric Heating

As the 4 heating mechanisms are very similar in nature - all depend on internal friction to generate heat - the term dielectric heating will hereafter be used to refer to the heating of materials using microwaves.

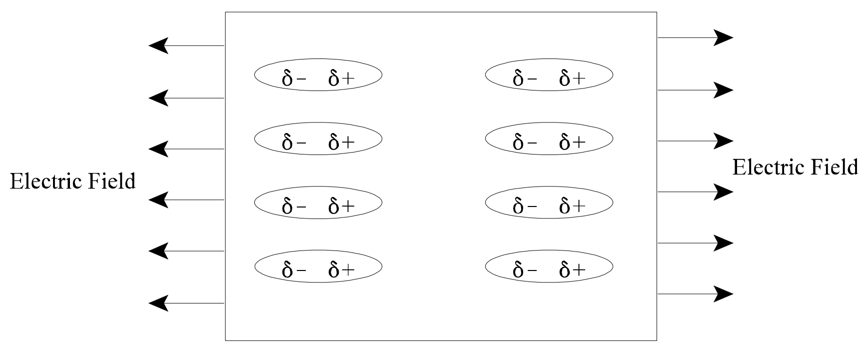


Figure 2.8 : Diagram of aligned dipoles in an electric field

If a dielectric material is placed in an electric field the difference in electric charge in the individual particles will align according to the applied electric field, as is shown in Figure 2.8. In an alternating field, these particles will turn through 180° each time the field changes. Generally the frequency of non-telecommunications use are restricted to 915MHz and 2.45 GHz (variations are possible depending on location).

Therefore the particles will re-align themselves 915 million times per second (or 2.45 billion times - depending on the frequency). This causes internal friction which, in turn, heats up the material. Materials that heats in a microwave field are called dielectrics or lossy materials.

Conventional heating methods are dependent on conduction for heating the material. This process is relatively inefficient and problematic especially when heating non-conducting materials need to heated as the surface may overheat because of the small conductivity. However, dielectric heating generates the heat from "within" (as explained in Section 2.5) and are thus capable of rapidly heating most dielectric materials (Hulls, 1992).

2.4) Theoretical Aspects of MW Heating

The rate at which a material heats due to the power dissipated may be expressed as (Stuchly et al, 1983) :

$$\frac{dT}{dt} = \frac{P_d}{C_p\ \rho} \qquad \text{Equation 2.3}$$

Where :

	T	is the temperature of the heated material, °C
	t	the time, s
	P_d	is the power density dissipated in the heated material, $W.m^{-3}$
	C_p	is the specific heat capacity, $J.kg^{-1}.°C^{-1}$
	ρ	is the density of the material, $kg.m^{-3}$

Power density may be expressed as :

$$P_d = \sigma \ |E_i|^2 \qquad \text{Equation 2.4}$$

Where : σ is the equivalent conductivity, $S.m^{-1}$

E_i is the internal electric field, $V.m^{-1}$

The conductivity of most lossy materials is dependent on the frequency. The loss tangent is a more useful parameter - being relatively independent of frequency for many materials (Roddy, 1986) :

$$\tan \delta = \frac{\sigma}{\omega \ \epsilon_r^{'} \ \epsilon_0} \qquad \text{Equation 2.5}$$

Where : ω is the angular frequency of the microwave, $rad.s^{-1}$

ϵ_r' is the relative permittivity (dielectric constant)

ϵ_0 is the permittivity of free space, 8.854 $pF.m^{-1}$

The dielectric permittivity, in general a complex quantity, is used to describe the electrical properties of a material (Batt et al, 1994) :

$$\hat{\epsilon} = \epsilon^{\prime} - j \ \epsilon^{\prime\prime} \qquad \text{Equation 2.6}$$

Where : $\hat{\epsilon}$ is the complex permittivity, $F.m^{-1}$

ϵ' is the real part of the complex permittivity, called the dielectric or dispersion factor, $F.m^{-1}$

ϵ'' is the imaginary part of the complex permittivity , called the loss factor, $F.m^{-1}$

j is the imaginary number $\sqrt{-1}$

The real term, ϵ', describes the energy stored within the material and the imaginary term, ϵ'', is the loss factor and quantifies the dipolar relaxation loss, the conduction and Maxwell-Wagner losses (Bradshaw et al, 1998; Batt et al, 1994). As the permittivities are generally very small numbers it is customary to divide by the permittivity of free space, ϵ_0, to give the relative permittivities :

$$\begin{aligned} \hat{\epsilon}_r &= \frac{\hat{\epsilon}}{\epsilon_0} \\ &= \frac{\epsilon'}{\epsilon_0} - j\,\frac{\epsilon''}{\epsilon_0} \\ &= \epsilon_r' - j\,\epsilon_r'' \end{aligned} \qquad \text{Equation 2.7}$$

$\hat{\epsilon}_r$ is the relative permittivity

ϵ_r' is the relative dielectric factor

ϵ_r'' is the relative loss factor

Low loss materials (such as alumina) are those with values for the relative loss factor of less than 0.01, which means that the materials will couple poorly with microwaves, and generally would not heat up significantly. Materials with ϵ” greater than 5 usually have small penetration depths.

The loss factor, and the loss tangent are often used to quantify the amount of dielectric heating possible. The loss tangent is defined below (von Hippel, 1954) :

$$\tan\ \delta = \frac{\epsilon''}{\epsilon'} \qquad \text{Equation 2.8}$$

The power absorbed by the material is directly proportional to the loss factor, the frequency and the intensity of the electric field as is shown in Equation 2.9 (Harrison, 1997), derived from Eq 2.4:

$$P_d = 2\ \pi\ f\, E_i^2\ \epsilon_0\ \epsilon'' \qquad \text{Equation 2.9}$$

Equation 2.9 ay be written in terms of relative permittivity and loss tangent as well (Church et al, 1988) :

$$P_d = 2\ \pi\ f\, E_i^2\ \epsilon_r'\ \tan\ \delta \qquad \text{Equation 2.10}$$

The dielectric heating process and the heat lost due to radiation are shown in Equations 2.11 and 2.12 respectively (by the combination of Eq 2.3 Eq 2.9and the Stefan Bolzman relationship for radiation losses).

$$\frac{dT}{dt} = \frac{K \epsilon^{//} f E^2}{\rho \ C_p} \qquad \text{Equation 2.11}$$

$$\frac{dT}{dt} = \frac{-e \ \sigma_{sb} \ A_s}{\rho \ C_p \ V_s} \qquad \text{Equation 2.12}$$

Where : K is a constant

e is the emissivity of the sample

σ_{sb} is the Stefan Bolzman constant, 56.7051 $nW.m^{-2}.K^{-4}$

A_s is the sample area, m^2

V_s is the sample volume, m^3

It can be seen from the above equations that the permittivity has a direct effect on the heating characteristics of materials. It is therefore important to know the factors affecting the loss factor and dielectric factor.

2.5) Factors Affecting Dielectric Heating

Microwave heating depends on several factors such as dielectric permittivity, conductive losses, temperature, field intensity, frequency, density (Goyette et al, 1990) as well as cavity design (Roddy, 1986; Rybakov and Semenov, 1999). Describing the effect each of these have on dielectric heating is not a simple matter, as interactions between these factors also occur.

2.5.1) *Dielectric Permittivity*

The power absorbed by a unit volume of dielectric material may be described by (Goyette, et al, 1990)

$$P_a = 2 \pi \epsilon_0 f E_{100}^2 \epsilon_{eff}'' \left[W.m^{-3} \right] \qquad \text{Equation 2.13}$$

Where : E_{100} is the local electric field in $V.m^{-1}$

ϵ_{eff}'' is the effective permittivity given by :

$$\epsilon_{eff}'' = \epsilon'' + \frac{\sigma}{2 \pi \epsilon_0 f} \qquad \text{Equation 2.14}$$

The effective permittivity describes the combination of capacitive losses in the heated material and the conductive losses. The real part of the permittivity also affects the dielectric heating as it has a direct effect on the applied electric field, E_0 :

$$E_{100} = \frac{3 \epsilon'}{2 \epsilon' + 1} E_0 \qquad \text{Equation 2.15}$$

2.5.2) Conductive Losses

Conductive losses affects the effective permittivity as described in Eq 2.14 (Goyette et al, 1990).

2.5.3) *Temperature and Frequency*

Nelson et al (1989) showed that the dielectric properties of metal oxides such as hematite, ilmenite and manganese oxide are strong functions of the frequency of radiation, while the dielectric properties of pyroxene and goethite is independent of frequency.

The permittivity is dependant on temperature (Rowson, 1986; Goyette et al, 1990). This is intuitively correct as the alignment of the dipoles becomes increasingly more difficult as the temperature increase due to the increasing kinetic energy of the dipoles.

Heating ethanol at 2.2 GHz is a good example of the temperature dependance of dielectric heating. At room temperature, ethanol relaxes at 1 GHz. As temperature increases, its relaxation frequency increases. Initially, the value of ϵ'' increases as well at 2.2 GHz, but at 50°C, the relaxation frequency exceeds 2.2 GHz, which results in a decreasing ϵ''. Therefore the dielectric losses increase and heating becomes increasingly inefficient at 2.2 GHz.

2.5.4) *Sample Geometry and Mineralogy*

For some geometries in the above example, the real part of the permittivity increases with temperature, affecting the local field within the sample. As the field within the samples increases, the substance will increase in temperature at a steadily increasing rate. This is called a thermal avalanche. Thermal avalanche's are of particular importance in composites, as different materials respond differently to dielectric heating.

Walkiewicz et al (1988) reported on the effect of particle size on heating properties of graphite. It was found that finer size graphite responds better to microwave radiation that the larger sizes. Gasner (1984) findings on the pyrolysis of bituminous coal confirmed the results of Walkiewicz. Particle size also affects the voidage and thus the bulk density of the sample. The bulk density of the sample affects the heating characteristics. Kingman et al, 2000, reported on the effect of mineralogy on microwave assisted grinding, which is dependant on the heating properties of the material.

2.5.5) Density and Composition

The composition of the dielectric material is also affects the permittivity. This can be explained as the mechanism for dielectric heating depends on the presence and relative freedom of dipoles in the material (Goyette et al, 1990).

To illustrate, in the literature some authors stated that hematite (Fe_2O_3) is a good absorber of microwave energy (Chen et al, 1984; Walkiewicz, 1988). Others (Wright et al, 1989) stated the opposite. Hematite occurs in different mineral forms, having different crystal latices and impurities. The different mineral forms affects the dielectric properties of hematite.

Arai et al (1995) developed mathematical models based on mixture equations for the estimation of complex permittivities in ceramic materials. Theoretically, these mixture equations may be used to estimate the dielectric constant of a mixture of known composition and knowing the dielectric properties of the individual components.

Kraszewski and Nelson, 1999, showed that similar techniques may be used to determine the composition of a coal-limestone mixture, accurately and more quickly than the traditional combustion techniques.

Nelson (1996) showed that the permittivity of solid coal and limestone may be calculated from measurements of permittivity of pulverized samples and applying mixture equations, showing that the permittivity is a function of composition. Florek and Lovas (1995) showed that the permittivity is also a function of the particle grain size.

Church and Webb (1986), Flemming et al (1989) and Batt et al (1994) describes different techniques for the measurement of dielectric properties. Church (1989) developed a model to predict the amount of microwave radiation absorbed for different minerals. This was then used to prove that mineral properties may selectively be modified to improve separation.

2.5.6) Cavity Design

Rybakov and Semenov al (1999) stated that for industrial utilization of microwave technology it is important to implement the homogenous processing (heating) of large volumes. It is also claimed that this may be achieved using millimetre-wave radiation and super multimode cavities as applicators.

2.6) The Penetration Depth

It has been stated that microwaves heat materials from the inside out (Hulls, 1992). This implies penetration of the microwaves into the dielectric. Two measures for indication of microwave penetration into materials are in use : the penetration depth and the skin depth.

The distance over which the electric field is reduced to e^{-1} is called the skin depth whereas the penetration depth is the distance over which the power is reduced by e^{-1}. As power is proportional to the square of electric field, the penetration depth is half of the skin depth. Penetration depth may be expressed by Equation 2.16 (Bradshaw et al, 1998) :

$$d = \frac{\lambda}{2\pi\sqrt{2\epsilon'}}\left[\sqrt{1+\left(\frac{\epsilon''_{eff}}{\epsilon'}\right)^2}-1\right]^{\frac{-1}{2}} \qquad \text{Equation 2.16}$$

Where d is the penetration depth, m.

The dielectric constant is a function of the field strength and pattern and therefore also of the penetration depth. The change in field strength amplitude with penetration depth is shown in Figure 2.9 :

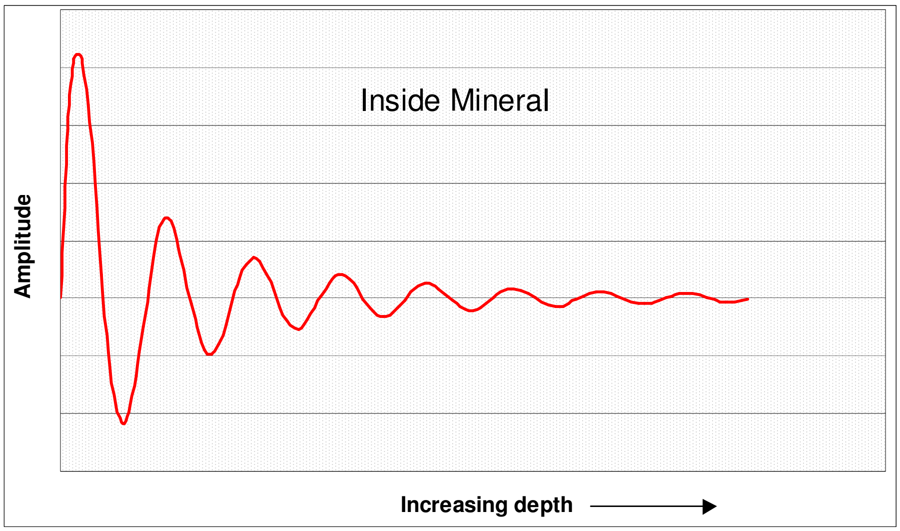


Figure 2.9 : Diagram showing that the amplitude of the microwave decreases as it penetrates a material (after von Hippel (1954) and Metaxas and Meredith (1983))

2.7) Electric Field Strength Calculations

The heating rate of minerals is directly related to the electric field strength of the microwave unit. Therefore, a higher power microwave unit, with a lower electric field strength, will be less efficient than a lower power microwave unit, with a higher electric field intensity.

The general equation describing the energy increase (or decrease) is (Abbot and van Ness, 1989) :

$$C_p = \left(\frac{\delta Q}{\Delta T}\right)_P \qquad \text{Equation 2.17}$$

Where : δQ is the amount of energy required, J

ΔT is the change in Temperature, °C

C_p is the heat capacity of the material at constant pressure, $J.K^{-1}$

Therefore it follows that for a mass, m of material :

$$Q = m\ \hat{C}_p\ \Delta T \qquad \text{Equation 2.18}$$

Where : m mass, kg

$\hat{C}_p$ Specific heat capacity, $J.kg^{-1}.K^{-1}$

The absorbed power will therefore be :

$$P_a = \frac{m\ \hat{C}_p\ \Delta T}{\Delta t} \qquad \text{Equation 2.19}$$

Where : P_a is the absorbed power, W

Δt represents the time, s

Combining Equation 2.3 and 2.19 gives :

$$\frac{dT}{dt} = \frac{2 \pi f E_i \, 2 \, \epsilon_o \, \epsilon^{//} V}{m \, \hat{C}_p} \qquad \text{Equation 2.20}$$

Solving for E_i :

$$E_i = \sqrt{\frac{\rho \; \hat{C}_p \; dT}{2 \; \pi \; f \, \epsilon_o \; \epsilon^{//} \; dT}} \qquad \text{Equation 2.21}$$

Where : ρ is the density of the material, $kg.m^{-3}$

Therefore, for a substance for which the relation between temperature and relative loss factor is known (such as water for which $\epsilon^{//} = \frac{320}{T}$, with T in Kelvin) the change in temperature may be measured from which the field strength of the microwave unit may be calculated.

The relationship between external and internal electric field strengths are dependant on the geometry of the system and the dielectric properties of the materials (Meredith, 1998). For the special case of a sphere in an otherwise uniform field :

$$E_i = \left(\frac{3}{2 + \epsilon^{/}}\right) E_o \qquad \text{Equation 2.22}$$

2.8) Determination of Dielectric Constants and Loss Factors.

A material's dielectric properties at microwave frequencies can be estimated by the measurement of the propagation characteristics of an electromagnetic wave through the medium. Different techniques exist for the measurement, the choice of any particular method depends on the frequency, the properties of the material as well as the form and availability of the sample (Lance, 1964).

The dielectric properties of Palabora ore was measured (at 915MHz and 298K) using the resonant cavity method, which is based on the perturbation theory. Perturbation methods are used to study the effects of small changes. Because of the small perturbation, it is assumed that the solution deviates little from the ideal, therefore the effect of the small changes can be calculated from the ideal. The permittivity is dependent on the frequency it is measured at as well as the temperature. For future work regarding the measurement of dielectric properties, it is recommended that these properties are measured at the frequency of the microwave unit used over a whole range of temperatures.

This method is based on the perturbation of a TM_{ono} cavity. Variable frequency multimode cavities are used to give spot frequencies of 650 MHz, 1410 MHz and 2215 MHz.. A sample (heated in a conventional heater) is moved rapidly into a high electric field area. The change in resonant frequency is a function of the dielectric constant, and the change in cavity Q gives an indication of the loss factor for the material (Lance, 1964, Baden- Fuller, 1979).

The Q-factor for a cavity is the ratio of the energy stored to the energy dissipated as is described by the formula :

$$Q\text{-}factor = 2\pi f_0 \frac{Energy\ Stored}{PowerDissipated} = \frac{f_0}{\delta f} \qquad \text{Eq 2.23}$$

Batt et al (1994) reports that the repeatability of this type of measurement for $\epsilon' < 40$ is good. However, the measurement for materials of higher permittivities are not as repeatable. There is also poor agreement between different methods for the calculation of ϵ''.

2.9) Microwave Safety Considerations

Microwaves are potentially hazardous : their effect may not be noticed until damage to living organisms has been done. The body is designed to warn against excessive external heat not internally generated heat as would be the case if microwave radiation penetrates the skin causing internal heating (Baden-Fuller, 1979).

Energy balance considerations (based on a standard man in standard conditions) suggest that 10 $W.cm^{-3}$ (or 100 $W.m^{-2}$) is a safe upper limit, even in the case of infinite exposure. The reason for this is because thermoregulatory systems compensate for the absorbed power. A power level as low as 10 $W.m^{-2}$ may be considered as having no heating effect - even in extreme conditions of temperature and humidity (Baden-Fuller, 1979).

However, evidence (disputed) suggests non-thermal effects through the nervous system. Claims are made that exposure over a period of years to power levels in excess of 2 $W.m^{-2}$ lead to nervous system disturbances - even though occupational exposure to healthy adults suggest that there is no effect (Baden-Fuller, 1979). Table 2.1 gives the standard radiation exposure limits for various countries.

Table 2.1 : International Microwave Radiation Exposure Standards (Stuchly et al, 1983)

Country	Date	Type	Frequency Range	Exposure Limits
Canada	1979	Occupational	10-10 000 MHz	1 $mW.cm^{-3}$
		Public	0.01-300 GHz	1 $mW.cm^{-3}$
Sweden	1976	Occupational	10 - 300 MHz	5 $mW.cm^{-3}$
UK	1971	Occupational	0.03 - 30 GHz	10 $mW.cm^{-3}$
USA	1982	Occupational	30 - 300 MHz	1 $mW.cm^{-3}$
EU	1980	Occupational	0.3 - 300 GHz	10 $mW.cm^{-3}$

The International Microwave Power institute has developed a voluntary industrial standard of 10 $mW.cm^{-3}$ for equipment operating at a minimum load (Kingman et al, 1998).

CHAPTER 3

COMMINUTION AND THERMALLY ASSISTED LIBERATION

3.1) Introduction

Comminution, derived from the Latin *comminuere*, refers to the mechanical breakdown of solids into smaller particles (Napier-Munn et al, 1999). Comminution encompasses all crushing units, tumbling mills, stirred mills as well as sizing processes (Napier-Munn, 1999). The role of comminution is not only size reduction, but more importantly, the liberation of minerals from each other (Wills and Atkinson, 1993).

Only approximately 1% of the energy input into comminution equipment is available for size reduction, making the comminution process one of the most intensively researched areas in the world (Wills and Atkinson, 1993). Esk (1986) conducted a study on the energy requirements of mineral processing circuits. Of particular interest is the breakdown in energy consumption for a copper sulphide concentrator.

Table 3.1 : Energy requirements of a copper sulphide concentrator (after Esk, 1986)

Operation	**Electrical Energy Usage (kWh/t)**	**Operation**	**Electrical Energy Usage (kWh/t)**
Crushing	2.04	Filtration	0.21
Grinding	9.8	Regrinding	0.26
Flotation	1.28	Water Pumping	2.8

As can be seen from Table 3.1, 62% of the energy in the concentrator is consumed by the grinding stages alone. It has been estimated (Napier-Munn et al, 1999) that 1.5% of the total energy consumption in the U.S. is utilized in the comminution processes of mineral concentrators alone. According to King (1994) there is already indications that the energy cost of comminution will determine whether or not mineral resources may be economically exploited.

Any reduction in energy requirements for the milling process would result in huge savings. Thermally assisted liberation (T.A.L.) has been suggested as a possible method for the reduction in comminution energy costs (Wills et al, 1987). This method relies on the cyclical heating and cooling of a host rock, introducing thermal stresses within the mineral lattice due to the repeated expansion and contraction of the structure (Kingman et al, 1996).

Figure 3.1 shows intergranular fracture (on the left) which causes increased liberation, a reduction in fines and an increase in grindability. On the right a picture of transgranular fracture is shown - i.e. the cracks go through the middle of the particles - also increasing the grindability, but increasing the amount of fines produced and does not necessarily aid the recovery of valuable mineral species.

Conventional T.A.L. has the fundamental problem that the total mass of ore is heated. In a microwave heating process, the ore's dielectric properties are utilized and therefore specific mineral phases respond at different rates. This allows for extremely short treatment times (as some of the minerals have extremely quick response times), thermally shocking the ore (first by heating and subsequent quenching), producing fractures at the grain boundaries, thereby lowering the grinding energy required for liberation.

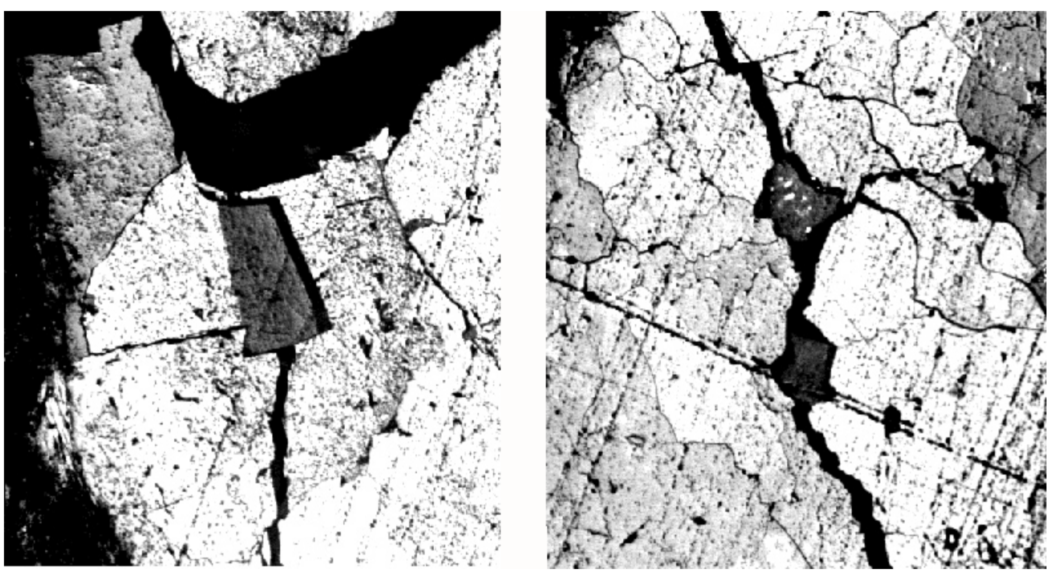


Figure 3.1 : Intergranular (left) and transgranular (right) cracks

3.2) Comminution

The main concern here is the grinding stage. Consequently the focus of this section is the description of grindability and methods used to quantify grindability. The comminution phase may be made more efficient if the grindability, a measure of the ease of grinding (Deniz et al, 1996), can be increased.

3.2.1) Principles of Comminution

Bond (1961) described comminution in terms of 3 principles. The First Principle of Comminution is that each particle has an energy level associated with it. Therefore, the total energy input into the crushing device must be equal to the difference between the energy level of the product and the feed.

The Second Principle recognises the fact the useful energy input in the comminution phase is proportional to the increase in length of the cracks. The Third Principle relates to the fact that the breaking strength of a rock is determined by the weakest flaw in the mineral structure (Bond, 1961a,b). Bond (1961a) describes various methods and equations that may be used to estimate the work index. These include the Crushing Test, the Rod Mill Grindability Test, the Ball Mill Grindability Test and the Hardgrove Grindability Rating.

3.2.2) Calculation of Mill Power Requirements

The energy (dW) required for a small change in size (dD) of a unit mass may be described as a power function of the particle size (D) (Coulson et al, 1996; Wills, 1997) :

$$\frac{dW}{dD} = -k\ D^{p} \qquad \text{Equation 3.1}$$

Where : k is a constant

p comminution exponent

In 1867, von Rittinger (Coulson et al, 1996) proposed that p = -2 in Eq 3.1 and derived :

$$W = k_R\, f_c \left(\frac{1}{P} - \frac{1}{F}\right) \qquad \text{Equation 3.2}$$

Where : f_c is the crushing strength

k_R is Rittinger's constant

As can be seen from Equation 3.2, von Rittinger believed that the energy required for the size reduction is proportional to the increase in surface area. Kick, in 1885 (Coulson et al, 1996), proposed that p = -1 in Eq 3.1 which gives :

$$W = k_k\, f_c\, \ln\left(\frac{F}{P}\right) \qquad \text{Equation 3.3}$$

Where : k_K is Kick's constant

Kick's equation takes into account the reduction ratio - and implies therefore that elastic deformation occurs before fracture.

Bond suggested that p = -1.5 (Coulson et al, 1996) and derived that :

$$W = W_i \sqrt{\frac{100}{P}} \left(1 - \frac{1}{\sqrt{R}}\right) \quad where \quad R = \frac{F}{P} \qquad \text{Equation 3.4}$$

Where : W_i is the work index of the material.

The work index, W_i, describes the resistance of the material to crushing and grinding. Equation 3.4 is called the Third Theory equation. The problem with this type of analysis is that the breakage characteristic of an ore depends on the particle size (Coulson et al, 1996; Personal Communication : Dr NA Rowson; Wills, 1997). Napier-Munn et al, 1999, pointed out the following limitations of Bond's method :

1) The performance of a real closed circuit is not accurately predicted when the throughput is increased, unless the classifier performance is adjusted.
2) The Bond test can not be used in cases where the size distribution slopes change.
3) The test can not be used to predict the grinding behaviour of large rocks, such as in the case of autogenous mills.

4) The method does not work well for materials with unusual screening characteristics - however, this is a general problem for all methods relying on sieving.

5) The squire root relationship (in Eq 3.4) for the new crack length does not always hold

3.2.3) Estimation of the Work Index

One of the most widely used methods of quantification of mineral strength and grindability is the Bond Work Index The method which was used for the estimation of the work index uses Holmes' general equation (Warren Spring Laboratory, 1962, Bond, 1961b) :

$$W = W_i \left(1 - \left(\frac{1}{R}\right)^r\right) \left(\frac{100}{P}\right)^r \qquad \text{Equation 3.5}$$

Where : P, F is defined as the 80% passing sizes of the product and feed respectively

R is the reduction ratio, $R = \frac{F}{P}$

r is the comminution exponent

The work index is defined as the "kWhrs per short ton to reduce the material from an infinite feed size to 80% passing 100µm" (Bond FC, 1961a).

However, in the experimental estimation of the work index, all the material is crushed to 100% passing 2.8 mm, to standardise the test procedure (Warren Spring Laboratory, 1962). The -2.8 mm material is screened using standard sieves and the 80% passing size is determined from a plot of log *(cumulative mass percentage finer than any size)* against log *(size in μm)*.

A unit volume of 700 ml is then placed in a ball mill with a diameter of 304.8 mm and a length to diameter ratio of 1:1. The mill is loaded with 285 steel balls with a mass of 20.13 kg and is set to rotate at 70 revolutions per minute.

The composition of the mill load is outlined in Table 3.2 (Warren Spring Laboratory, 1962) :

Table 3.2 : Composition of the Bond Mill's Ball Load

Nominal Ball Size (cm)	**Number of Balls**
3.81	43
3.18	67
2.54	10
1.9	71
1.27	94
Total	**285**

The critical speed of a mill, i.e. the speed at which the charge at the liner surface would centrifuge, is given by (Napier-Munn, 1999) :

$$N_c = \frac{42.3}{\sqrt{D}} \qquad \text{Equation 3.6}$$

Where : N_c is the theoretical critical mill speed, rpm

D is the mill diameter, m

For a Bond Mill, the critical speed is 76.6 rpm. After 50 revolutions (as an initial estimate), the mill contents are removed and the material are screened with the smallest sieve the size of the required product size (P_1). The mass of material finer that P_1 produced in the grinding process can then be calculated.

The grindability of the of the ore is defined as the mass of -P_1 (M_{-P1}) material produced divided by the number of revolutions, $_{Nc}$:

$$G = \frac{M_{(-P_1)}}{N_c} \qquad \text{Equation 3.8}$$

The load +P_1 should be replaced in the mill, and the mass of -P_1 material removed should be replaced with new feed of the same mass as the -P_1 material and the same particle size distribution as the original feed. Ideally, the grindability should be calculated at various screen sizes. If accuracy is not critical, then the grindability may be calculated at only one screen size (Bond, 1961a).

The work index may then be estimated :

$$W_i = \frac{4.45}{P_1^{0.22}\ G^{0.8}\left(\frac{1}{\sqrt{P}} - \frac{1}{\sqrt{F}}\right)} \qquad \text{Equation 3.9}$$

The work index, Wi, may then be substituted into Bond's Third Law equation (Eq 3.4) or into Holmes' equation (Eq 3.5), to estimate the energy requirement of the grinding stage.

The grindability should be calculated until it remains constant for 3 cycles and the mill recirculating load is approximately 250% (Magdalinovic, 1987). However, this takes a long time (between 7 and 10 cycles) and is prone to many experimental errors.

3.2.4) Alternative Methods

Magdalinovic (1987) and Armstrong (1985) derived alternative methods for the estimation of the Work Index. Magdalinovic's (1987) method suggested that the grindability be calculated from :

$$G = \frac{m - \frac{1}{3.5} M (1 - r_0)}{N_c} \qquad \text{Equation 3.10}$$

Where :	G	mass of new undersize obtained per mill revolution, kg
	M	mass of the unit volume sample, kg
	m	mass of undersize, m =(1/3.5) M, kg
	r_0	is the proportion of new oversize mill feed

From the grindability the work index can be calculated as before. It has been proved

(Magdalinovic, 1988) that this method works for copper ore, limestone and andesite.

Berry and Bruce (1966) describes a simple method for the estimation of the work index.. In Bond's Third Theory equation (Eq 3.4), it can be seen that two different ore samples of the same mass, with approximately equal feed sizes under identical milling conditions, will require approximately the same work input, W.

Therefore, the work index of a test ore may be estimated if the work index of a reference ore is known :

$$W_{it} = W_{ir} \frac{\dfrac{10}{\sqrt{P_r}} - \dfrac{10}{\sqrt{F_r}}}{\dfrac{10}{\sqrt{P_t}} - \dfrac{10}{\sqrt{F_t}}} \qquad \text{Equation 3.11}$$

Where : r and t refers to the reference and test ores respectively

3.2.5) Common Values of the Bond Work Index

Values of the Bond Work Index for a variety of minerals is given in Table 3.3.

Table 3.3 : Values for W_i for a Variety of Materials (Wills, 1997; Kingman, 1999)

Material	***Work Index***	***Material***	***Work Index***
Barite	4.73	Fluorspar	8.91
Bauxite	8.78	Granite	15.13
Coal	13	Graphite	43.56
Dolomite	11.27	Limestone	12.74
Emery	56.7	Quartzite	9.58
Quartz	13.57	Palabora Cu-Ore	13.6

3.2.6) Alternative Methods to Quantify Rock Strength

One of the main problems with the Bond Work index is the constraint that the ore needs to be less than 2.8 mm in diameter. As the 80% passing size of the feed to rod mills are frequently larger than 15mm, the size limitation of the Bond method is a serious constraint.

There are also problems with relative techniques (such as the Berry and Bruce method) such as the accuracy and true meaning of results. An added disadvantage of these methods is that it is difficult to assess the influence of material specific breakage properties and machine-specific properties (Napier-Munn et al, 1999).

This coupled with the fact that the Bond method is tedious and has the possibility of many experimental errors, other techniques have to be found to quantify the effect of microwave radiation on the ore.

Single particle breakage tests have been developed for the determination of the comminution behaviour of rock material. These tests are typically either slow, indirect tensile tests or high velocity impact tests. It is the tensile strength of rock governs its ultimate failure as rocks are much weaker in tension than in compression (Napier-Munn et al, (1999) claim that the tensile strength of a material is only equal to approximately 10% of its compressive strength).

The point load test may be used to determine the tensile strength. A standardised, point-load strength test was initially developed by Broch & Franklin (1972) although the actual stress distribution within rock cores under point load still remains ambiguous (Chau and Wong, 1996)

The stresses in point-loaded specimens was analysed mathematically and photoelastically by Hiramatsu & Oka (1966). An equation for the calculation of the tensile strength was suggested although the method of testing was not perfect due to the stresses in the test piece. Generally there are few theoretical solutions for the point load test, due to these complexities of the resultant stress distributions developed within.

Peng (1976) attempted to model the stress distribution in cylindrical discs subjected to double point loading. Biaxial tensile and compressive stress regions were observed within the test specimens, but the exact stress at which crack propagation initiated was not determined. This was mainly due to the complex nature of the stress distributions within the specimen. Peng stated, however, that the method was a reliable means of determining the preferred orientation of a fracture plane.

Mellor and Hawkes (1971) found that failure initiated away from the compressive regions under loaded platens. Vutukuri (1974) and Hudson et al (1972) both questioned the validity of the Brazilian test. Vutukuri (1974) found that under certain experimental conditions crack propagation could initiate at the loading points and thus invalidate the test.

Hudson (1969) suggests that the compressive component of the stress field does not influence the tensile fracture of the test specimen within the Brazilian test. Hudson et al (1972) argued that the propagation originates from the crushed zone below the loading points and considered the Brazilian test being invalid for tensile strength measurement. Clark (1992) used numerical modelling techniques to evaluate the principal stress difference within test specimens in the Brazilian test. Progressive changes in stress concentration were calculated and presented as contour plots. In addition to a central tensile crack, shear cracks that initiate and propagate from the loaded surface into the sample was observed.

During the impact test the rock sample is crushed between two hard surfaces. The rate of loading is substantially greater than in the relatively slow indirect tensile tests. Many types of impact tests have been described in the literature i.e. the Ultra fast load cell and the Hopkinson pressure bar. Such tests have all been designed to relate the size distribution or new surface of the comminuted products to the specific fracture energy.

Early pendulum tests were conducted to study the effects of velocity on single particle breakage. Vutukuri et al (1974) concluded that an increase in impact velocity produced a greater amount of fines upon breakage. A twin pendulum test was developed at the Julius Kruttschnitt Mineral Research Centre (Napier-Munn et al, 1999). The twin pendulum device was used to fracture single particles and relate the comminution energy required to the resultant progeny. The test was used to develop ore-specific breakage functions for various comminution models.

Brown (1997) used a similar swing pendulum device to fracture rock cores. The resultant product size distribution was described by a fragmentation fractal dimension, and an attempt was made to relate this to the size of the parent particle. The twin pendulum device consists of an impact and a rebound pendulum which are suspended from a rigid framework. The impact pendulum is raised to a certain height and released onto the test specimen, which is located on the rebound pendulum.

The specific comminution energy (E_{cs}), is then evaluated by determining the input energy associated within the impact pendulum, the residual energy within the impact pendulum after impact and the energy transmitted to the rebound pendulum (Napier-Munn et al, 1999).

Napier-Munn et al (1999) discussed the twin pendulum test and identified some limitations with this device. The range of input energy and particle size range is restricted and the test procedure is somewhat time-consuming. There is also an inherent error with the calculation of the breakage energy as a secondary motion of the rebound pendulum is sometimes observed.

Among the first widely accepted drop weight tests, was the test developed by Protodyakonov in 1962 (Napier-Munn et al, 1999) and was used to determine the resistance of rock to failure. The test, consisted of a laboratory scale cylinder, into which test specimens were placed. A known mass was then repeatedly released from a certain height onto the sample. A strength coefficient was obtained, which could be related to the compressive strength of the rock specimen.

Evans and Pomeroy developed a similar procedure to that of Protodyakonov for measuring the strength of coal - namely the Impact Strength Index (ISI) . The authors proposed correlations of ISI values and the compressive strength of cubed specimens.

More recently, a drop weight test was developed at the Julius Kruttschnitt Mineral Research Centre as an alternative to the twin pendulum test. The test itself serves to overcome many of the inherent limitations associated with twin pendulum devices. Bearman et al (1997) related the specific comminution energy to the material fracture toughness. He concluded that correlations should exist between any tensile test and the drop weight tester, as the method of breakage is similar. The drop weight test rig consists of an impact plate mounted on guide rails. The impact mass falls under gravity, crushing a single rock particle, which is located on a hardened steel impact plate, mounted on a concrete block. A wide range of input energies, related to the progeny size, can be produced by varying the release height of the impact plate.

Napier-Munn et al (1999) stated that the drop weight tester provide analogous data to the twin pendulum device and has several advantages. The extended range of input energies and particle size, reduced testing time, greater precision and amount of test applications make the drop weight test rig the favoured testing device.

Some of the advantages of the drop weight apparatus above the pendulum devices are listed below (Bearman et al, 1997) :

- extended input energy range compared to the twin pendulum devices.
- reduction in experiment time
- a wider particle size range can be tested
- particle bed breakage tests may be conducted

Based on the specific energy input, E_{is} in kWh.t^{-1}, required, the height from which the drop mass should be released can be calculated (Napier-Munn et al, 1999; Bearman et al, 1997)

$$h_i = \frac{\bar{m} \cdot E_{is}}{0.0272 \cdot M_d} \qquad \text{Equation 3.14}$$

Where : h_i height from which the drop weight is dropped, cm

$\bar{m}$ mean particle mass, kg

M_d mass of the drop weight, kg

Generally 10 mm is added to the height, h_i, to compensate for the height above the base where the drop mass comes to rest. The results obtained from the drop weight test provides a relationship between the energy required for breakages and the size distribution produced by the energy.

The parameter most commonly used to describe the breakage of the ore from the drop weight test is the t-parameter. The usual size distribution curve is normalised with respect to the input size. In other words, the percentage passing various fractions of the input size is calculated (ie t10 is the fraction passing a $\frac{1}{10}^{th}$ of the input size).

The parameter, t10, is related to the specific comminution energy by the equation :

$$t10 = A\left(1 - e^{-b\,E_{cs}}\right) \qquad \text{Equation 3.15}$$

Where : t10 is the percentage passing a tenth of the original input size

Ecs is the specific comminution energy in $kWh.t^{-1}$

A, b are ore breakage parameters

The numerical value of A represents the theoretical limiting value of t10, whereas b is the slope fo the t10 versus E_{cs} graph. In addition to which, A and b are related to the Mode I fracture toughness which is defined as the resistance of rocks to catastrophic crack extension (Bearman et al, 1997; Napier-Munn et al, 1999). Bearman at al, 1997 gave the relationship as :

$$\begin{aligned} b &= 2.2465\ K_{ic}^{-1.6986} \\ A \cdot b &= 126.96\ K_{ic}^{-1.8463} \end{aligned} \qquad \text{Equation 3.16}$$

In theory, this provides an alternative method for the calculation of the Mode I fracture tougness, K_{ic}. From a plot of t10 versus E_{cs}, the parameters of A and b can be determined. Once these parameters have been estimated, Eq 3.16 can be used to determine K_{ic}.

Another test type is the three point bend test. When a cylindrical specimen is subjected to loading within the three-point bend test it is strained by tensile, compressive and shear stress. Tensile stress develops on the convex side of the beam and compressive stress on the concave side. Vutukuri et al (1974) reviewed the work of several authors within the field of bending tests.

Numerous practical limitations existed in the test, such as wedging beneath the concentrated loads, stress concentration at surface cracks and flaws, torsional stress during the application of load and frictional forces between loading points and the specimen surface. A high-speed image and thermal stress analysis has shown crack propagation to initiate in the tensile area of the loaded specimen and the mechanism of failure is not dependent upon such factors.

The test consists of cylindrical rock core specimens which are supported by two end plates and diametrically applied load. The support and load plates are fabricated from mild steel, in order not to induce excessive compressive and shear stresses within test specimens at points of contact. This is necessary in ensuring that crack propagation occurs in the convex side of the specimen, within the area of pure tension. The span of the support platens can easily be adjusted, enabling a wide range of sample sizes to be tested.

3.3) Thermally Assisted Liberation

3.3.1) Introduction

Thermally assisted liberation (T.A.L.) has been studied as a potential method to improve liberation and comminution characteristics. Various authors (Walkiewicz et al, 1991; Veasey and Wills, 1991; Walkiewicz, 1993; Tavares and King, 1996) studied this phenomena and the results were positive.

Thermally assisted liberation is the process of cyclical heating and quenching of ore to introduce fractures in the mineral lattice. As different minerals respond at different rates to temperature changes (rate of expansion or contraction), differential stresses is created at the grain boundaries. These localised stresses in the mineral lattice weakens the structure of the mineral, thereby facilitating comminution as well as assisting in liberation of mineral species.

The major disadvantage of conventional T.A.L. has always been the economics of the process. Conventional furnaces and kilns require vast amounts of energy and/or fuel to heat the entire particle. Wonnacott and Wills (1990) calculated the reduction in work index of Cornish tin ore achieved by heating the ore to different temperatures. The cost of heating the ore (based on the heat capacity of each constituent) was found to be between 364 pence per ton and 439 pence per ton, depending on the final temperature of the ore. The reduction in required milling power was calculated to be 0.31 pence per ton.

3.3.2) Literature Review of TAL on Mineral Species

One of the most common gangue minerals is quartz. A large amount of studies have therefore been devoted to determine the effect of temperature changes on quartz structure (Tavares and King, 1995). It was found that thermal pretreatment alone did not have any significant change on breakage strength of quartz. However, water quenching of quartz after heating, had the effect of significantly reducing the strength (measured using an ultra fast load cell) of the quartz.

Air quenching did not significantly affect the strength of the material. It was found that quartz can accommodate temperature changes of up to 100°C/min (air cooling only produced around 80°C/min. The rapid cooling rates associated with water quenching (several hundred degrees Celsius per minute) produced intense cracking due to high stresses.

The high stresses within the material structure is attributed to rapid contraction of the outer layers of the quartz along with compression in the centre of the particle. Due to the increased heat transfer coefficients of salt water, quenching in a 10% NaCl solution further reduces the material strength.

It was found that the quartz particle size affects the efficiency of TAL as well. More pronounced effects are observed for smaller particles. This indicates the very disseminated nature of the microflaws produced during TAL.

The reduction in fracture energies of quartz have been attributed to α to β-quartz phase inversion (at 573°C) with a volume change of 0.86%.

Significant effects on hematite particle strength were observed. Air quenching affected particle strength but only marginally. However, fracture energies were reduced by up to 90% upon water quenching. The α-β phase transformation may have contributed to the weakening (Tavares and King, 1995).

Both iron and copper ore showed reductions in fracture energy of approximately 30% following heating to 600°C and 800°C, respectively, and quenching (Tavares and King, 1995).

Wills et al, 1987, investigated thermally assisted liberation of cassiterite. It was found that heat treatment at 650°C followed by quenching decreasing the grinding energy requirement by up to 55%. However, this is off-set by the cost of heat treatment.
It was reported that an economic process, involving heat treatment would have to improve recovery as well. An increase in recovery of only 1% in the case of tin from South Crofty would make thermal pre-treatment an economical alternative.

Wonnacott and Wills (1990) investigated the effects of thermally assisted liberation of cassiterite. It was reported that dense liquid analysis and shaking table separation failed to determine an effect on processing. From photomicrography, a large number of transgranular cracks were observed (see Fig 3.1). A possible explanation for the lack of evidence for changes in metallurgical processing due to transgranular fracture have been ascribed to the indiscriminate rod milling process.

Şener and Őzbayoğlu, 1998, investigated the effect of thermal pre-treatment on the grindability of ulexite ($NaCaB_5O_9 \cdot 8H_2O$) and colemanite ($Ca_2B_6O_{11} \cdot H_2O$), reporting changes in grinding characteristics for both minerals after thermal treatment in the temperature range 200°C to 360°C.

This is ascribed to water being quickly released from the crystal structures, causing the mineral to become amorphous or recrystallize into a new crystalline phase. Ulexite exfoliates slowly due to the gradual release of water, causing microcracks and interstices in the structure.

3.3.3) Microwave Assisted Thermally Assisted Liberation

Veasey and Wills (1991) reported that for thermally assisted liberation to be feasible, the heat transfer to the mineral structure would have to be optimised. Microwave heating has been suggested as a means to accomplish optimisation due to the heating method, described in Chapter 2. Shown below is a graph of temperature versus microwave exposure time for pyrite and quartz.

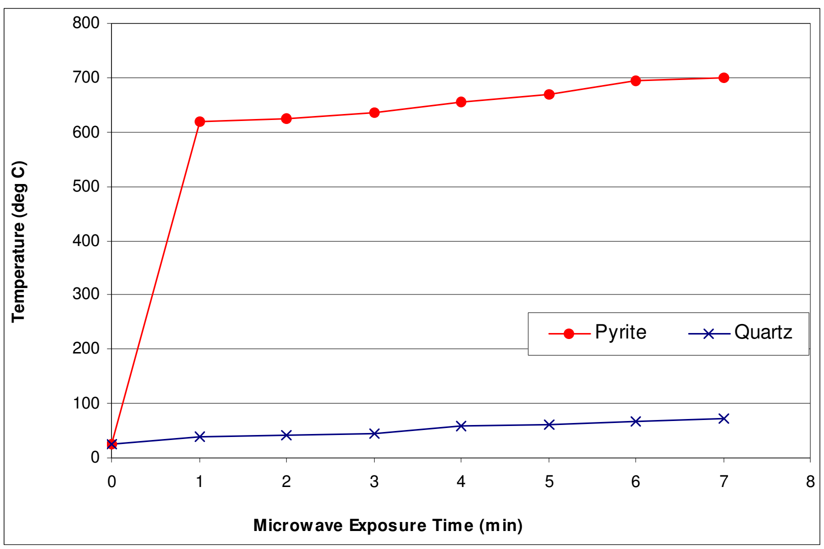


Figure 3.2 : Comparison of the heating rate of a good receptor, pyrite, and microwave transparent quartz (Harrison and Rowson, 1996)

Because of the different heating rates, of pyrite (value) and quartz (gangue), a mineral ore consisting of these two species need only be exposed to microwave radiation for a short interval to accomplish differential heating and therefore induce the necessary stresses at the grain boundaries to weaken the mineral. Of added advantage is that most valuable minerals are good microwave receptors, whereas gangue are typically transparent. No bulk heating of the material is therefore necessary as is the case for conventional heating.

Güngör and Atalay, 1998, states that “to create cracks and micro-fractures at the mineral grain, the ore must be composed of minerals which have different heating characteristics when they are exposed to microwave radiation”.

Walkiewicz et al, 1988, reported on the advantages of thermally assisted liberation :

i) Fines Reduction in Downstream Processing
ii) Increases Ore Grinding
iii) Increased Liberation
iv) Reduced Plant Wear

Bonometti et al, 1998, reported on additional advantages such as volumetric heating instead of surface absorption as is the case with conventional heating, uniform heating as well as high heating rates. With the main disadvantage of T.A.L. being economic in nature, microwave technology may offer a unique solution.

CHAPTER 4

APPLICATION OF MICROWAVE ENERGY IN MINERAL PROCESSING

4.1) History of Industrial, Scientific, Medical and Domestic Applications of Microwave Power

Research into industrial, scientific, medical and domestic (ISM&D) applications of microwaves developed at slow rate (Stuchly et al, 1983). The first reference to the industrial application of microwaves can be found in the patent literature around 1937 by Kassner (Osepchuk, 1984), who suggested the use of microwaves to heat materials. This idea was first commercially exploited by the Raytheon company in the 1940s. However, the original units were bulky, heavy and expensive : the original microwave units sold by the Raytheon Corporation in 1947 were 1.7 m tall, weighed over 340 kg costing just over $5000 (Gallawa, 2000).

During 1973 a large number of publications appeared in the Journal of Microwave Power relating to food processing in various countries (Schiffman, 1973; Meisel, 1973; Suzuki and Oshima, 1973; Giles, 1973; Kase, 1973). The general finding of these articles are that although the amount of research being carried out in this field is notably less than in the telecommunications industry, some advances have been made and due to the increasing labour cost and energy shortages of the era, microwaves became increasingly attractive.

The production of ceramics depend on heating a compacted powder to such temperatures that sintering occurs, forming a dense, strong solid (McPherson, 1988). The conventional process of heating the powder up slowly to the required temperature before allowing it to cool down slowly, was carried out in kilns. It was realised that microwaves could be used to achieve similar effects - albeit at a much faster rate. This would result in huge energy savings, increasing yield and better quality control (Chablinksy, 1989, McPherson, 1988).

Agrawal (1999) reports on the use of microwave heating for sintering of ceramics, composites, metals and transparent materials. It was found that microwaves offer an alternative method for the sintering of tungsten-carbide composites. Microwaves have advantages over the conventional methods as well, as they produce finer microstructures and lower the sintering conditions. Although most metals are excellent reflectors of microwaves at room temperature, in powdered form, most metals and alloys couple extremely well with microwaves, producing sinters of improved quality. Slower sintering times for ceramics have also been reported, with an acceptable quality.

Goldstein et al, 1999, studied the sintering of Al_2O_3/TiC powders in air. Reduced sintering times have been achieved that can not be reproduced in a conventional system.

Scharz et al (1973) and Dalton (1973) respectively showed that microwaves may be successfully applied in the vulcanization of rubber and in non-contact measurement in the steel industry.

Measurement devices which were developed include a Doppler-based length measurement device, a thickness gauge and a blast furnace rangemeter (Dalton, 1973). Many other uses have also been suggested ranging from agriculture to the textile and paper industries (Stuchly et al, 1983; Chablinsky, 1989). Agricultural applications includes insect control. Grain containing insects can be treated in a microwave heater, killing the insects without damaging the grain (as insects and grain have different dielectric loss factors).

Seeds with water impermeable seedcoats germinate late and have not developed sufficiently by harvesting time. Irradiation of these seeds results in improved germination without side-affects. Morozov et al, 1999, conducted experiments on the effect of different band frequencies of microwave radiation on sowing properties and plant growth. It was found that improvements on the seed germination as well as growth properties is possible, with an increased carrot harvest of up to 16% is possible.

4.2) Applications of Microwaves in the Mineral Industry

Most of the industrial applications of microwave technology, excluding those in the telecommunications industry, are based on heating of water molecules such as in domestic microwave ovens. However, as Roddy (1986) states that "ovens and heaters for industrial applications generally have to be custom made for the particular application". Large scale microwave heaters may be adequate for drying purposes as they have been specifically designed for the dielectric properties for water, but the dielectric properties of minerals differ vastly from that of water.

However, due to constraints by most governments on the frequencies allowed for microwave applications outside the telecommunications industry, researchers are restricted to conducting “non-optimum” research. Regardless of this restraint, some interesting and useful applications in the minerals industry have surfaced over the past decades.

4.2.1) The Effects of Microwave Radiation on Metal Oxides and Sulphides

Chen et al (1984) conducted experiments to determine the response of various minerals to microwave heating (Table 4.1) :

Table 4.1 : The response of various minerals to microwave radiation (Chen et al, 1984)

Mineral	Power (W)	Response
Cassiterite	40	Little heating
Chalcopyrite	15	Heats well, liberation of sulphur fumes
Galena	30	Heats well, liberation of sulphur fumes
Magnetite	30	Heats well
Pyrite	30	Heats well, liberation of sulphur fumes
Sphalerite	>100	No heat generation

Walkiewicz et al (1993) confirmed that microwave energy can be used to induce thermal stress cracking. Taconite was heated in a microwave chamber at 2.45 GHz at 3 kW. It was found that the work index may be reduced at all tested bulk sample temperatures, with a 13% decrease at 197°C.

It was also shown that the samples do not need to be heated to high temperatures. The only requirement would be that the heating be rapid and selective.

Gasnier et al (1994) used a monomode microwave to conduct chemical synthesis experiments on oxide and hydroxide powders. It was found that reduction and oxidation occurs readily, but that the exposure time, volume of mineral powder, the dielectric properties and microwave power play a vital role in the processes involved.

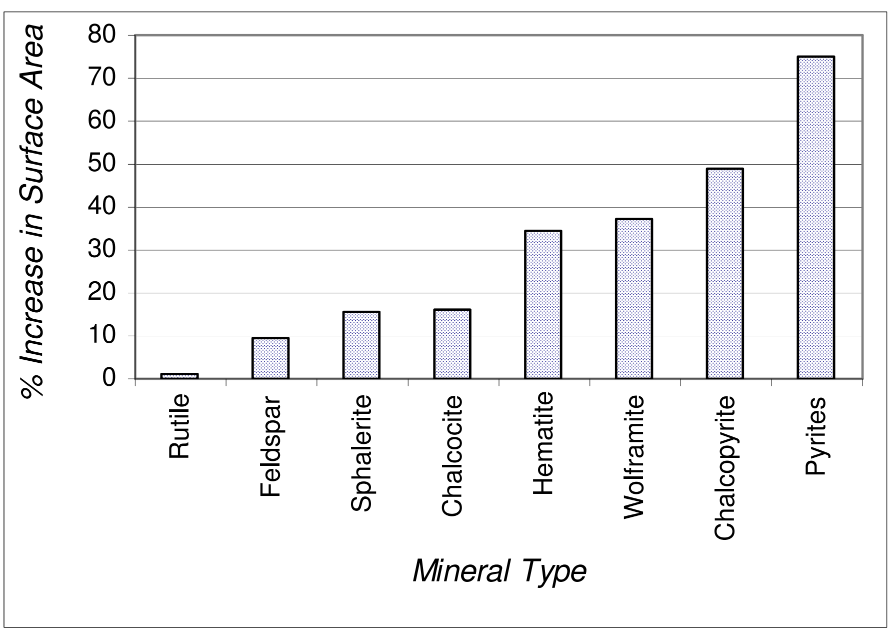
Tavares and King (1996) irradiated a sample of copper bearing ore. They found no significant weakening of the ore, although a small amount of fracture along the grain boundaries was observed.

Florek et al (1996a) found that microwave heating induces phase changes on the grain boundaries of the minerals, which change in the magnetic properties of the mineral. It was suggested that pyrite, chalcopyrite and carbonate siderite undergoes either oxidation or sulphatization to form new phases which differ in magnetic susceptibility.

Kingman (1996) showed that the work index of Norwegian ilmenite can be reduced by approximately 80% if the sample is quenched after 60 s of microwave treatment at 1.3

kW, compared to a reduction of approximately 25% for a non-quenched sample.

It was also shown that an increase in microwave power level affects the decrease in work index. In the same study it was also shown that the magnetic susceptibility of the magnetite ore decreases after microwave treatment while magnetic susceptibility of the ilmenite increases. This can be explained by the oxidation of magnetite to hematite which is less magnetic. In the case of ilmenite, the increase in temperature makes the alignment of the atoms easier giving rise to a more structured lattice. By irradiating various ores for 300 s, Harrison and Rowson (1996b) obtained data for the percentage increase in surface area and comparative work index. These are shown in Figure 4.1 and 4.2.



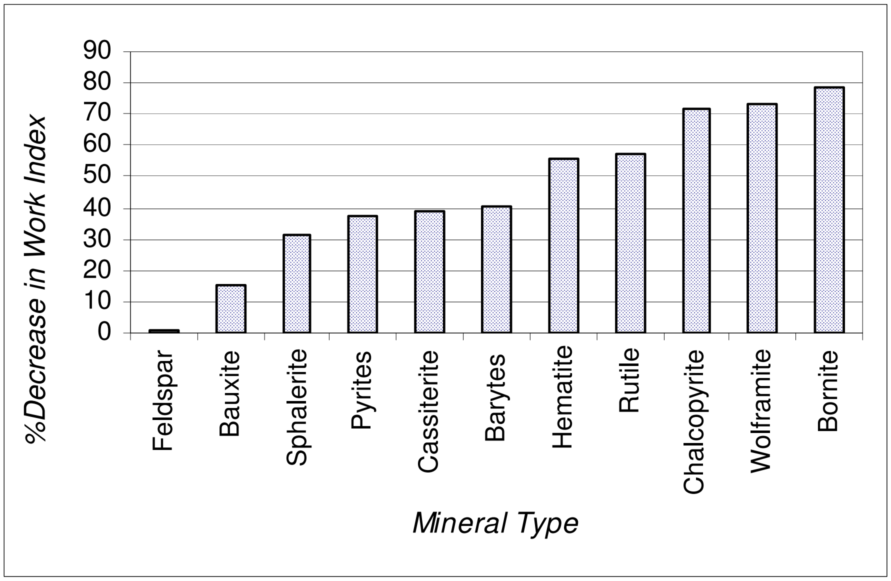


Figure 4.1 : Histogram showing the percentage increase in surface area for various minerals after 300s of microwave radiation at 650 W (Harrison and Rowson, 1996b)

Figure 4.2 : Histogram showing the percentage decrease in work index area for various minerals after 300s of microwave radiation at 650 W (Harrison and Rowson, 1996b)

Harrison and Rowson (1997a) conducted a study on the effect of conventional and microwave (650 W at 2.45 GHz) heating on bornite, chalcocite, chalcopyrite, magnetite and pyrite. An analysis of the surface area shows an increase in all cases after heat treatment. All five minerals showed a decrease in comparative work index. No changes in the crystal lattice was observed after heat treatment.

An investigation into the magnetic properties of the minerals confirms the result by Kingman (1996) that oxidation along the grain boundaries affect the magnetic susceptibility. The results for conventional and microwave heating are similar, although the changes occurs in a much shorter time period when microwaves are used.

Experiments were conducted on chalcopyrite (Florek et al, 1996a; Florek et al, 1997) from Rudňany - which is not susceptible to magnetic treatment. However, after microwave treatment at 750W for 40 s, the magnetic susceptibility was increased 100 fold. Analysis of the microwaved product confirmed the formation of new phases such as maghemite and magnetite.

Joret et al, 1997, investigated the non-thermal effects of microwave radiation on the dissolution rate of metal oxides (in particular Co_3O_4, and CeO_2) in nitric acid. It was found that microwave energy does not provide any activation for the dissolution reactions, but only acts to heat up the solution. However, it was stated that this does in an indirect way affect the activation of the reaction, as an increase in temperature, accelerates the reaction.

Peng J and Liu C, 1992, report on the leaching kinetics of sphalerite with ferric chloride in a microwave field. It was found that due to the fast rate of internal heating, the kinetics of the leaching system is positively affected.

4.2.2) Effects of Microwave Radiation on Coal

Rowson and Rice (1990) used a low power microwave (500W) to desulphurise coal. It was found that although the pyrite (FeS_2) was preferentially heated, the final temperature reached was insufficient to convert the non magnetic pyrite to the magnetic pyrrhotite.

However, mixing the coal with NaOH or KOH, resulted in a sufficient increase in temperature to enhance the magnetic properties of the pyrite and to dissolve the pyrite and organic sulphur, resulting in a reduction of sulphur content of approximately 70%. Harrison and Rowson (1996a) showed that the comparative work index of coal can be reduced by up to 30% in a 650W, 2.45 GHz microwave.

Harrison and Rowson (1996a) and Harrison and Rowson (1997b) showed that conventional and microwave treatment of coal improves the grindability to a similar extent. However, microwave treatment of coal has the added benefit of reducing the sulphur and ash content as well as a safer, more controllable form of drying (Butcher and Rowson, 1995; Harrison and Rowson, 1997b).

4.2.3) Effects of Microwave Radiation on Gold Processing

Woodcock et al (1989) investigated the effect of microwave radiation on various parts of a gold processing circuit. Microwave radiation may be applied in the roasting phase to remove the arsenic and sulphur. Mixing the concentrate with sodium hydroxide before microwave treatment resulted in the formation of various soluble salts of sulphur and arsenic and a high recovery gold.

One of the most common methods for gold extraction is the carbon-in-pulp method, where the gold is adsorbed onto the activated carbon. Adsorbed gold is then removed by elution and the carbon treated in acid bath to remove contaminants before being regenerated at approximately 660°C in a steam atmosphere. The re-activated carbon is then returned to the CIP circuit.

Conventionally, activated carbon is regenerated in rotary kilns or vertical tube furnaces. However, the granulated activated carbon can be easily heated by microwave radiation. The advantages of microwave heating in this application is that the carbon may be heated rapidly with better temperature control and possible energy savings (Bradshaw et al, 1998). Initial test work by Bradshaw et al (1998) indicates that microwave regeneration of activated carbon is economically feasible and gives activated carbon with high abrasion resistance.

4.2.4) The Effects of Microwave Radiation on Iron Production

The reduction of iron ores is dependant on the surface area available for gas-solid contact (Standish and Pramasunto, 1991; Xia and Pickles, 1997). Because of the differential heating and subsequent stress cracking caused by microwave radiation of minerals and the increase in surface area which results, microwave radiation may be applied in the reduction of iron ore particles in a CO atmosphere.

It was found (Standish and Pramasunto, 1991) that microwave treatment at 1300 W for 6 minutes gives the optimum enhancement of reduction and that microwave pretreatment of iron ore may be an alternative to sintering or pelletising.

Zhong et al, 1996, conducted experiments on the reduction of iron ore with coal in a 15 kW microwave cavity of volume 0.06 m^3 in an inert nitrogen atmosphere to inhibit re-oxidation of the iron.. It was found that microwave heating has the potential for significantly reducing the reaction time compared to conventional heating. Comparing the time required for 80% of the reduction to occur, the conventional case requires almost 30 min whereas the microwave case requires only 7.5 min, a reduction of more than 75%.

It is also stated that one of the major advantages of using microwaves is that the microwave energy dissipates rapidly throughout the whole volume of the sample, heating it directly, and not by conduction as is normally the case. The last of the advantages of the application of microwave heating above conventional heating mentioned, is the fact that a dust free, high energy flue gas is produced.

4.3) Parameters Affecting Microwave Radiation

4.3.1) The Effect of Particle Size on Microwave Radiation

Standish (1989) conducted experiments in order to verify the effects of particle size on the heating rate. It was found that particle size does in fact affect the heating rate, although literature presents some contradictory results.

Walkiewicz et al (1991) treated Michigan magnetite, martitic hematite, specular hematite and Minnesota taconite ores in a 2.45 GHz microwave at 3 kW. Optical evaluation of the treated samples showed that stress cracking does occur in the iron ores after 25 s of microwave treatment. It was also found that based on energy savings only, microwave treatment is not cost effective.

However, a decrease in grindability, will result in less wear of the mill, mill liner and milling medium. An increase in the mill throughput and decrease in the recycled ore will also result.

Salsman et al (1996) showed the heating rate of sulfide ores to be directly proportional to the mass of high loss material - indicating that shielding of mineral grains does not affect the rate of microwave heating. It was also shown that the heating rate was directly proportional to the grain size of the sulfide ores.

4.3.2) The Effect of Microwave Power on Mineral Treatment

Dobson et al, 1992, conducted experiments with a variety of minerals to determine the optimum frequency to heat different minerals. It was shown that minerals may have specific frequencies where energy may be absorbed more efficiently. Because the telecommunications industry utilises such a wide band of frequencies, ISM&D applications have been restricted to frequencies of 915 MHz and 2.45 GHz, so it is unlikely that the optimum microwave heating frequency for any mineral will ever be utilised.

Worner et al (1989) reported that only approximately 70% of the electrical power input into a microwave can be utilised. However, Worner et al (1989) considered that the many advantages of microwave power outweigh the low electrical efficiency.

The advantages of microwave heating in the minerals industry include (Worner et al, 1989) :

- If minerals can be heated by microwave radiation, heating to very high temperatures in a short period of time is possible.
- Generally, minerals couple better with microwaves as the temperature increase.
- Lower bulk temperatures are required for high temperature reactions.
- Microwaves in the pyrometallurgical industry enables quick start-up and shutdown which would assist in increasing the degree of control

- Selective heating of mineral ores.

McGill et al (1988) conducted experiments to determine the effect of microwave power level on the heating rate of minerals. It was found that, except for low-loss materials (such as SiO_2 and $CaCO_3$ which does not heat well) or high loss materials (such as PbS or Fe_3O_4 which heats extremely well), the heating rates increase with an increase in power level.

4.4) Patents in the Mineral Processing Field Utilizing Microwave Technology

4.4.1) Microwave Treatment of Metal Bearing Ores and Concentrates

Tranquila (1996) filed a patent for “bringing about a metallurgical effect in a metal-containing ore or concentrate” by treating the ore or concentrate in a microwave cavity with a maximised electrical field strength in the region occupied by the ore.

Tranquila, 1996, states that the cavity and magnetron needs to be tuned to maximise the electric field strength inside the ore or concentrate. In addition to this, it is mentioned that in contrast to previous methods, this method seeks to minimise the energy dissipation in the process while maximising the field strength in the cavity.

In short, the inventor proposes to process a “thin stream of said ore rapidly through a resonant microwave cavity". The process will be able to treat ore with particles smaller less than 6 mm in size and a concentrate containing particles smaller than 75µm. The residence time of the ore/concentrate in the 50kW microwave unit will be around 6 seconds.

4.4.2) Apparatus and Method for Processing Dielectric Materials with Microwave Energy

Haagensen et al, 1985, filed a patent for the continuous heating, drying, curing and de-infesting a wide variety of materials. The process utilizes a number of tunable cavity devices, fitted with a movable piston, arranged vertically with a dielectric conduit running vertically through each cavity. The purpose of the movable piston is to tune the microwave in each cavity to ensure the maximum possible efficiency. Various microwave power levels and frequencies may be chosen for the best possible operation.

4.4.3) Process for the Recovery of Copper from its Ores

Kruesi et al, 1980, filed a patent for the recovery of copper from its ores in conventional processes requiring heat, which is generated by microwave energy.

Conventional mining and processing of copper ore, requires vast amounts of energy, as the ore typically has to be ground very finely to ensure liberation. In addition to which, the ore still has to be smelted or converted to copper. Many processes rely on the oxidation of copper sulphide concentrates. Often, the copper sulphides are overlain by mixed copper oxide, copper sulphide ore, in which case recovery of copper is difficult. However, such ores may be treated with a chlorine ion donor, in the absence of air, to form soluble copper chlorides from which copper recovery is easier. Kruesi et al, 1980, proposes the use of microwave energy to heat the copper minerals to reaction temperatures in order for the formation of water soluble chlorides.

CHAPTER 5

MICROWAVE TREATMENT OF NEVES CORVO ORE

(Appendix A : Somincor Trials is on the CD at the back of this thesis)

5.1) Introduction

Somincor Pty Ltd is located in the Iberian Pyrite Belt, in the Alentjo region of southern Portugal, approximately 220 km southeast of Lisbon. The ore body, basically a massive polymetallic sulphide body, was discovered in 1977 and is similar to other deposits in the Pyrite Belt, but its high copper and tin grades make it unique (Byrne et al, 1995, Somincor, 1994).

More than 150 000 tonnes of copper metal in a 25% copper concentrate are produced annually from approximately 1.8 million tonnes of massive sulphide ore mined (Byrne et al, 1995; Somincor, 1994).

Systematic exploration of the western parts of the Iberian Pyrite Belt began in 1972. This led to the identification of several gravimetric anomalies. Analysis of the drill cores led to the discovery of the Neves ore body in 1977, situated 350 m below the surface, and the Corvo ore body, 500 m below the surface. During 1978 and 1979 identified the Graça and Zambujal ore bodies. In 1988, the Lombador ore body was discovered.

The Neves-Corvo ore bodies can best be described as lenses of massive sulphides - similar to the other deposits in the Pyrite Belt, but unique due to the high copper and tin grades and the strong metal zonation.

The sulphide lens in the Corvo ore body is 30 m at depths of between 230 m and 800 m, overlain by barren pyrite. Cassiterite, the main tin ore, is closely associated with the copper ores. Zinc mineralization develops laterally to the southeast of the copper and tin ores within the massive sulphide.

Linked to the Corvo by a “bridge” of continuous sulphide mineralisation, is the 80 m thick Graça ore body at a depth of between 230 m and 450 m. Following the trend in Corvo, massive sulphide tin ores occur through the copper ores.

The Neves ore body, consists of two lenses joined by a thin bridge. The maximum thickness of the body is 55 m and is approximately 350 m below the surface. The southern lens contains predominantly zinc ore with high lead, silver and copper grades, underlain by tin bearing copper ore. The northern lens consists of predominantly copper ore.

5.2) Processing of Somincor Copper Ore

Approximately 1.4 million tonnes per year is processed at the mine. The processing circuit at Somincor consists of two parts. The first is the comminution circuit and the second part is the flotation circuit. In the comminution section, run-of-mine ore (underground crushers crushing to -250 mm) is fed to the primary crusher, the product of which is screened at 19 mm. The oversize feeds the closed circuit secondary crusher whereas the undersize feeds the rod mill. From the rod mill the ore is milled in 2 ball mills to a product pulp containing 35% solids with an 80% passing size of 35 micron. This is the feed to the flotation section.

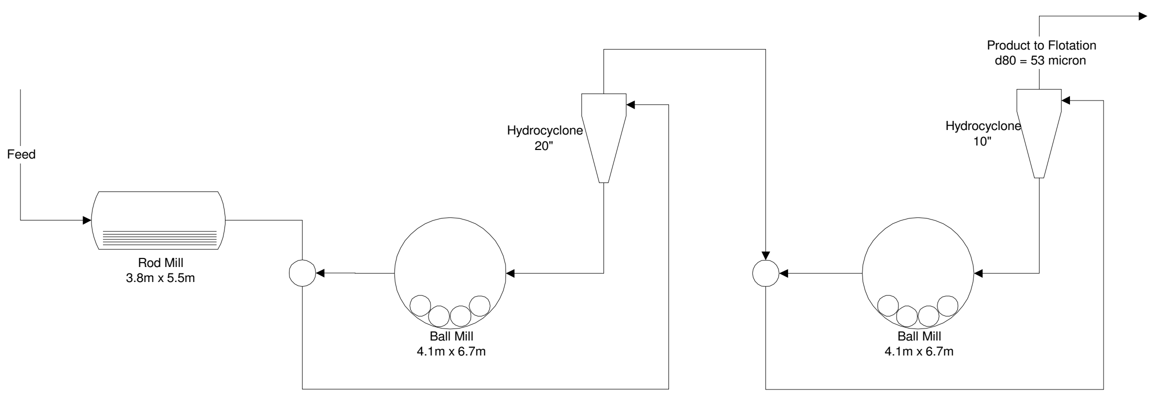


Figure 5.1 : Schematic representation of the milling circuit at Somincor

The pulp is conditioned and pH adjusted before entering the scalping section of the flotation cells. Approximately 40% of the final concentrate is obtained from the scalping section. The tails is fed to the conventional rougher and cleaning circuit. Tailings from the rougher is pumped to a thickener ahead of the scavenger.

The scavenger concentrate and tailings from the first cleaner are pumped to a regrind section (2 closed circuit ball mills), producing a product of 80% passing size of 20 micron. This is fed to a separate circuit, the concentrate joining the rougher concentrate in the cleaner circuit. The tailings join the rougher tail in the thickener and the scavenger tail is pumped to the tailings dam.

Tin is also mined at the mine and after the initial crushing stage, the ores are each fed to its own grinding and flotation circuits. Copper is also recovered from the tin processing plant, but this is not in the scope of the current work.

At present, only the massive copper (MC) ore is mined. However, large massive copper zinc (MCZ) deposits are present, but have yet to be mined and processed. Part of the project is to quantify the effect of microwaves on the 2 different types of ore and the effect on the processing. Therefore, a detailed mineralogical evaluation have been carried out on representative samples of MC and MCZ material (by Rio Tinto Technical Services).

5.3) Mineralogy of the MC and MCZ Ores

Even though the composition of the MC and MCZ material are similar, a mineralogical examination on each is necessary. Small changes in mineralogy may result in a completely different response to microwave radiation.

5.3.1) Mineralogy of the MC Ore

The MC material consists mainly of chalcopyrite ($CuFeS_2$) and pyrite (FeS_2) together with lesser amounts of sphalerite (ZnS), galena (PbS), arsenopyrite (FeAsS), tin bearing phases and gangue. Ferroan dolomite (ideally $CaMg(CO_3)_2$) and quartz (SiO_2) comprises the bulk of the transparent gangue in the head sample. These occur as discrete, euhedral crystals that represent growth in open pore spaces, which have typically been filled by the more common minerals such as chalcopyrite. This is shown in Figure 5.2A.

Pyrite is the major gangue mineral and typically occurs as sub rounded grains and subhedral crystals, which may exhibit a degree of fracturing. These fractures are commonly filled by chalcopyrite, galena or sphalerite, with chalcopyrite occurring along the grain boundaries. The rounded pyrite grains and crystals occur within chalcopyrite rich regions, indicating that the pyrite may have been subjected to a degree of dissolution and replacement by the chalcopyrite, as is shown in Figure 5.2B.

The dominant ore mineral is chalcopyrite which occurs mainly along fractures and along the grain boundaries of pyrite. Aggregates of chalcopyrite may exceed several millimetres in diameter. The chalcopyrite has been subjected to a degree of recrystallization and remobilization forming simple grain boundaries with galena, sphalerite and stannite

(Cu_2FeSnS_4). Where galena, sphalerite and stannite are present in minor amounts, they occur as film like aggregates along the margins of chalcopyrite grains as is shown in Figure 5.2C and 5.2D.

The dominant zinc bearing mineral is sphalerite, occurring as recrystallised aggregates within chalcopyrite. The small amounts of lead present in this sample occur as fine-grained, disseminated galena. The presence of discrete tin bearing phases is characteristic of the Neves Corvo ores. These euhedral crystals and granular aggregates generally occur as finely disseminated grains. The second common tin bearing mineral is stannite, which may be locally abundant (Figure 5.2C). The main arsenic bearing mineral is arsenopyrite, occurring as euhedral crystals exhibiting acicular and rhomb shaped morphologies. These particles are fine grained with a small degree of dissolution and replacement by chalcopyrite (Figure 5.2D).

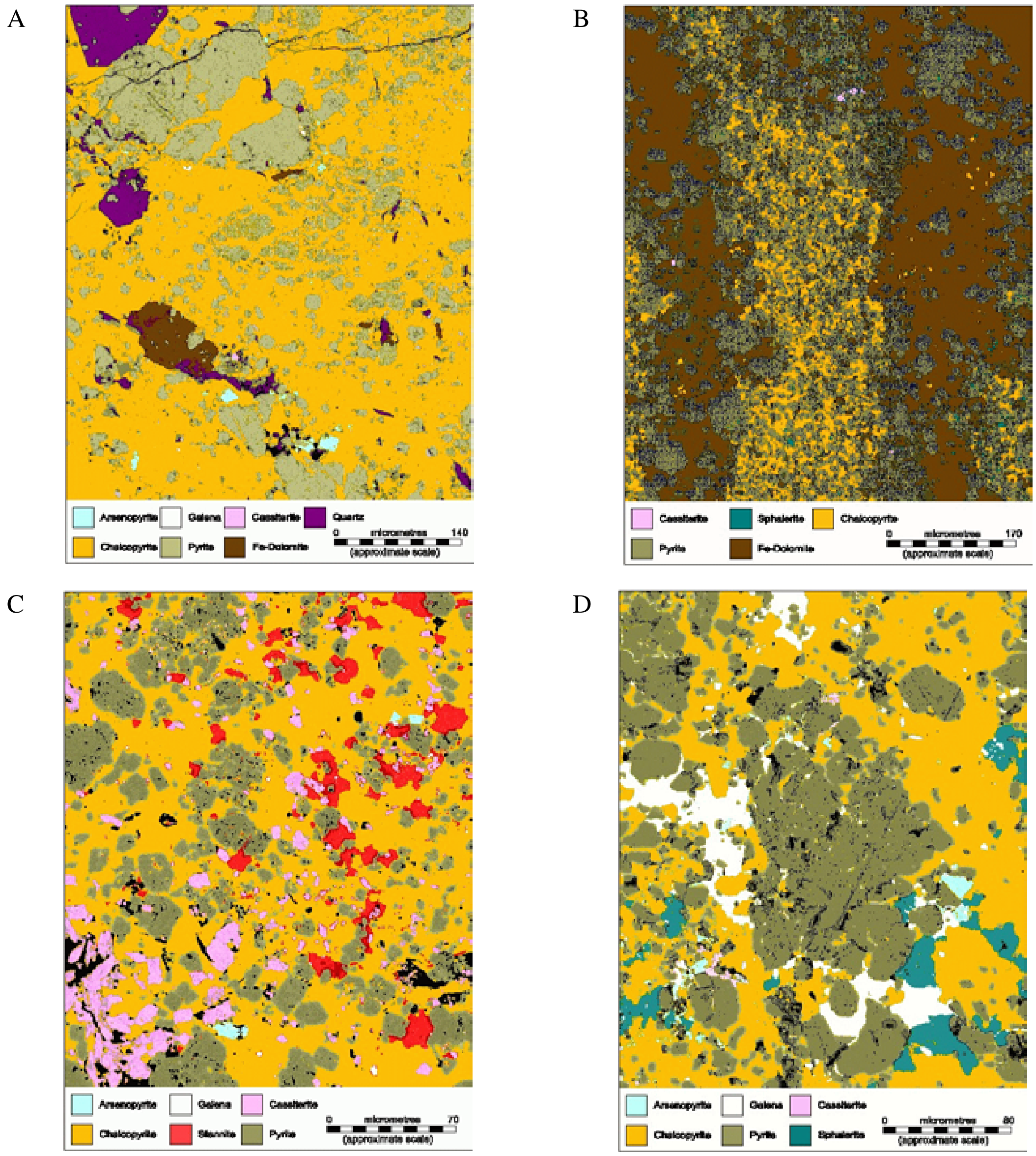


Figure 5.2 : False coloured backscattered SEM images of untreated MC material.

A) Euhedral crystals and granular aggregates of pyrite associated with chalcopyrite

B) Discrete chalcopyrite and pyrite bands with ferroan dolomite filling in former pore spaces.

C) Local abundance of cassiterite (as discrete crystals and aggregates), and stannite, present along the grain boundaries of chalcopyrite forming film-like aggregates.

D) The grain boundary relationships between chalcopyrite, galena and sphalerite.

5.3.2) The Mineralogy of MCZ Ore

The general mineralogy of the material is similar to the massive sulphide ore discussed in Section 5.3.1. However, the MCZ material is particularly rich in Zn and consists mainly of pyrite and sphalerite with lesser amounts of chalcopyrite, galena, arsenopyrite and transparent gangue. Trace amounts of cassiterite and members of the tetrahedrite-tennantite solid solution series (ideally $Cu_{12}Sb_4S_{13}(Cu,Fe)_{12}As_4S_{13}$) are also present.

Siderite ($FeCO_3$) and muscovite ($KAl_2(Si_3Al)O_{10}(OH)_2$) are present in minor quantities. Muscovite is the dominant transparent gangue mineral, occurring as fine grained micaceous aggregates often intimately intergrown with one or more of the sulphide minerals (Fig 5.3A). Siderite typically occurs within the sulphide rich aggregates or in association with the muscovite (Fig 5.3A).

Pyrite is the dominant gangue minerals and typically occurs as granular aggregates and euhedral crystals (Fig 5.3A-D). The bulk of the pyrite have been subjected to some degree of recrystallisation.. The pyrite is generally fine-grained in nature with discrete crystals rarely exceeding 100µm in maximum dimension. The larger pyrite aggregates are typically fine-grained with discrete crystals less than 50µm in size. These aggregates are more abundant in former open pore spaces that have largely been filled by transparent gangue, sphalerite, galena and chalcopyrite (Fig 5.3B). Pyrite may contain some fractures which have been filled by one or more of chalcopyrite, galena and sphalerite.

Arsenopyrite, the major arsenic bearing mineral, is common but subordinate to pyrite, typically occurring as euhedral crystals exhibiting acicular and rhomb-shaped morphologies. Grain size varies between 50μm and 5μm , with chalcopyrite, galena and sphalerite occurring along the margins of the crystals of arsenopyrite.. Arsenopyrite may be locally abundant, occurring as aggregates of crystals closely associated with pyrite. Minor amounts of Sb may also be present.

Sphalerite is the dominant Zn-bearing mineral and is relatively abundant. It generally occurs as fine-grained, granular aggregates present along the margins of pyrite and arsenopyrite grains and crystals filling former pore spaces and fractures (Fig 5.3B). The sphalerite is often intimately intergrown with galena, which is present along the margins of the sphalerite grains. Where galena is abundant the sphalerite occurs as disseminated grains ranging in size between 5 and 20μm in size. When galena is not abundant sphalerite occurs as narrow film like aggregates along the margins of the sphalerite grains.

Chalcopyrite may occur as finely disseminated grains in sphalerite aggregates the grains rarely exceeding a few micrometres in size. The presence of small amounts of fine-grained chalcopyrite in sphalerite is often referred to as chalcopyrite disease. Most of the chalcopyrite in this sample have been subjected to recrystallisation and occurs as film like aggregates along the margins of sphalerite grains (Fig 5.3D).

Chalcopyrite is the dominant copper-bearing mineral and occurs along the grain boundaries of pyrite and arsenopyrite, filling former pore spaces and fractures. Chalcopyrite may be locally abundant as large, granular aggregates, exhibiting simple grain boundary relationships with sphalerite and galena (Fig 5.3B and 5.3C)

The main Pb-bearing mineral is galena which occurs along fractures and grain boundaries of the pyrite and arsenopyrite grains. It is also often associated with one or more of chalcopyrite, sphalerite and rare tetrahedrite. When associated with sphalerite it occurs as narrow film-like aggregates. Galena may be locally abundant where it typically exhibits a high degree of recrystallisation and may form polycrystalline intergrowths with abundant granular sphalerite (Fig 5.3C).

A small number of accessory minerals are present. Cassiterite occurs as tiny, micrometre sized euhedral crystals and rounded grains which are finely disseminated throughout the sample. The majority of the cassiterite occurs within the sphalerite-rich aggregates. Minor amounts of Ag is present in the sulphosalts (tetrahedrite-tennantite).

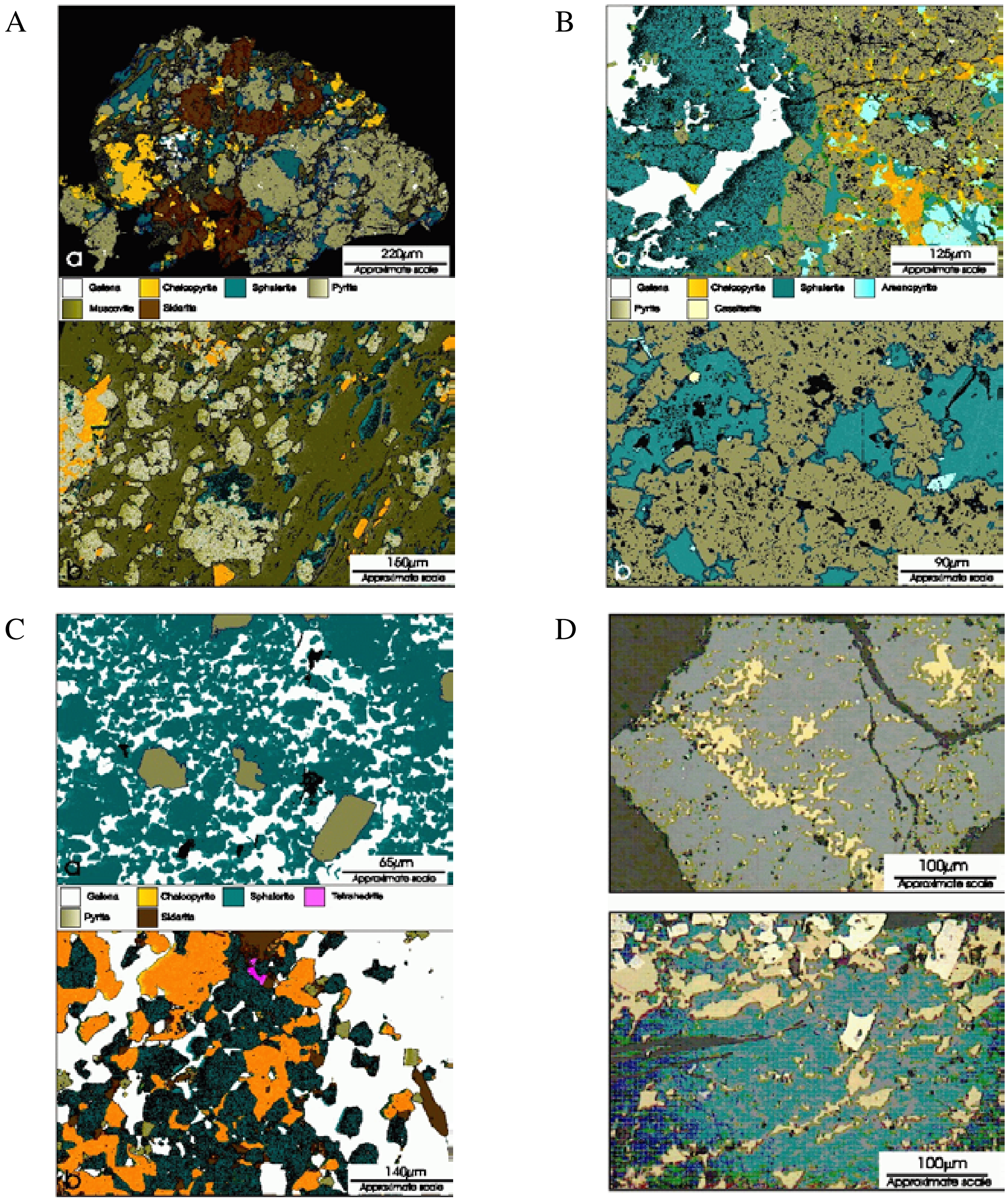


Figure 5.3 : False coloured back scattered SEM images of untreated MCZ material

A) The nature of muscovite and siderite present in pyrite-sphalerite-chalcopyrite rich rock fragments

B-a) The presence of sphalerite and chalcopyrite along the margins of pyrite rich aggregates

B-b) A sphalerite rich area containing arsenopyrite and cassiterite.

C) Granular sphalerite rich areas with chalcopyrite and galena along the margins.

D) is a colour light reflected photomicrographs of film-like chalcopyrite along sphalerite grains.

5.4) Experimental Procedure

5.4.1) Investigation into the Effect of Microwave Radiation on Mineralogy

Experimental work started with the quantification of the effect of microwave radiation on the mineralogy of Somincor copper ore. Samples of 50g were exposed to microwave radiation for different time periods in a 1.5 kW monomode microwave unit. Although further tests were conducted in a 2.6 kW multimode cavity, these small samples caused severe arcing. This was overcome by using a water cooled magnetron and monomode cavity. The 80% passing size of the MC and MCZ material was 6300μm and 5700μm respectively.

5.4.2) Effect of Microwave Radiation on Grindability

Not all ores respond to microwave radiation in the same way (Kingman et al, 2000). To determine whether the MC and MCZ material are affected by microwave radiation and to quantify any possible changes in grindability, representative samples were exposed to microwave radiation for different time periods. After the treatment, the samples were water quenched and dried before being milled in a rod mill. From the size distributions before and after grinding, the change in grindability may be assessed, using the Berry and Bruce method (as discussed in Chapter 3).

Initial trials were conducted on MC material with 80% passing size of approximately 3300μm (particle size distribution shown in Appendix A1. Additional trials were then carried out on MC and MCZ material with 80% passing size of 6300μm and 5700μm respectively (particle size distribution data may be found in Appendix A2 and A3 for the grindability trials conducted on MC and MCZ material respectively).

A rod mill (height = 275 mm, diameter = 155 mm), loaded with 5 rods (height = 265 mm, diameter = 25 mm) was used to determined the relative grindability. The samples, each weighing 500 g were placed in the rod mill and rotated for 5 min at 90 rpm. After the grinding cycle is completed, the 80% passing size was determined by plotting the *(cumulative mass percentage finer than any size)* against the *(size in μm)* - these plots can be seen in Appendix A4.

In addition to grindability trials the Bond Work Index was required. Therefore, a representative sample of the ore was obtained and crushed to 100% passing size of 2800 μm in preparation for the Bond Test. It was determined that 700 ml of the crushed ore, represents a mass of 2584 g. The particle size distribution of the -2.8 mm material can be found in Appendix A5. The procedure followed to determine the work index have been described in Section 3.3 in Chapter 3.

5.4.3) Effect of Microwave Radiation on Flotation Characteristics

Microwave radiation may reduce the flotability of the ore to such an extent that flotation is no longer an economically viable concentrator option. Therefore, flotation trials were conducted on the low zinc Somincor material to determine and quantify any reduction in flotation efficiency.

In practice the 80% passing size of the ore for flotation is approximately 53 μm, with a slurry pH of 10.5 (adjusted using calcium hydroxide). Byrne et al (1995) conducted experiments using Aero3501 (Iso Amyl Diothiophosphate) and KAX (Potassium Amyl Xanthate) as collectors and MIBC as frother. Various dosages was used to determine the effect on reagent concentration and type on recovery. A brief summary of reagent type and concentration is given in Table 5.1 :

Table 5.1 : Summary of collector concentration trials on Somincor ore (after Byrne et al, 1995)

Collector and Concentration		*Conclusion*
KAX	100 g/t	KAX gives higher Cu recoveries than Aero at the same concentration. Aero may be better for selective flotation of chalcopyrite. Stagewise addition of collector is preferred due to the separate production of 2 concentrate streams (high and low antimony)
	150 g/t	
	200 g/t	
Aero3501	100 g/t	
	150 g/t	
	200 g/t	

The samples to be used for flotation analysis were crushed to an 80% passing diameter of 53μm. A Denver batch flotation cell have been used for all experiments. Two and a half litres of water was added to the three litre cell. The ore sample (450g) was the added to the water and the stirrer switched on. The slurry was conditioned for three minutes to allow for particle wetting - adjusting the pH to 10.5 using lime (plant practice). The collector, KAX (Potassium Amyl Xanthate), was then added at a dose of 300 ppm and two minutes extra was allowed for conditioning.

Five drops of the frother MIBC (Methyl IsoButyl Carbinol) were then added and the slurry conditioned for one minute more. The air flow was turned on and the first concentrate was then obtained by collecting the froth for one minute (keeping the water level approximately constant). The second and third concentrate was obtained in the same manner for the same time period. The air flow was then turned off, 300 ppm of collector was added and the slurry left for two minutes for conditioning while the pH was readjusted to 10.5. After two minutes, three more drops of MIBC was added as frother. One minute more was then allowed for conditioning.

Scavenging was carried out for two minutes and the tails collected for three and a half minute (by which time the froth was barren). For the MCZ material, the scavenging and tail collection was combined for five minutes.

The collected froths were then filtered and dried overnight before being analysed for copper content by acid digestion (Donaldson, 1973; Schoeler and Powel, 1955). Representative samples (between 0.25 and 0.9 g each) were obtained from the dried froths. Each sample was digested by 10mℓ of a 50% HNO_3 solution on a hot plate until only a dry pulp remained. The pulp was dissolved in 25 mℓ dilute (10%) HNO_3 solution. The resulting solution was diluted and analysed by atomic adsorption. The digestion trials were the repeated to ensure repeatability of the method as well as accuracy. In addition, selected samples were analysed independently by SGS Laboratories.

5.5) Results and Discussion

5.5.1) Effect of MW Radiation on Mineralogy

<u>Effect of Microwave Radiation on the Mineralogy of MC Ore</u>

The majority of grains in the 15s microwave-treated sample were not affected by the radiation. A small number of pyrite grains have been altered to a Fe-S phase along the boundaries (shown in Figure 5.4A). The Fe-S phase is a common alteration product of pyrite according to the reaction :

$$2\ FeS_2 + 2\ O_2 \rightarrow 2\ FeS + SO_2$$

Changes due to microwave radiation occurs mostly at the margins of the ore fragments. A small number of chalcopyrite grains have undergone partial melting with the development of gas cavities and an increase in Fe content. The increase in Fe content seems to be related to the partial melting of the associated pyrite grains.

Higher power magnification of the partially melted chalcopyrite confirms that the altered chalcopyrite is not homogenous but consists of tiny rounded grains of a Cu rich Fe-S phase. At temperatures greater than 557°C chalcopyrite decomposes to form a mixture of pyrite and a Cu-Fe-S solid solution (the intermediate solid solution). A number of pyrite grains exhibit extensive replacement by the Fe-S phase (Figure 5.4A) Microwave treatment for 45 seconds yields an increased number of altered particles. A large number of ore fragments illustrates partial melting, extensive replacement and fracturing. Generally, chalcopyrite-rich ore fragments undergoes either partial melting or fracturing (Figure 5.4B).

A number of particles shows the development of bornite and covellite phases on the margins of altered chalcopyrite-rich fragments (Figure 5.4C). After 75 s of microwave radiation, extensive partial melting have occurred within the chalcopyrite aggregate. These aggregates typically exhibit highly irregular morphologies and numerous gas cavities. Relict sphalerite, galena, and stannite grains survive within these fragments. In some cases stannite and sphalerite have been subjected to partial melting. A small percentage of the dolomite has also been subjected alteration. Dolomite exhibits alteration along the grain boundaries. Microwave radiation of the samples for 90s yields similar products as in the case of 75s treatment, although the proportion of altered fragments are slightly higher. Bornite and covellite alteration products are present in a number of partially melted chalcopyrite-rich aggregates. A Si-rich glass phase was also observed (Figure 5.4D).

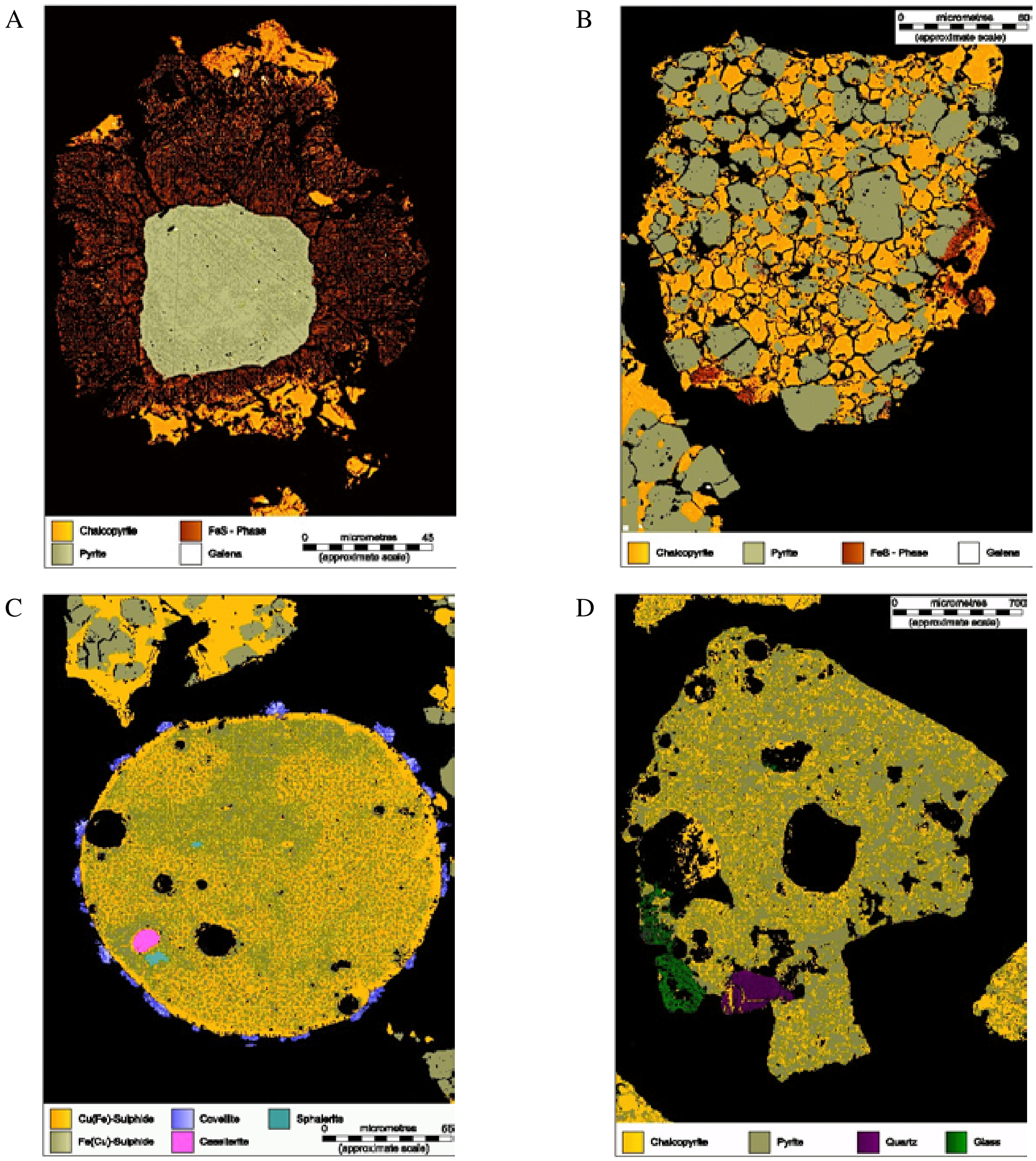


Figure 5.4 : False coloured backscattered SEM images of exposed MC ore

A) A former pyrite grain which have been extensively replaced by the Fe-S phase
B) The extensively fractured nature of an altered pyrite-chalcopyrite aggregate after exposure.
C) The intermediate solid solution and pyrite alteration phase with gas cavities and a covellite-like phase along the grain boundaries.
D) The partial melt products of a pyrite-chalcopyrite grain with quartz and Si-rich glass.

Effects of Microwave Radiation on the Mineralogy of MCZ Ore

Extensive alteration of the pyrite has occurred. A significant proportion of the ore has been altered to a Fe-S phase - similar in composition to pyrrhotite ($Fe_{1-x}S$) as is shown in Figures 5.5A and 5.5B. Although the Fe-S alteration product exhibits a variety of textures, it typically occurs as a fine grained and porous aggregate resulting from the removal of sulphur from the pyrite during microwave treatment. Locally the pyrite may have been subjected to more extensive alteration and the formation of subordinate amounts of iron oxide within the porous Fe-S aggregates (Fig 5.5A-a). The Fe-S phase may occur as rims on partially altered grains or as fine grained partially altered pyrite rich aggregates. The Fe-S phase may completely replace pyrite within an aggregate containing other phases that appear to be unaltered suggesting that pyrite is particularly susceptible to microwave treatment (Fig 5.5B-b).

The fine grained and porous Fe-S aggregates commonly contain minor amounts of unaltered sphalerite, chalcopyrite and galena. These phases may exhibit partial alteration in the form of fracturing along the grain boundaries, partial dissolution as well as compositional variation and remobilisation (Fig 5.5C)

Galena is mainly unaltered by microwave exposure except for the formation gas cavities in larger ore fragments. In addition the galena have been subjected to some degree of remobilisation and partial melting. The melted galena may cement together particles of pyrite and sphalerite. Remobilisation may also cause the galena to fill in the fractures along the grain boundaries of altered sphalerite and chalcopyrite aggregates.

Sphalerite, may exhibit a marked degree of fracturing occurring along the sphalerite grain boundaries (Fig 5.5B). This is particularly evident in sphalerite-rich aggregates. The fracturing may relate to the reduction in volume associated with the removal of sulphur from the sphalerite during microwave treatment.

Chalcopyrite exhibits a wide range of textures indicative of microwave treatment. It may occur as relatively unaltered grains within extensively altered pyrite aggregates. The bulk of the chalcopyrite exhibits little or no partial melting - but partial melting may occur with no change in textural or compositional evidence (Fig 5.5C). Fracturing along the grain boundaries of the chalcopyrite is present in minor amounts.

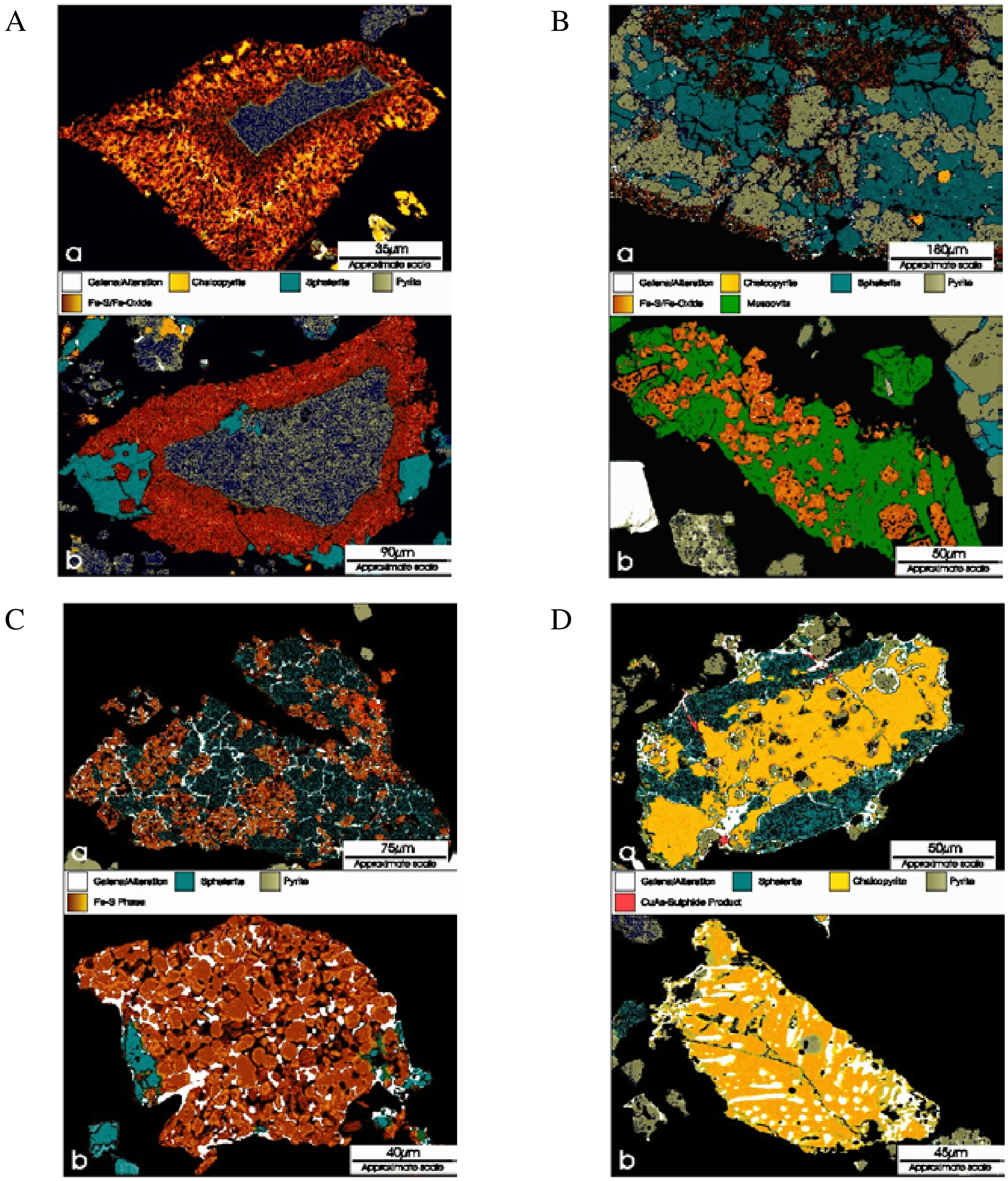


Figure 5.5 : False coloured backscattered SEM images of exposed MCZ material

A-a) The incipient replacement of pyrite by the alteration product Fe-S.

A-b) Unaltered chalcopyrite is also present in the rim.

B) The replacement of pyrite by the alteration product as well as the fracturing of sphalerite

C) The remobilisation of As bearing galena filling in fractures in a sphalerite aggregate.

D) The occurrence of partially melted chalcopyrite, galena and sphalerite aggregates.

5.5.2) Effect of Microwave Radiation on Grindability

<u>Calculation of Bond Work Index</u>

The -75 µm particles were removed after each grinding cycle (i.e. P_1 = 75 µm in Eq 3.9). Table 5.2 summarizes the data obtained for the work index (an Excel worksheet containing the full set of results and calculations may be found in Appendix A6) :

Table 5.2 : The results of the Standard Bond Work Index Test for MC ore from the Somincor Mine (green shaded cells show that the last three grinding cycles gave similar circulating loads - as required for the calculation of the Bond Work Index).

A	B	C	D	E	F	G	H
		MILL FEED			MILL DISCHARGE		
Grinding Cycle	New feed (g)	<75 µM (g)	Revs	<75µM Present	<75µM Produced	Undersize (g/rev)	% Circ Load
1	2584.072	178.553	50	226.680	48.12656156	4.53	1039.96
2	226.668	15.663	200	305.15	289.487	1.45	746.82
3	305.15	21.085	250	580.11	559.025	2.24	345.45
4	580.11	40.084	300	658.44	618.356	2.06	292.45
5	658.44	45.497	325	782.56	737.063	2.27	230.21
6	782.56	54.07	312	767.38	713.31	2.29	236.74
7	767.38	55.747	312	806.78	751.033	2.41	220.29
8	806.78	51.79	304	749.52	697.73	2.30	244.76
9	749.52	50.864	305	736.12	685.256	2.25	251.04
10	736.12	50.864	304	731.32	680.456	2.24	253.34

Using these values, a Bond work index of 9.7 kWh.t^{-1} was determined for the MC material (using Equation 3.9). The accepted values on the plant is between 12 and 14 kWh.t^{-1}.

The value determined does not fall in this range of values, however, as the work index depends on the individual components of the ore body, it is thought that the sample used to determine the work index was not a representative sample of the ore body. Due to the grinding characteristics of the two material types - it took approximately te same time to grind to the same particle size distribution - it was concluded that the work index of the MC material is the same as that of the MCZ material.

The Effect of Microwave Radiation on MC and MCZ Ore

From the Berry and Bruce method discussed in Chapter 3, the relative work index may be calculated from :

$$W_{it} = W_{ir} \frac{\frac{10}{\sqrt{P_r}} - \frac{10}{\sqrt{F_r}}}{\frac{10}{\sqrt{P_t}} - \frac{10}{\sqrt{F_t}}} \qquad \text{Equation 3.11}$$

(where P and F refers to the product and feed 80% passing sizes). In this case, the reference ore (indicated by the subscript r) is the untreated material and the test ore (indicated by the subscript t) refers to the material exposed to microwave radiation. To illustrate the effect of microwave radiation on the 80% passing size of the material, a plot of cumulative fraction vs particle size (µm) is shown below :

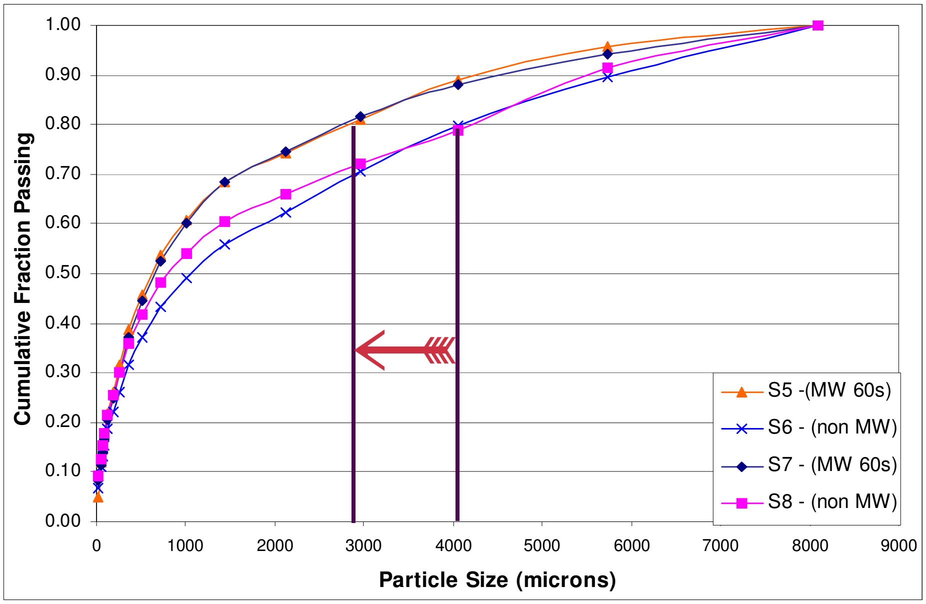


Figure 5.6 : Difference in particle size distribution after milling between samples exposed to microwave radiation (S5 & S7) and reference samples (S6 & S8).

Figure 5.6 illustrates two important points. Firstly exposure to microwave radiation for 60s has the effect of reducing the 80% passing size after milling for five minutes from 4050µm to less than 2800µm. This corresponds to a reduction in work index (using Eq 3.11) of 40%. Secondly it is shown that the experiments are repeatable. This is important as it verifies that the experimental procedure is precise.

The change in work index have been calculated for all different exposure times for the MC and MCZ microwave trials (particle size distributions are shown in Appendix A4) and the results are shown in Figure 5.7 and 5.8 :

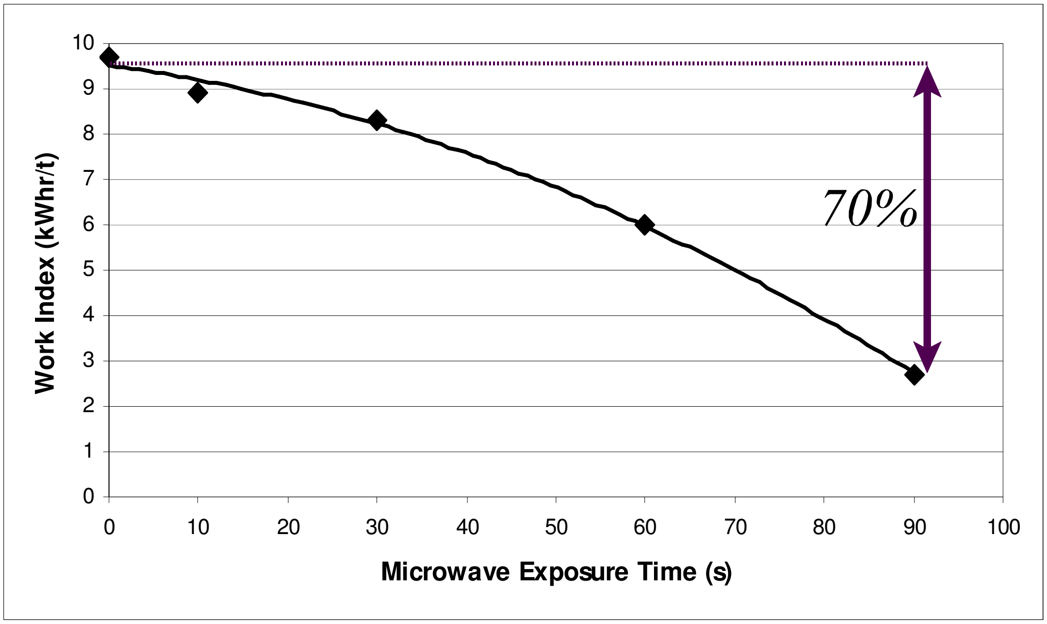


Figure 5.7 : Work index versus microwave exposure time for MC ore

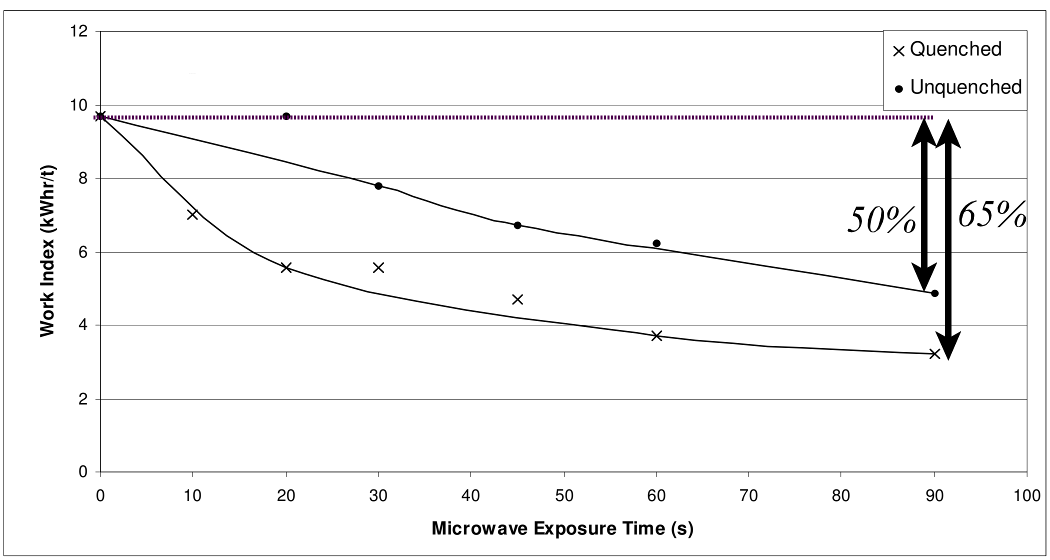


Figure 5.8 : Work index versus microwave exposure time for MCZ ore showing the effect of quenching on work index reduction.

For the MC ore the reduction in work index after 90s exposure to microwave radiation is 70%. For MCZ ore the reductions after 90s of microwave exposure are 50% for the unquenched ore compared to a reduction of 65% for water quenched samples.

The reason for the large reduction in work index is that extensive intergranular fractures occur during microwave treatment and subsequent quenching (Figure 5.4A, 5.4B, 5.3A, 5.5B and 5.5C). The thermal stresses induced during microwave heating weakens the ore structure and makes it more amenable to comminution processes.

Comparing the reduction of work index versus treatment time for the low and high zinc, reveals that the initial reduction in work index is much faster for the high zinc material. This may be explained by the increased sphalerite content in the MCZ material. In the MC material the main ore components are pyrite and chalcopyrite, which initially heat at similar rates, giving rise to little differential expansion, the primary cause of fracture.

However, for the high zinc material, there are large quantities of sphalerite (not a good heater) present. Therefore, stresses between pyrite and sphalerite and chalcopyrite and sphalerite gives rise to larger initial reductions in work index.

Using the MCZ material, the effect of loading on the effectiveness of microwave pre-treatment was investigated. The results are shown in Figure 5.9 :

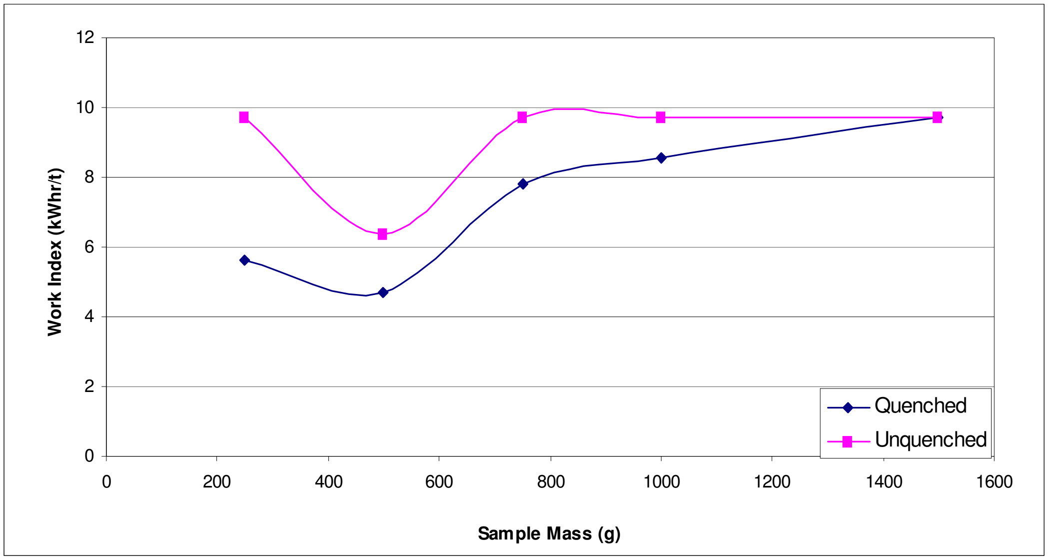


Figure 5.9 : Work index versus microwave exposure time for MCZ ore showing the effect of loading during microwave exposure on work index reduction (exposure time in all cases was 45s at 2.6 kW).

The observed trend, for both quenched and non-quenched material is a maximum reduction in work index at 500g sample mass with progressively smaller reductions in work index as the sample mass increase - with the smallest reductions for the case of non-quenched material. The reduction in work index for the 250g in both cases are slightly higher than the reduction for the 500g sample. The fact that the reduction in work index decreases with an increase in mass loading, makes sense intuitively. More energy is required to heat increased loads. The reason for the lower reduction in work index in the case of the smallest sample size requires a closer look at the mineralogy of the samples exposed to microwave radiation for prolonged periods of time.

Figures 5.4D and 5.5D illustrate partial melt products as well as the formation of a glass phase (formed as a result of the melting of quartz). Both of these affects the strength of the material. In addition, there is a critical mass associated with microwave treatment of materials. At masses below a certain threshold mass, treatment becomes ineffective.

5.5.3) Effect of Microwave Radiation on Flotation Characteristics

The first result to be obtained from the flotation trials are the mass recovery per stage, as shown in the graphs in Appendix A7. However, flotation efficiency is quantified by the recovery of the valuable mineral, not by mass recovery alone. The copper content of each stage of flotation was determined by acid digestion and atomic adsorption (results in Appendix A8).

Shown in Figures 5.10 and 5.11, are plots of the copper recoveries versus flotation time for a sample exposed to microwave radiation for 60s and a reference sample for the MC material and quenched and unquenched copper recoveries after 45s microwave exposure for the MCZ material :

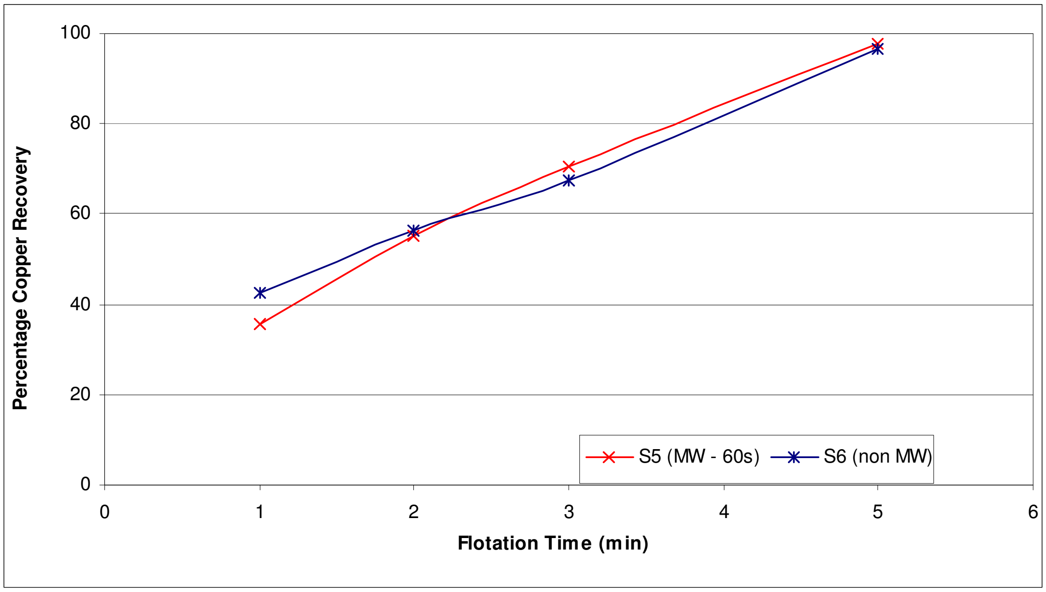


Figure 5.10 : Percentage copper recovery versus flotation time for MC material.

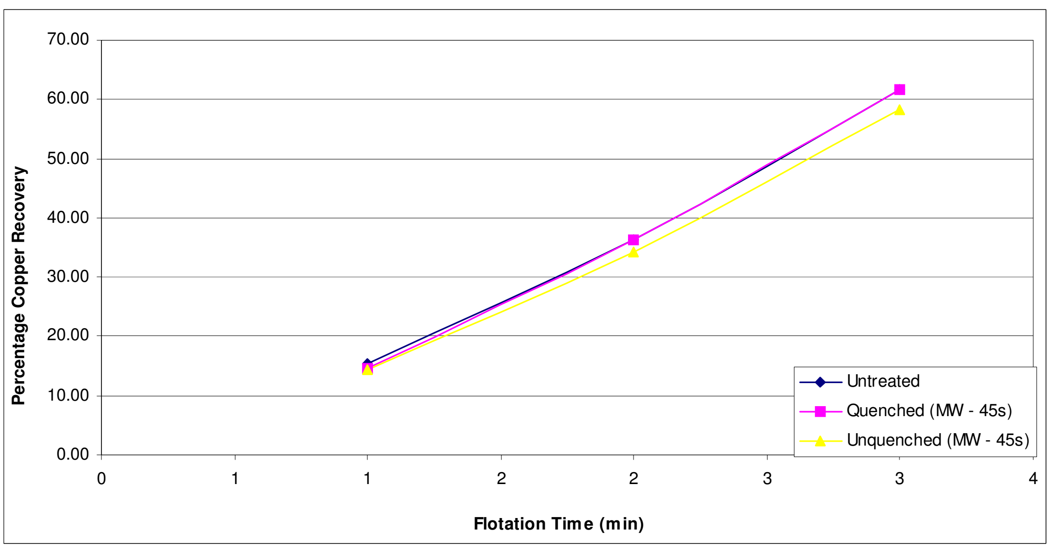


Figure 5.11 : Percentage copper recovery versus flotation time for MCZ material.

Figure 5.4C shows the formation of a covellite-like phase on the particle surface. Figure 5.4A and 5.5 A shows the formation of a Fe-S alteration product replacing the pyrite phase. All these mineralogical changes suggest that the surface properties of the material would have changed as well.

As flotation is a surface chemistry dependent process, it was initially thought that these surface changes would affect the flotation characteristics of the ore. The results of laboratory scale flotation trials have shown that this is not the case.

No significant effect on the copper grade after flotation can be observed. Microwave exposure does not seem to affect the hydrophobicity of ore. This is of vital importance, as even small changes on the flotability of an ore will significantly change the economics of the process. Negative implications of microwave radiation on the flotability of the ore would have voided any cost reductions in the grinding circuit.

5.6) Conclusion

The work index of the reference low zinc material was determined to be 9.7 $kWh.t^{-1}$. The variation from the accepted values of 12 and 14 $kWh.t^{-1}$ indicates that the work index is variable across the geological breadth of the ore deposit. If the material used during laboratory testing have been mined at a different location or are not representative of the ore body, the calculated work index of 9.7 $kWh.t^{-1}$ will be feasible.

From the grindability trials, it is concluded that the material is responsive to microwave radiation. For the MC ore the reduction in work index after 90s exposure to microwave radiation is 70% (all MC-samples were quenched after microwave treatment, due to lack of samples, these trials could not be repeated for the unquenched case). For MCZ ore the reductions after 90s of microwave exposure are 50% for the unquenched ore compared to a reduction of 65% for water quenched samples.

No significant effect on the copper grade after flotation can be observed. Microwave exposure does not seem to affect the hydrophobicity of ore. This is of vital importance, as even small changes on the flotability of an ore will significantly change the economics of the process. Negative implications of microwave radiation on the flotability of the ore would have voided any cost reductions in the grinding circuit.

The following papers regarding work in this chapter have been published/accepted for publication :

1) "The effect of microwave radiation on the processing of Neves Corvo ore" by Vorster, Kingman and Rowson in International Journal of Mineral Processing, 2001, vol 63, p 29-44.
2) "The influence of mineralogy on microwave assisted grinding" by Kingman, Vorster and Rowson, Minerals Engineering, 2000, vol 13, no 3, pp 313-327, Pergamon
3) "Applications of microwave radiation for the processing of minerals" by Vorster, Kingman and Rowson in the Proceedings of the 9th SAICHE National Meeting, 2000

These papers have been included in the back of this thesis, under the "Papers"-section. In addition two presentations have been delivered regarding this work (both of these presentations have been included on the CD at the back of this thesis) :

1) "The effect of microwave radiation on the processing of Neves Corvo ore" was delivered at the UMIST Research Day in 1999 (umist99.exe)
2) "Applications of microwave radiation for the processing of minerals" was delivered at the 9th SAICHE National Meeting in October 2000 (saiche00.exe)

CHAPTER 6

THE EFFECT OF MICROWAVE RADIATION ON FE-TI ORE FROM THE MAMBULA COMPLEX

(Appendix B : Mambula Trials is on the CD at the back of this thesis)

6.1) Introduction

Richards Bay Minerals, RBM, was formed in 1976 to exploit the minerals wealth of the dunes surrounding Richards Bay in south-eastern South Africa. It is jointly owned by Rio Tinto plc (50%) and Billiton plc (50%).

The presence of relevant heavy metal minerals (for the production of Zircon, high purity iron and titanium) in beach sand were first reported in the 1920's, but a detailed investigation of the 17 km long, 2 km wide coastal area was only carried out in 1971. The mining rights to the Mambula ore reserve (north and south of the original deposit) was acquired in 1985. Currently only beach sand is commercially mined, using a dredge mining operation (RBM Ltd, 2000).

Original trials by RBM produced poor results for the recovery of ilmenite and high recoveries of magnetite/titanium magnetite of the Mambula complex. Microwave radiation have been suggested as a possible method for increasing the liberation of the granular ilmenite (RBM Ltd, 2000, Personal Communication : Kevin Pietersen, 2000).

The major minerals in the Fe-Ti ore from the Mambula complex are magnetite, ilmenite, hematite and pleonaste (aFe-Mg spinel) with clinochlore (a chlorite), magnesiohastingsite (a Ti-bearing Amphibole), diaspore (Al-oxyhydroxide) comprising the minor mineral component of the ore body. Trace Minerals consists of gahnite (Zn-spinel), akermanite (CaMg-silicate), periclase (MgO) and pyrrhotite (Fe-sulphide). The ilmenite grade of the ore is between 23% and 30% (14-15% TiO_2) with between 60 and 65% iron oxides. Some of the magnetite contains up to 2% TiO_2, this may be due to lamellae, and also due to some substitution of TiO_2 into magnetite/hematite (i.e. titano-magnetite) (Personal Communication Kevin Pieterse, 2000).

The main liberation required is that of the granular ilmenite from the granular spinel and magnetite. The lamellae and inclusions of magnetite and spinel need not be liberated from the ilmenite. To date only 80% liberation of the ilmenite has been achieved by conventional processing.

6.2) Mineralogy

Two types of material from the Mambula complex have been examined. The first type of material (Referred to as S1) is light grey in colour with little or no oxidation and replacement by hematite. The second type of material (referred to as S2) is darker in colour with a higher degree of oxidation as well as evidence of secondary iron-oxides/hydroxides on the surface of ore fragments. Figure 6.1 illustrates the difference in appearance between the 2 samples.

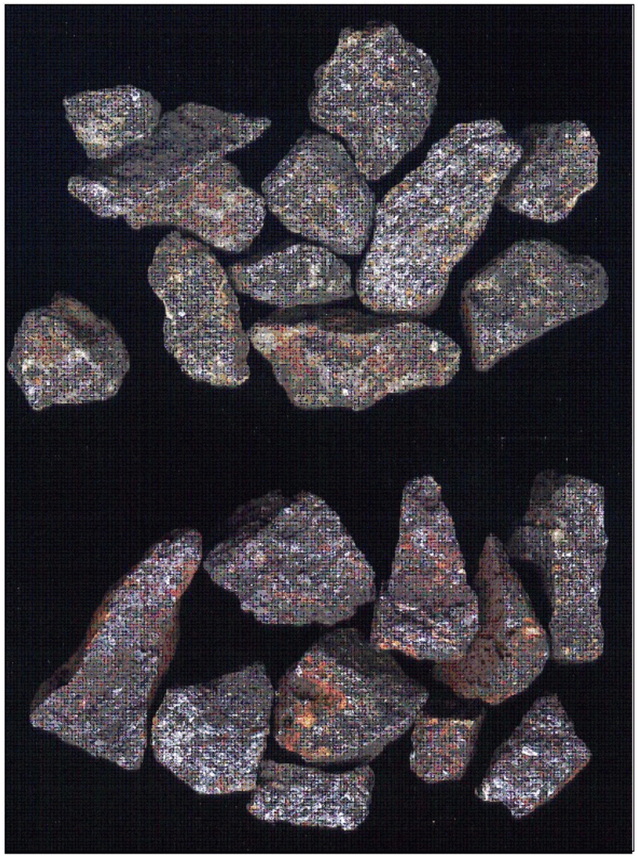


Figure 6.1 : A digitised scanned image of particles from S1 (top) and S2 (bottom). S2 exhibits a darker, more metallic grey colour as well as a greater degree of oxidation than S1.

One of the dominant gangue minerals is plagioclase, which exhibits reaction rims consisting of hornblende [$Ca_2(Mg,Fe)_4AlSi_7AlO_{22}(OH)_2$] and subordinate orthopyroxene [$(Mg,Fe)_2Si_2O_6$]. Another common gangue mineral is clinopyroxene [$(Ca,Mg,Fe)_2Si_2O_6$], which may also exhibit reaction rims or coronas as shown in Figure 6.2 Da. A large number of tiny exsolved pleonaste inclusions (typically $(Fe,Mg)Al_2O_4$) are also present although they rarely exceed a few micrometers in size.

With the increased degree of oxidation in S2 the transparent gangue minerals may exhibit a certain degree of decomposition and replacement by Si-rich clays, which may, in turn, exhibit a degree of replacement by secondary hematite (Figure 6.2 Ab). Abundant within the oxidized ore are secondary Al-hydroxides such as gibbsite [$Al(OH)_3$] and diaspore [AlO(OH)].

The dominant oxide mineral is magnetite. The magnetite exhibits textural features reflecting annealing and exsolution during amphibolite facies metamorphism. The majority of the magnetite is present as large, granular aggregates in the form of polygonal crystals. Ilmenite, also with polygonal morphology, is intergrown with the magnetite. The magnetite crystals typically contain fine grained lamellae of exsolved pleonaste, mostly of micrometer-sized lamellae (Figure 6.2 B). A smaller proportion of the pleonaste exsolution bodies occur as euhedral crystals which may exceed 5μm in size.

The magnetite consists of mainly Fe and O with a small proportion of Ti. The magnetite may also be traversed by ilmenite lamellae that range in size from a few micrometers to several hundred micrometers (Figure 6.2 B&C). The ilmenite lamellae may represent the migration of Ti during crystallization and metamorphism. Figure 6.2 Ca shows the tiny, Ti-rich inclusions in the magnetite surrounding the ilmenite lamellae. The magnetite core is depleted in Ti. Minute pleonaste bodies may be present along the boundaries of the ilmenite lamellae. Minor amounts of magnetite and subordinate ilmenite are present as complex intergrowths in the transparent gangue (Figure 6.2 Cb).

Sample S1 exhibits only a minor degree of oxidation and replacement by hematite - typically along the grain boundaries and fractures (Figure 6.2 Ba). Sample S2 exhibits highly variable, but more extensive oxidation and replacement by hematite (Figure 6.2 A). Hematite formed from the oxidation of magnetite is referred to a martite by a process called martitisation.

Ilmenite, mainly occurring as polygonal crystals intimately associated with the magnetite aggregates (Figure 6.2 A), is common but subordinate in abundance to magnetite. The ilmenite crystals may exceed 100μm in length and typically exhibit lamellar twinning. The ilmenite may have undergone oxidation with the development of micrometre sized hematite lamellae along cleavage planes (Figure 6.2 Aa). However, the majority of the ilmenite exhibits little or no oxidation even in regions of extensive ore oxidation.

Pleonaste (a variety of spinel consisting mainly of Al, O, Mg and Fe)is a common accessory mineral, occurring as relatively large, millimetre-sized crystals and aggregates as well as fine grained exsolution lamellae within titanium bearing magnetite (Figure 6.2 B,C&D). Large, granular pleonaste grains may exhibit some degree of fracturing and subsequent oxidation and replacement by aluminium hydroxide, in particular along the more extensively oxidized ore fragments (Figure 6.2 D). Minute magnetite grains may also be finely disseminated within the pleonaste, possible because of exsolution of excess Fe. Within the Ti-bearing magnetite the pleonaste may occur as tiny, micrometre sized exsolution lamellae (Figure 6.2 B).

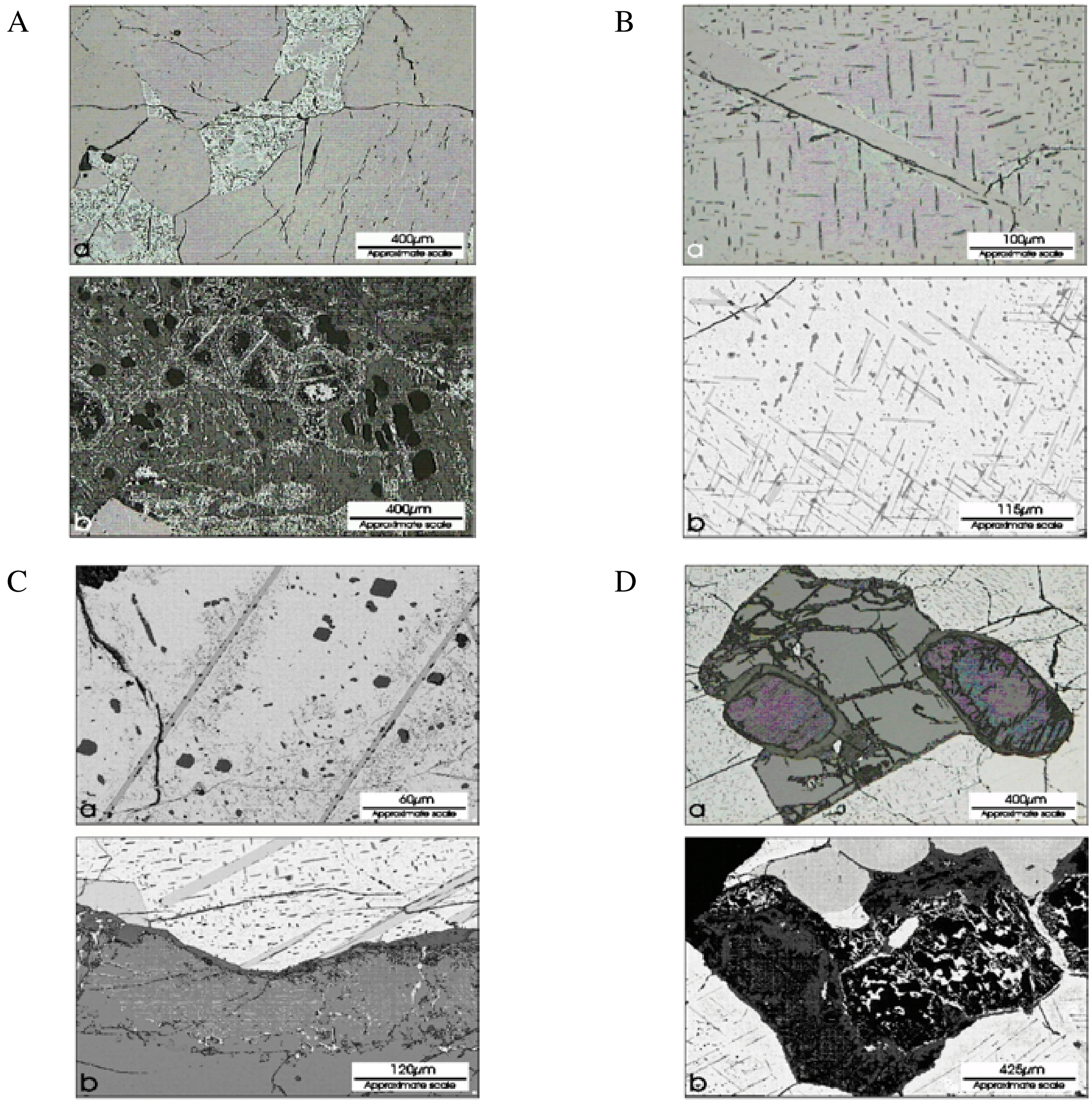


Figure 6.2 : SEM images of untreated Mambula ore

Aa) Extensive oxidation of the magnetite (darker grey) within the ore

Ab) Decomposition and replacement of transparent gangue minerals (dark grey) by hematite (light grey).

Ba) Ilmenite lamella (pale, pinkish brown) present in the magnetite (light grey).

Bb) Tiny ilmenite (medium grey) and pleonaste (dark grey) within the magnetite.

C) Migration of Ti-rich inclusions (mottled patches) to 2 ilmenite lamella (medium grey) in a Ti-bearing magnetite grain (light grey).

D) Nature of a large, fractured pleonaste (medium grey) grain is shown within polygonal magnetite (light grey). Reaction rims may be observed around the clinopyroxene grains (medium grey).

Db) Gangue (dark grey) replaced by hematite (light grey/white).

6.3) Experimental Procedure

6.3.1) Mineralogy

Microwaves may change the mineralogy of the ore to such an extent that mineral processing is no longer feasible (Kingman et al, 2000; Vorster et al, 2000). In order to determine whether changes to mineralogy did occur and to quantify the type of changes, 500g samples of the material were exposed to microwave radiation for 90s in a 2.6 kW multimode microwave cavity and the products examined using a scanning electron microscope.

6.3.2) Grindability

The 'as-received' material was too large for mineral processing (+10cm). They were crushed in a laboratory scale jaw crusher to a 80% passing size of 15mm. The first set of trials involved samples made up of the full particle size distribution (-19.6,+0mm) and two samples were exposed to microwave radiation for 60s before being water quenched after heating and dried overnight. The samples were then milled in a rod mill and the particle size distribution determined at different time intervals. Because the majority of the fine material is lost when quenching occurs, the next set of trials involved only material greater than 1.7mm in diameter and samples were exposed to microwave radiation for 90s. In addition, milling was carried out in a ball mill as opposed to a rod mill. The final set of experiments consisted only of -9.6+1.7mm material. After milling, the particle size distributions were determined by sieve analysis and the effect of microwave treatment on grindability assessed (the initial particle size distributions are shown in Appendix B1).

6.3.3) Magnetic Susceptibility

Two stage magnetic separation trials were then conducted on 50g sub-samples. The milled product was screened at 1.18 mm and the fines were riffled (on spinning riffles) to obtain the sub-samples. It was decided to carry out the magnetic separation trials wet, as this would overcome the problems of bridging in the high intensity separator as well as being more efficient.

The 50g material was then mixed with water (approximately 500mℓ) before being subjected to low intensity magnetic separation, the purpose of which was to remove the highly magnetic magnetite (forming the first concentrate - C1).

The low intensity stage was followed by high intensity wet separation at 0.8T. The pulp was fed to the high intensity separator, the magnetic fraction of the material forming the second concentrate (C2) and the non-magnetic fraction the tails (T).

The first and second concentrates as well as the tails were then filtered in a pressure filter, before being dried overnight. After drying, the material was riffled and small samples (between 0.5 and 1g) were sent for FeO and Ti analysis.

6.4) Results and Discussion

6.4.1) Effect of Microwave Radiation on Mineralogy

Examination of a large number of polished sections prepared from samples exposed to microwave radiation revealed no significant textural and mineralogical changes. Some surface oxidation, particularly of the finer grained materials (-4+2mm size fraction) was evident, together with apparent dehydration of the associated Fe-hydroxides (Figure 6.3 A). These features are, however, relatively rare and may represent natural oxidation of the particles.

Textural changes between treated and untreated ores are not readily discernible because of the high degree of variability both within and between the two head samples. Much of the fine-grained pleonaste lamellae within the treated ores appear to be finer than that of the untreated ore, possibly representing the partial resorbtion of the aluminous spinels by the magnetite at elevated temperature, but it may also simply reflect sampling variability.

A direct result of the microwave treatment were observed in the form of glass-rich fragments (Figures 6.3 B). These glass-rich fragments contain gas cavities that are direct evidence of partial melting of the former silicate minerals. Fine-grained dendritic magnetite crystals together with partially altered relict oxide grains mat be contained within the glass rich fragments. On contact with hematite, reduction appears to have occurred with a fine rim of magnetite being observed (Figure 6.3 Bb). During this investigation only two occurrences of glass were recognised.

A B

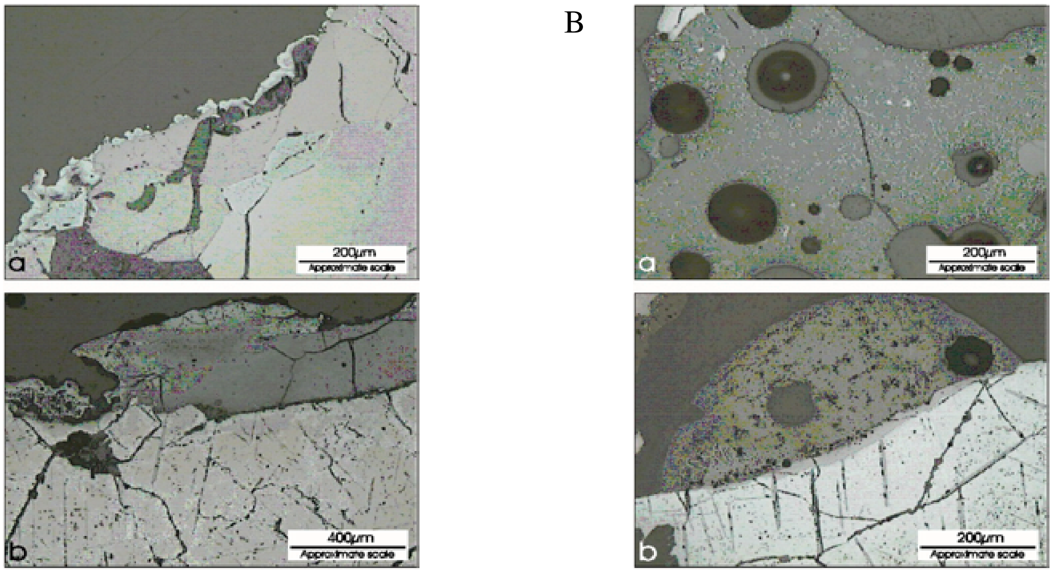


Figure 6.3 : SEM Images of microwave products

Aa) A fine rim of botryoidal hematite (light grey) present along the margins of a magnetite-ilmenite aggregate.

Ab) Rim of Fe-hydroxide (dark grey) present along the margins of a partially oxidised magnetite aggregate (pinkish grey shades). Fe-oxide/hydroxide rims may reflect partial dehydration and oxidation during microwave exposure.

B) Glass (dark grey) containing fine-grained dendritic magnetite (light grey). Note the numerous gas cavities within the glass.

6.4.2) The Effect of Microwave Radiation on Grindability

Figure 6.4 illustrates the work index for the Mambula ore after 90s exposure at 2.6 kW microwave radiation calculated using the Berry and Bruce method after different milling times. This shows that the change in work index is unaffected by milling time. As the initial work indices, W_{i1} and W_{i2}, are unknown (and could not be estimated due to the limited availability of the sample), the work indices of the samples after microwave exposure are indicated as a fraction of W_{i1} and W_{i2}.

Figure 6.4 further illustrates that the work index after microwave exposure has been reduced by 19% for S1 (or $0.81 \cdot W_{i1}$) and by 26% for S2 (or $0.74 \cdot W_{i2}$) (full set of results regarding the particle size distributions are given in Appendix B2).

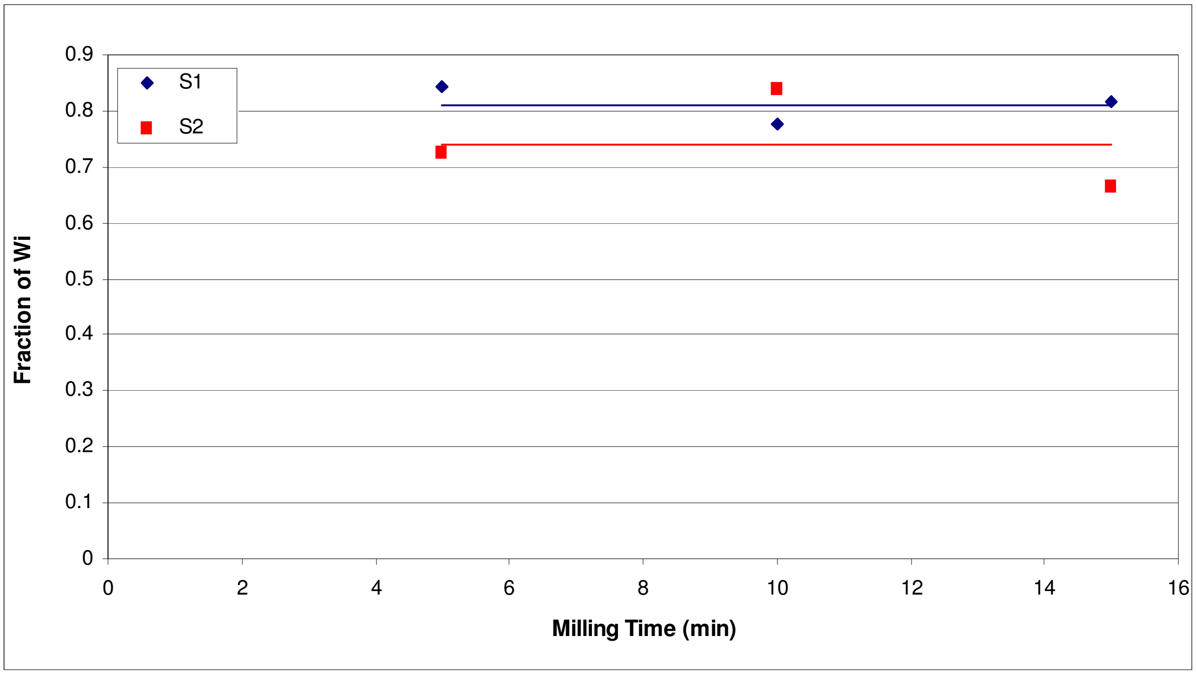


Figure 6.4 : Effect of microwave radiation on work index after different milling times (ball mill)

The small changes in work index are expected as the mineralogy of the ore have not been significantly altered. No fractures, cracks, bubbles or extensive glass phases are observed after 90s of microwave exposure in a 2.6 kW microwave cavity.

Comparison of the 50-70% reduction in work index achieved on Somincor copper ore (Vorster et al, 2001) after 45s exposure in the same microwave cavity, confirms the finding by Kingman et al (2000) that the mineralogy determines whether or not an ore's grindability may be significantly reduced by microwave radiation. Kingman et al (2000) stated that an ore with consistent mineralogy containing good microwave absorbers in a transparent gangue matrix are most responsive to microwave radiation, whereas ores containing small, finely disseminated particles respond poorly. Mambula ore type have a consistent mineralogy of good absorbers (~62% magnetite and ~26% ilmenite - Personal Communication : K Pietersen, 2000) but lacks a transparent gangue matrix. As a result the bulk of the ore heats up at similar rates when heated and intergranular fracture do not occur readily, resulting in small changes in work index.

Although even a 20% reduction in work index may be considered significant regarding energy savings on the comminution circuit, the microwave energy input exceeds the reduction in energy costs by far. Exposure of 500g ore for 90s in a 2.6kW microwave cavity corresponds to 130 $kWh.t^{-1}$. For break-even cost, i.e. where the reduction in work index equals the microwave energy input, the ore would need to have a work index of 650 $kWh.t^{-1}$. Most mineral ores have work indices between 10 and 30 $kWh.t^{-1}$. Extremely difficult to grind and hard minerals such as graphite and emery have work indices of only 43.5 and 56.7 $kWh.t^{-1}$ respectively (Wills, 1997).

6.4.3) Effect of Microwave Radiation on the Magnetic Susceptibility

The percentage of iron and titanium recovered in each of the stages during the magnetic separation trails are shown in the bar charts Fig 6.5 (Iron) and Figure 6.6 (titanium) below. Samples S1-2, S2-1 and S2-2 are reference samples and have not been exposed to microwave radiation whereas S1-3, S1-4, S2-3 and S2-4 have all been exposed to microwave radiation for 90s (full set of results in Appendix B3).

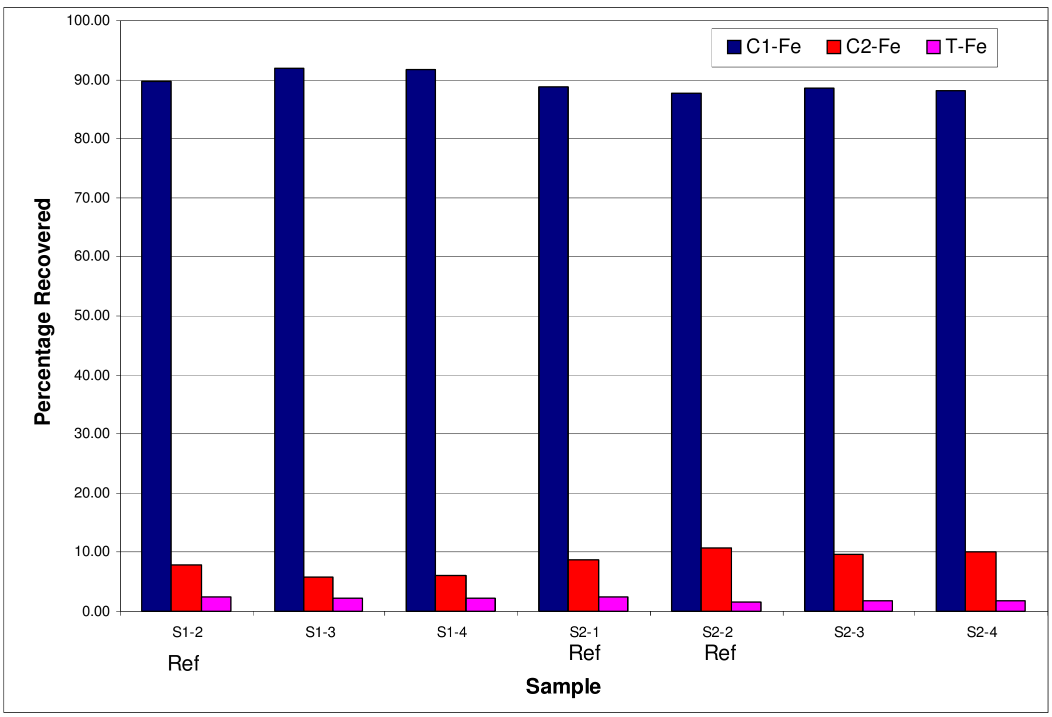


Figure 6.5 : Bar chart showing the recovery of iron of the different samples

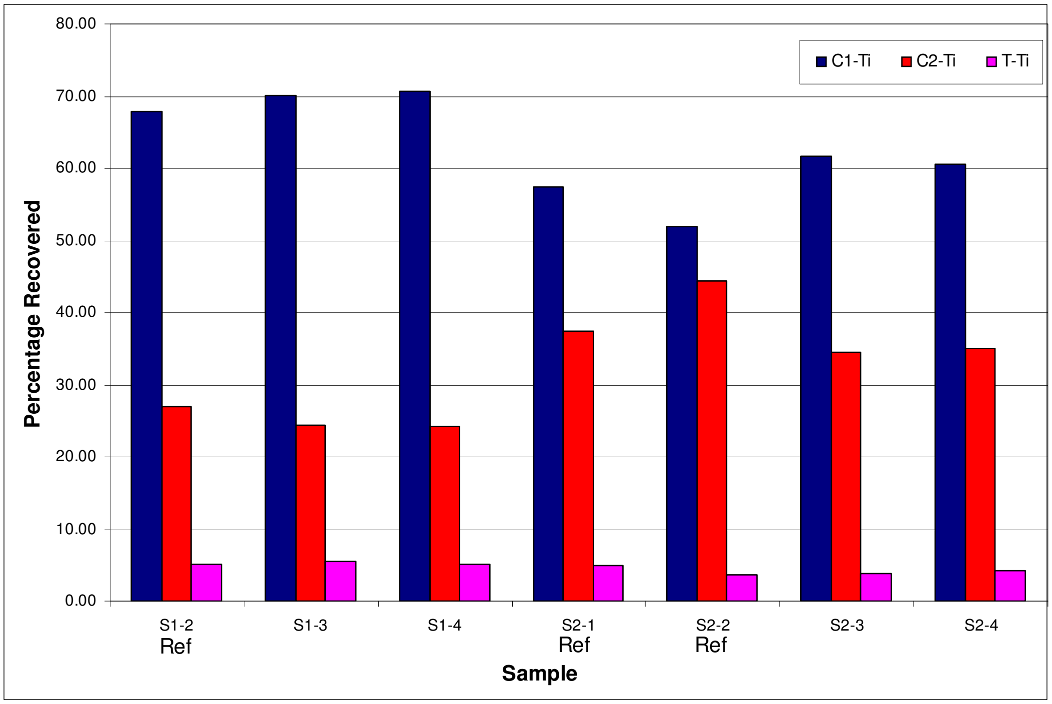


Figure 6.6 : Bar chart showing the recovery of titanium of the different samples

No significant change in Fe-recovery between samples exposed to microwave radiation and the reference samples can be observed. There is some improvement in Ti-recovery in C1 (unwanted). However, this would need to be confirmed by extensive further test work. The Ti content in C2 (0.8T concentrate) appears to drop. This may be due to changes in magnetic susceptibility of the ilmenite phase.

Again the mineralogy confirms the results from the magnetic separation trials. No significant mineralogical change occurred as a result of microwave radiation. Therefore there will not be significant differences between microwave exposed and unexposed material.

6.5) Conclusion

The reduction in work indices of the 2 samples from the Mambula-complex after 90s exposure to 2.6 kW microwave radiation are 19% for S1 and 26% for S2. The small magnitude of the changes after such a prolonged exposure time as well as no significant structural or mineralogical changes of the ore (confirmed by the SEM examination) suggests that no further investigation into possible reductions in work index is required. Partial melting and a small degree of oxidation did occur, but these did not affect the grindability significantly.

The microwave energy input into achieving a 20% reduction in work index is 130 $kWh.t^{-1}$. This again confirms that microwave radiation is not the solution for this problem.

There is no evidence of changes in magnetic susceptibility of the mineral species within the sample as a result of microwave radiation. Again this is confirmed by the mineralogy. Oxidation did occur to a small extent, as did partial melting and the formation of gas bubbles. But these changes are not frequent throughout the sample and do not affect the magnetic susceptibility of the Mambula ore.

CHAPTER 7

THE EFFECT OF MICROWAVE RADIATION ON PALABORA VERMICULITE

(Appendix C : Vermiculite Trials is on the CD at the back of this thesis)

7.1) Introduction

Phlogopite, muscovite and biotite are the most common species of the mica group. All minerals in this group have perfect crystallographic basal cleavage and, as a result, are “flaky”. The individual flakes adhere to form “books” and are pliable, resilient and tough with extremely smooth surfaces and high reflectivity. Individual flakes may be as thin as 20µm. All the minerals in the mica group have extremely low electrical and heat conductivities [Hamilton et al, 1974; Mineral Data, 1999].

Vermiculite, a hydrated laminar magnesium-aluminium-ironsilicate, is formed by the hydration (weathering) of phlogopite, essentially by the loss of alkali and addition of water [Hamilton et al, 1974] :

$$Mg_3\ (Al,Si)_4\ O_{10}\ (OH)_2 \cdot 4H_2O$$

When heated, vermiculite has the unusual property of exfoliating perpendicularly to the cleavage plane by up to 3000% (maximum). An unexfoliated “book” (thickness approximately 2mm) as well as exfoliated books (approximately 10mm thick) are illustrated in Figure 7.1.

Figure 7.1 : Shown above left is an unexpanded vermiculite “book” with some exfoliated “books” shown on the right [Mandoval Ltd, 1999].

A close-up of a single book (350× magnification) is shown in Figure 7.2 to illustrate the nature of the single layers comprising a “book”.

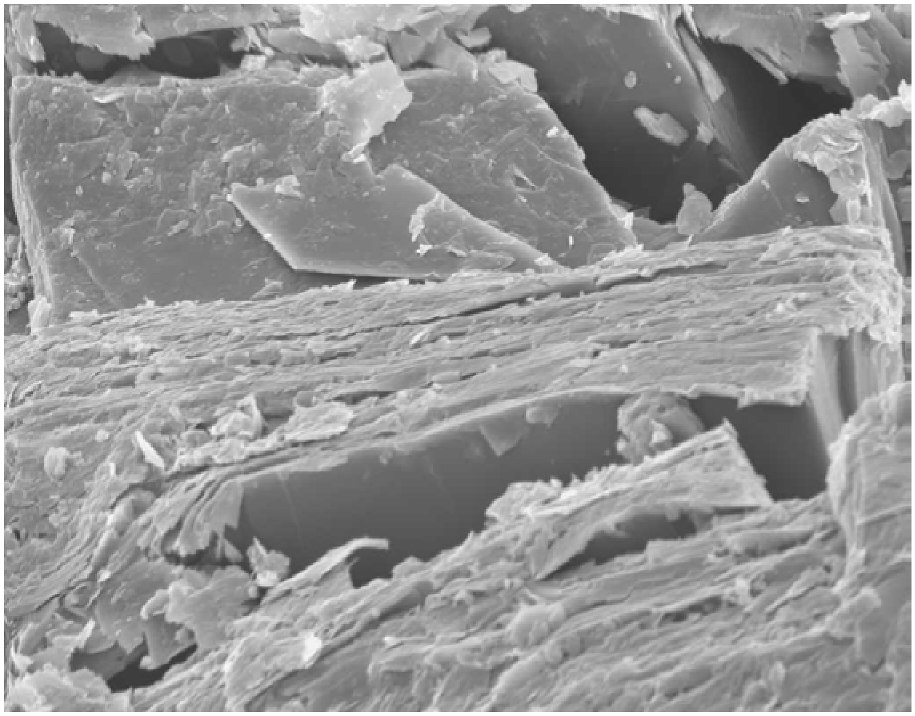


Figure 7.2 : 350× magnification of a single book illustrating the layers comprising a “book”

The general process for the expansion involves the release of water from the mineral lattice. Conventionally this is achieved by heating the vermiculite to temperatures in excess of 800°C until a suitable degree of exfoliation has occurred. This is an energy intensive process, usually accomplished in rotary kilns.

In general, micas are transparent to microwave radiation. In this aspect, however, vermiculite is unique amongst the micaceous minerals. Exposure to microwave radiation results in rapid exfoliation of the vermiculite layers without heating the bulk of the material to high temperatures.

7.2) Mining Operation at Palabora

In excess of two million tonnes of vermiculite ore is mined per annum, containing approximately 20% vermiculite. Vermiculite is mined from three sources at Palabora.

The first is the Palabora Vermiculite Operation Department (VOD) open pit (the ore body consists of a serpenetised olivine-phlogopite pegmatoid surrounded by a diopside-phlogopite pegmatoid). Up to a depth of 50m the phlogopite has been weathered to vermiculite, mainly through a leaching process whereby the Mg and K have been replaced by water [PMC Ltd, 1996]. The second source is the Phalaborwa Phosphate and Vermiculite (PP&V) deposits, a diopside-phlogopite pegmatoid with the first 50m weathered to vermiculite. The third source of vermiculite is the original VOD plant dump consisting of tailings which have become economically mineable [PMC Ltd, 1996].

Sizing and drying are the main components of the Ore Preparation Plant whereas winnowing occurs in the Ore Treatment Plant. The Ore Preparation Plant consists of a main grizzly situated over a 450 tonne surge bin. From the surge bin the ore is fed to a mineral sizer. From the sized ore, a primary double deck screen (50mm aperture top deck, 20mm aperture bottom deck) is used to retrieve the -20mm material. Oversize material from either deck can be crushed to -20mm or be routed to waste dumps.

The -20mm material is then dried in three rotating coal fired driers, in order to reduce the moisture content to between 6.4% and 4% [PMC Ltd, 1996].

In the Ore Treatment Plant, the vermiculite is progressively screened, winnowed and crushed in eight minor plants - such that five grades of vermiculite is produced Large, Medium, Fine, Superfine and Micron. The particle size distribution and loose bulk density is shown in Table 7.1.

Table 7.1 : Table detailing the grades of vermiculite, their particle size distribution as well as the bulk density [Mandoval Ltd, 1999]

Grade	**PSD (minimum 80% retained - mm)**	**Loose Bulk Density (kg/m^3)**
Large	-8.0+2.8	700 - 850
Medium	-4.0+1.4	800 - 950
Fine	-2.0+0.71	850 - 1050
Superfine	-1.0+0.35	850 - 1050
Micron	-0.71+0.25	850 - 1050

7.3) Uses of Vermiculite

Vermiculite is used in a multitude of industries (Table 7.2) ranging from agriculture to construction to the horticultural.

Table 7.2 : List of some industrial applications of Vermiculite

Agricultural	animal feed	Horticultural	blocking mixes
	anti-caking material		hydroponics
	bulking agent		micro-propagation
	fertilizer		potting mixes
	pesticide		rooting cuttings
	soil conditioner		seed germination
Construction	acoustic finishes	Other	absorbent packing
	air setting binder		brake pads/shoes
	board		drilling muds
	fire protection		filtration
	floor and roof screeds		fireproof safes
	insulating roof screed		insulation
	loft insulation		nuclear waste disposal

7.4) Physical Properties of Vermiculite

Vermiculite has a perfect cleavage plane [001] with a monoclinic crystal system and a Mohs hardness of 1.5. Crude vermiculite from Palabora Open Pit, is golden brown in colour (Figure 7.1) with a loose bulk density of between 700 and 1050 kg/m^3, however when compacted this increases to ±1250 kg/m^3. Exfoliated vermiculite has a range of densities from 60 to 135 kg/m^3, and chemical analysis of crude and exfoliated vermiculite are the same (Hamilton, 1974).

Approximate compositions of major and minor components are listed in Table 7.3 :

Table 7.3 : Chemical analysis of Palabora vermiculite [Mandoval Ltd, 1999]

Major Components	SiO_2	35 - 41%	**Minor Components**	CaO	2.0 - 6.0%
	Al_2O_3	6.0 - 9.5%		CO_2	0.6 - 2.5%
	Fe_2O_3	6.0 - 9.5%		TiO_2	0.6 - 1.4%
	MgO	21.5 - 25.5%		F	0.2 - 0.8%
	K_2O	3.0 - 6.0%		Cr_2O_3	0.01 - 0.15%
				P_2O_5	0.2 - 2.0%
				Cl	0 - 0.5%

Different grades of vermiculite has different volume yields, as specified in m^3 per tonne as is shown in Table 7.4. In addition to the loose bulk density and the volume yield, Table 7.4 also lists the values for the specific surface area (m^2/g).

Table 7.4 : Volume yields and loose bulk densities of exfoliated vermiculite [Mandoval Ltd, 1999]

Grade	Volume Yield (m^3/t)	Loose Bulk Density (kg/m^3)	Surface Area (m^2/g)
Large	13.0 - 14.5	60 - 75	3.8
Medium	11.5 - 13.0	70 - 85	4.0
Fine	10.5 - 11.5	75 - 85	4.4

Superfine	8.5 - 10.0	85 - 100	5.4
Micron	7.5 - 9.0	105 - 135	6.4

The occupational exposure limits for vermiculite according to Mandoval Ltd (1999) are specified as 10hg/m^3 inhalation dust and 1 mg/m^3 respirable dust. Vermiculite is non-combustible, not readily biodegradable, non-toxic and is not classified as hazardous under CHIP regulations 1994.

7.5) Previous Work and Theory

Very little research has been carried out regarding the application of microwave assisted exfoliation of vermiculite. In a 1970 patent, Wada described the application of electromagnetic radiation for the exfoliation of vermiculite with additional benefits such as ion exchangeability, humidity control, high water retention and odour elimination. Wada (1970) further describes that "much less energy is required for a certain expansion per unit volume of vermiculite as compared to hitherto known methods". He further states that the expansion is more effective in the presence of polar molecules or cations with a lower molecular weight than 5000.

Most micas are transparent to microwave radiation. However, vermiculite is a hydrated form of phlogopite. Water is an extremely good microwave receptor. When vermiculite is exposed to microwave radiation, the water molecules in the structure is released and the vermiculite expands. The process may be described as shown below :

$$Mg_3\ (Al,Si)_4\ O_{10}\ (OH)_2 \cdot 4H_2O \rightarrow Mg_3\ (Al,Si)_4\ O_{10}\ (OH)_2 \cdot 3H_2O + H_2O \qquad \text{Eq 7.1}$$

7.6) Experimental Procedure

7.6.1) Exfoliation

Different masses of each of the five grades have been exposed to microwave radiation for different time periods, and the volume of the exfoliated vermiculite was measured in a measuring cylinder. A 2.6 kW multimode cavity, a 1.5 kW monomode cavity as well as a conventional, laboratory scale furnace at 850°C have been used to exfoliate vermiculite samples.

In order to facilitate the exfoliation process of the finer grade material, a small amount of active carbon (2.5g in a 20g sample) was added for the microwave assisted exfoliation trials. As active carbon is an extremely good microwave receptor, the addition of active carbon would result in the mixture being heated much more rapidly initially, possibly increasing the volumetric yield and reducing residence time.

Due to constraints as to available sample mass, furnace size and exfoliated volume only relatively small batches of fine, superfine and micron grade material were exfoliated at any one time. In all cases exfoliation was continued until such time that no more visible exfoliation occurred.

In addition, batches of known mass were exposed to microwave radiation for fixed periods of time and the resulting volume measured. The volume per mass ratio were then plotted against the power per mass ratio in an attempt to determine the optimum exposure time.

7.6.2) The Effect of Microwave Radiation on Vermiculite Structure

As vermiculite is predominantly used for insulation purposes, changes to the thermal conductivity of vermiculite could have adverse effect on its applications in this industry. To assess any possible changes, the structure of microwave exfoliated vermiculite was compared to the structure of vermiculite exfoliated at 850°C using a scanning electron microscope.

7.6.3) Economic Evaluation of the Alternative Exfoliation Process

The power requirements of the process was estimated from the power rating and efficiency of the microwave units and the time used for exfoliation. A pilot scale microwave unit with a conveyer belt, have been used to continuously treat a mixture of large and medium grade vermiculite. The results from this experiment was used to estimate more accurately the economic feasibility of microwave exfoliation.

7.7) Results and Discussion

7.7.1) Exfoliation Trials on Different Grades

The results of the exfoliation trials on the five grades of vermiculite is shown in Table 7.5. The volumetric expansion of vermiculite per unit mass as quoted by Mandoval Ltd is also shown (data in Appendix C1 to C5).

Table 7.5 : Results of exfoliation trials comparing volumetric expansion quoted in literature with experimental volume yields achieved in different microwave units as well as at 850°C.

Grade	**Expansion per unit mass from Mandoval Ltd**	**Experimental values for expansion per unit mass**
Large	13.0-14.5 m^3/t	13.3 m^3/t @ 2.6 kW
Medium	11.5-13.0 m^3/t	7.1 m^3/t @ 2.6 kW
Fine	10.5-11.5 m^3/t	3.3 m^3/t @ 850°C
		4.7 m^3/t @ 2.6 kW
		4 m^3/t @ 1.5 kW
		5.5 m^3/t @ 2.6kW + Active Carbon
Superfine	8.5-10.0 m^3/t	2.0 m^3/t @ 850°C
		1.9 m^3/t @ 2.6 kW
		3.4 m^3/t @ 1.5 kW
		4.0 m^3/t @ 2.6kW + Active Carbon
Micron	7.5-9.0 m^3/t	2.4 m^3/t @ 850°C
		2.4 m^3/t @ 2.5 kW
		2.5 m^3/t @ 2.6kW + Active Carbon

The volume yield per tonne of vermiculite obtained in the laboratory scale experiments for the large grade vermiculite (on average 12.5 m^3/t) correlates well with the recorded values in literature (see Table 7.5). The medium grade vermiculite yielded approximately 7.2 m^3/t, which is less than the value quoted in literature (Table 7.5).

For the three finer grades, experiments were conducted on samples of 10g and 20g. The volume yields for these small samples, does not correlate well with the values in literature. Possible reasons for the poor correlation between literature and experimental results is ascribed mainly to the small sample size used. In order to compare microwave and conventionally exfoliated vermiculite, parallel experiments were conducted in microwave units as well as a conventional laboratory scale furnace. Due to the equipment size and the need to be able to directly compare the results, similar size samples had to be used, which was limited by the smallest piece of equipment.

Thus, although poor correlation between literature and experimental results are observed, comparing similar samples treated on laboratory scale, shows a clear advantage of microwave radiation to be used as the exfoliation method, not only in terms of increased expansion ratio (ratio of expanded volume to unexpanded volume) but also in terms of treatment time and therefore in energy consumption.

If, as is postulated , the exfoliation of vermiculite is due to the release of water from the structure, then it would be expected that no exfoliation would occur during the initial stages of microwave exposure, as the water molecules do not have enough energy to be released from the structure. As soon as the energy level of the vibrating water molecules exceed a threshold exfoliation should start and increase in rate as exposure to microwave radiation continues. When all the water have been released from the structure, no further exfoliation would be possible. In order to investigate this hypothesis, vermiculite have been exposed to microwave radiation for fixed time intervals and the volumetric expansion calculated per energy input (Appendix C6 contains the data). To illustrate, the volume per mass ratio versus power consumption per unit mass graph is shown in Figure 7.3 for coarse grade vermiculite (similar results for the finer grades as can be seen in Appendix C6).

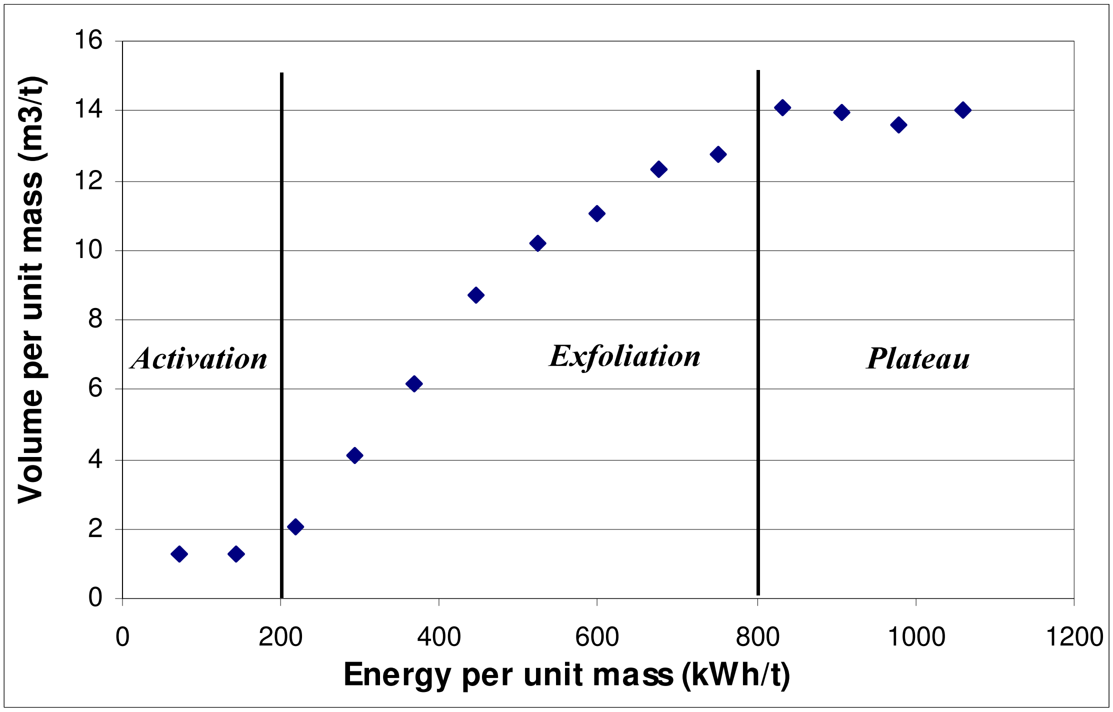


Figure 7.3 : Plot of volume to mass ratio versus energy to mass ratio for coarse grained vermiculite exfoliated at 2.6 kW

Figure 7.3 confirms the hypothesis. Initially, during the activation stage, no exfoliation occurs. This is followed by the exfoliation phase during which the water molecules have gained sufficient energy levels to be released from the structure. Finally, when all the water molecules have been released, no further exfoliation is possible and the curve levels off.

7.7.2) Effect of Microwave Radiation on Structure of Exfoliated Vermiculite

To investigate the possibility that the vermiculite exfoliated by microwave radiation has physical differences to conventionally exfoliated vermiculite, analysis of some particles by scanning electron microscopy was carried out - scanning electron microscope images of microwave exfoliated and thermal (i.e. exfoliation at 850°C) are shown in Figure 7.4.

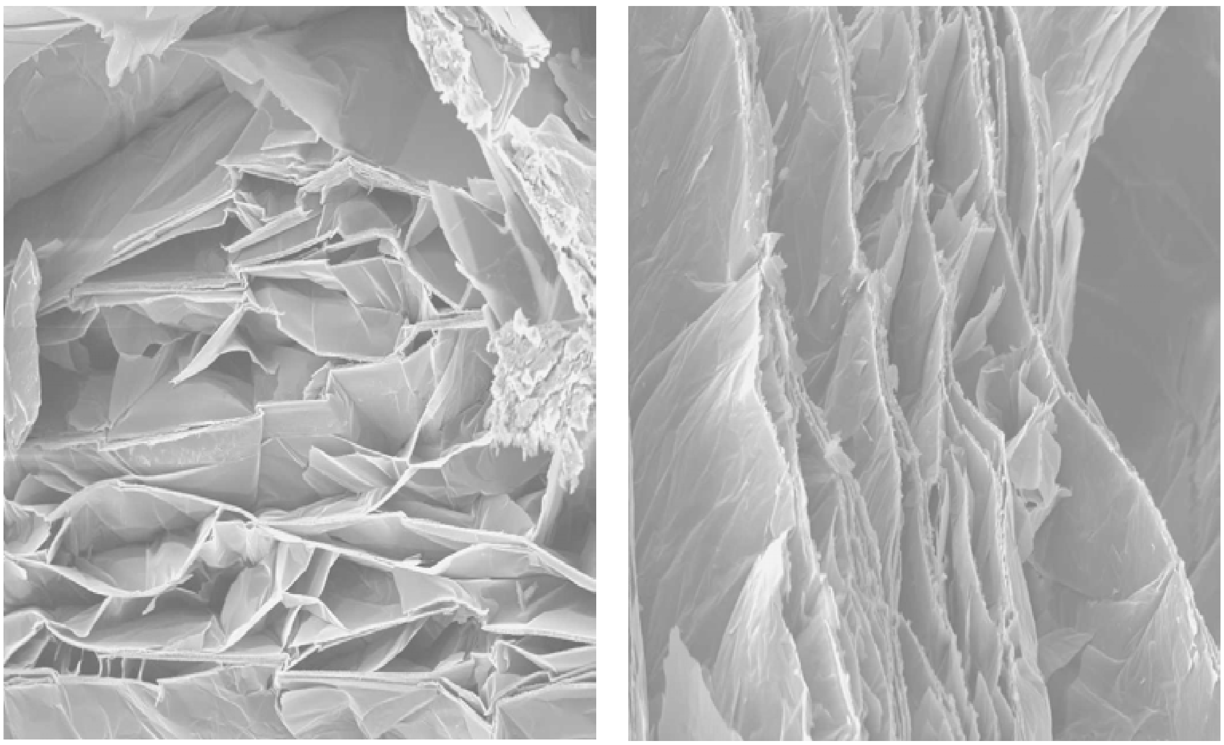


Figure 7.4 : Shown above are two scanning electron microscope images of a microwave exfoliated large grade vermiculite booklet (200×) on the left and compared to a thermally exfoliated large grade vermiculite booklet (350×) on the right.

The vermiculite exfoliated by microwave radiation (on the left in Figure 7.4) shows a highly irregular pattern of flakes, whereas the vermiculite exfoliated in a conventional furnace at 850°C shows a more regular structure (on the right Figure 7.4). This is to be expected as microwave radiation does not heat the bulk material evenly, but only affects the water molecules. In the case of the conventionally exfoliated material, the bulk material is brought to the same temperature, wherefore exfoliation in different parts of the structure will occur at the same rate. It is not currently known whether the irregular pattern of exfoliation affects the physical properties.

7.7.3) Economic Evaluation

Initial pilot scale studies were carried out in a 6.6 kW microwave heater fitted with a conveyer belt. Large grade vermiculite was expanded in this unit, with the required volume yield. In order to justify using a microwave instead of a conventional furnace, the energy requirements for the microwave system must be significantly less than that of the conventional process per unit mass.

Torbed Ltd exfoliates vermiculite in a fluidised bed. This process uses combustion products to fluidise a bed of vermiculite and in the process of heat transfer between the hot gases (at 1250°C) and vermiculite, exfoliation occurs. An energy balance was conducted on an industrial process (Torbed Ltd, 2000) and compared to an energy balance of the microwave system. Torbed Ltd employs 2 vessels for the exfoliation of vermiculite, the Torbed 400 (0.25 t/h) and Torbed 1000 (2 t/h). Both of these processes burns either oil or natural gas, the gases released after combustion is fed to a chamber filled with vermiculite. The speed of the combustion products fluidises and heats the vermiculite to approximately 1250°C.

After exfoliation the products is cooled to approximately 35°C. For the two systems the energy requirements are :

Torbed 400	0.25 t/h	oil fired	1.99 GJ/t
Torbed 400	0.25 t/h	gas fired	1.67 GJ/t
Torbed 1000	2 t/h	oil fired	1.84 GJ/t
Torbed 1000	2 t/h	gas fired	1.24 GJ/t

In addition cooling energy from 1200°C to 35°C requires approximately 1.2 GJ/t. The microwave system upon which the energy balance is based, was a 6.6 kW unit fitted with a conveyer belt. The unit itself has an electrical efficiency of 45%, thus increasing the power draw to 14.5 kW. For the worst case scenario (most energy inefficient), the energy requirements per ton is 352 MJ/t which is 3.5 times less than the energy requirements of the Torbed reactors (1.25kg exposed for 30s).

Additional experiments were conducted to verify the proposed exfoliation reaction Eq 7.1. Known masses of vermiculite (large and medium grade for the purposes of calculations) were exfoliated until no more visible exfoliation were observed. It was then assumed that further exposure to microwave radiation will only result in dehydration and not any further exfoliation. The vermiculite sample was then weighed and the percentage "mass loss" calculated. The mass loss was between 3.5g and 5.0g (average 4.25g) per 100g of non-exfoliated vermiculite. 100g of vermiculite equates to 0.24 mol. According to Eq 7.1, for every 0.24 mol vermiculite exfoliated, the same number of moles of water should be released. If the "mass loss" is attributed solely to water being released from the structure, then 0.24 mol of water should be released. 4.25g water is equivalent to 0.236 mol. Therefore, experimental results confirms that reaction (Eq 7.1) holds.

7.8) Conclusion

The volume yield per tonne of vermiculite obtained in the laboratory scale experiments for the large grade vermiculite (on average 12.5 m^3/t) correlates well with the recorded values in literature (see Table 7.4). The medium grade vermiculite yielded approximately 7.2 m^3/t, which is less than the value quoted in literature (Table 7.4). For the three finer grades, experiments were conducted on samples of 10g. The volume yields for these small samples, does not correlate well with the values in literature.

Although poor correlation between literature and experimental results are observed, comparing similar samples treated on laboratory scale, shows a clear advantage of microwave radiation to be used as the exfoliation method, not only in terms of increased expansion ratio (ratio of expanded volume to unexpanded volume) but also in terms of treatment time and therefore in energy consumption. It has also been shown that the exfoliation of vermiculite by microwave radiation consist of three phases : activation, exfoliation and an exfoliation plateau. This may be used to calculate the optimum exfoliation time for vermiculite.

Structural differences between microwave exfoliated and thermally exfoliated vermiculite exist. Microwave exfoliation results in a highly irregular pattern compared to thermal exfoliation but it is not yet known to what extent (if any) this affects the physical properties of vermiculite.

The following paper has been accepted for publication in Materials Science and Engineering :

1) "An alternative method for the exfoliation of vermiculite" by Vorster, Marland, Kingman and Rowson.

The paper has been included in the "papers" section of this thesis. The following presentation has been delivered regarding this work (the presentation is included in the CD at the back of this thesis) :

1) "A novel technique for the exfoliation of vermiculite" at UMIST Research Day 2000 (umist00.exe)

CHAPTER 8

MICROWAVE TREATMENT OF PALABORA ORE

(Appendix D : Palabora Trials is on the CD at the back of this thesis)

8.1) Introduction

The Palabora Mining Company (PMC) was formed in 1956 to exploit the vast richness offered by the Palabora Igneous Complex. The Palabora igneous complex can be described by a figure 8 lay-out, approximately 8 km from north to south and 3.2 km from east to west, with 3 open cast mines within : the phosphate pit, operated by Foskor, in the north west, in the north the vermiculite workings of PMC and in the south the PMC copper workings. Loolekop (site of the original mine) has long since vanished - replaced by the second largest opencast mine in the world as shown in Figure 8.1 :

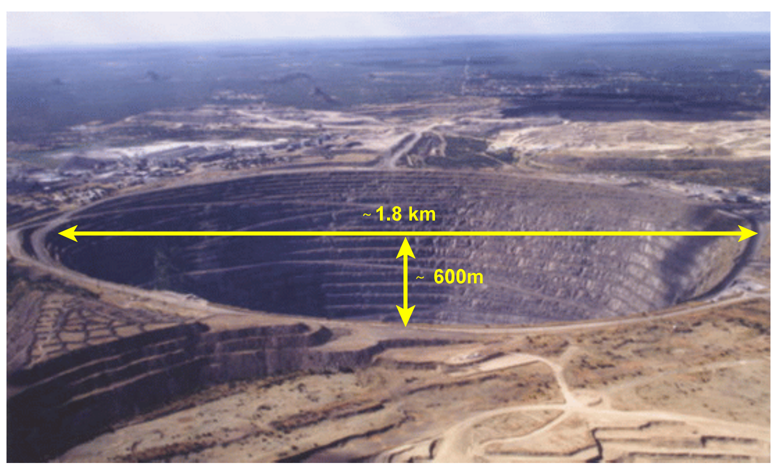


Figure 8.1 : The open cast mine viewed from space (left) and showing the dimensions

Only 2 of the 14 rock types are copper bearing. They are carbonatite and foskorite. An investigation into the geology of the 122 m level reveal the following distribution of mineral bearing rocks :

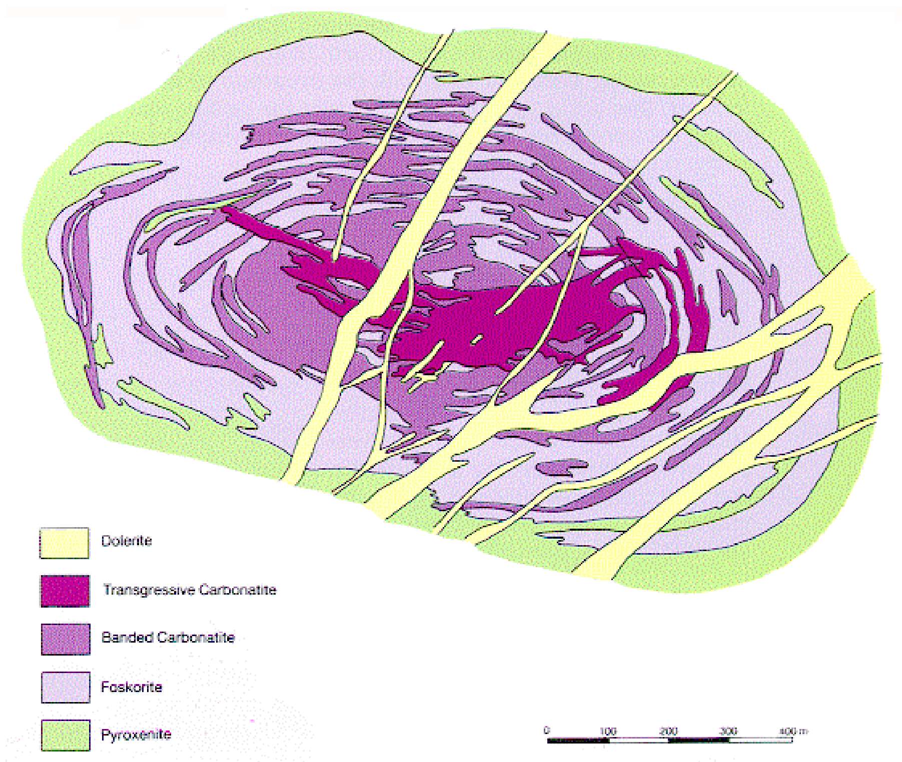


Figure 8.2 : Geology of the Open Pit at 122 m (PMC Ltd, 1998)

8.2) Processing of Palabora Copper Ore

Copper has been produced at the Loolekop site since at least 770 AD using primitive clay smelting ovens. These practices continued until late in the 19th century (PMC Ltd, 1998).

In 1912, Dr H Merensky carried out a geological survey of the area and started phosphate and vermiculite mining operations in 1930 and 1946 respectively. In the mid-1950's, it was proved that large quantities of low grade copper is present in the area. The Palabora Mining Company (PMC) was formed in 1956 as a joint venture between Rio Tinto and Newmont Mining Corporation to exploit the ore body. A schematic flow sheet of the ore processing is shown below :

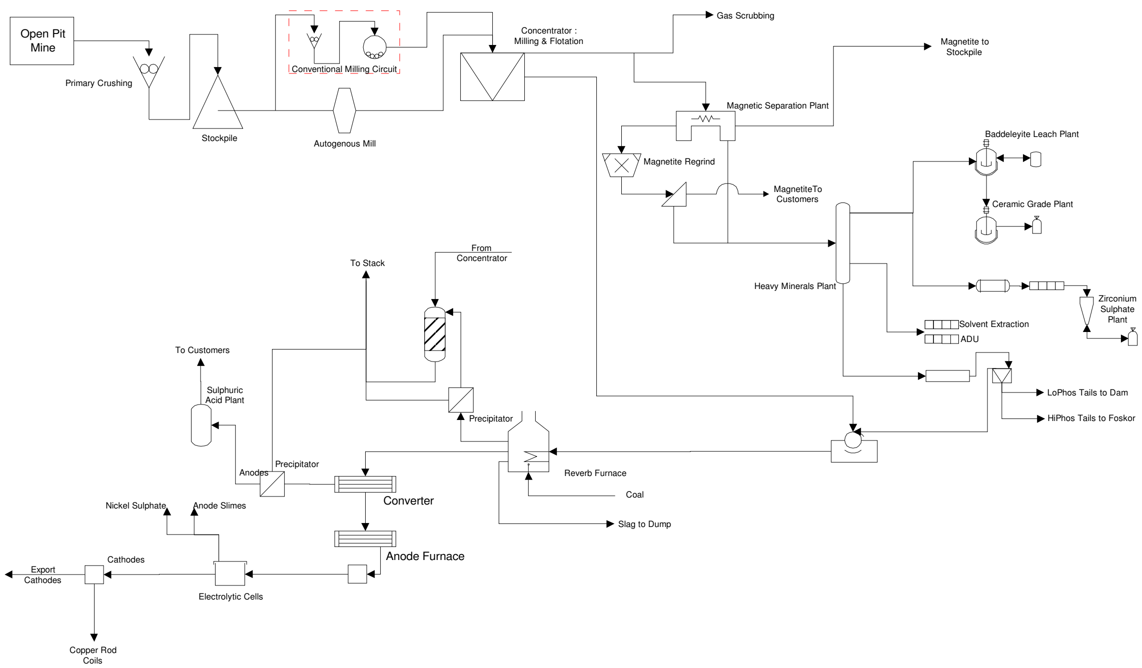


Figure 8.3 : Schematic process flowsheet of the Palabora Mining Company (PMC Ltd, 1998)

The point of entry of the crushed ore into the processing plant is at the concentrator. A schematic representation of the concentrator is shown below in Figure 8.4 :

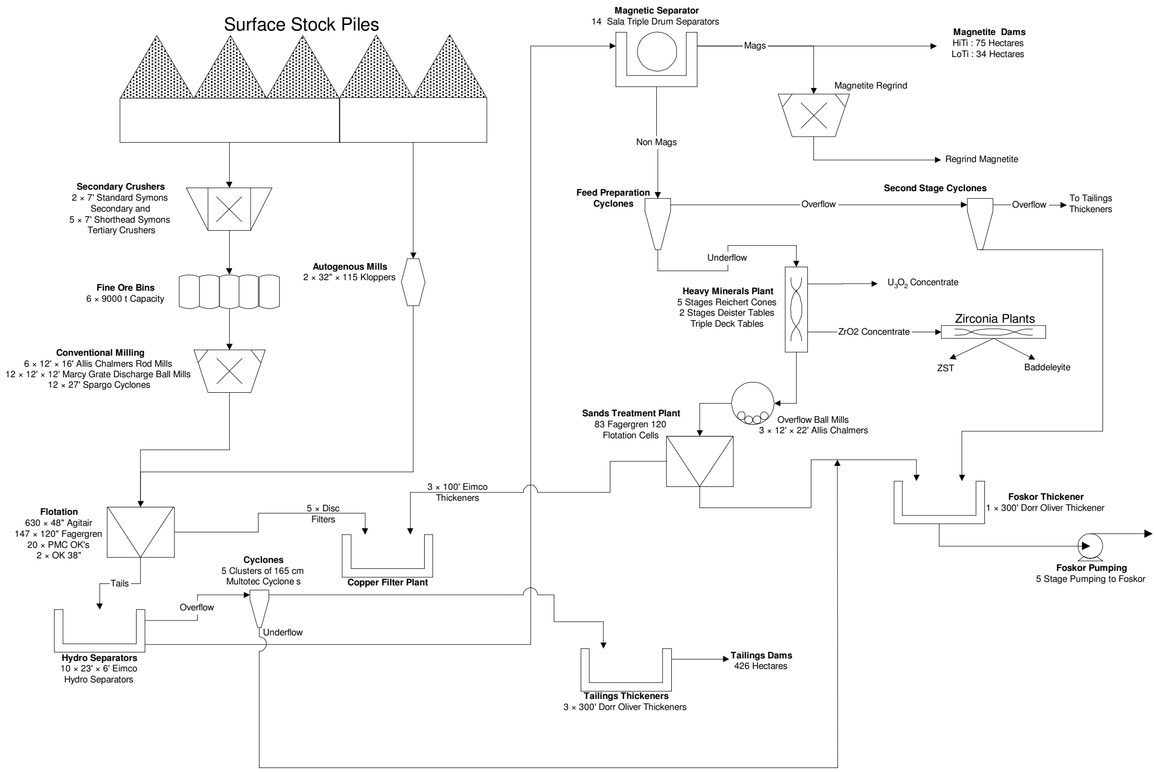


Figure 8.4 : Schematic representation of the processes in the concentrator at PMC (PMC Ltd, 1998)

There are two grinding circuits at Palabora, as can be seen in Figure 8.3 and 8.4. The first, the conventional milling circuit consist of 6 identical parallel circuits, each consisting of one rod mill, two ball mills and 2 cyclones. (Henderson, 1990).

Power is supplied by 900 kW motors, with the rod mill at 64% and ball mills at 72% of critical speed. Each rod mill grinds 380 t/h at 80% solids. The feed size are 19 mm and the discharge at 80% -1.2 mm. Cyclone underflow is 80% solids and the overflow 56% solids at 80% -300μm.

The second grinding circuit is the autogenous milling circuit, which consists of 2 autogenous mills of dimensions $9.76\text{m} \times 3.81\text{m}$ Koppers mills. Each mill is driven by 2 3.5 MW motors at ~10 revs/min at 70% solids.

Run-of-mine ore are fed at a rate of 600 t/h at 175 mm and discharged at 80% -300 μm. The mill product is screened at 2.5 mm, with the oversize recycled to the mill and the undersize cycloned. The cyclone overflow is pumped to the flotation section and the underflow is recycled to the mill (Henderson, 1990)

Two gangue minerals have a significant impact on the milling rate is dolorite and magnetite. Dolorite, with a work index of 30 $kWh.t^{-1}$ represents a high resistance to milling and has no economic importance. It causes specific problems in autogenous milling as grinds at a slower rate than the rest of the material, causing a build up of dolorite in the mill, which reduces the feed rate.

8.3) Mineralogy of the Palabora Copper Ore

A detailed mineralogical characterization of a head sample of Palabora copper ore was obtained from Rio Tinto Technology Development Ltd. The head sample contained approximately 25 % magnetite and 1 % copper sulphides such as chalcocite (Cu_2S), bornite (Cu_5FeS_4), chalcopyrite ($CuFeS_2$), cubanite ($CuFe_2S_3$), valleriite ($4(Fe,Cu)S{\cdot}3(Mg,Al)(OH)_2$) and pyrite (FeS) are also present.

However, transparent gangue minerals makes up the majority of the ore. Qualitative SEM analysis of the gangue minerals reveals that they consist of mainly 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 ($Mg_3Si_2O_5(OH)_4$), pyroxines and plagioclase feldspar.

Carbonate minerals, most frequently calcite and dolomite, usually occur as intergrowths with magnetite, sulphides or gangue - as is shown in Figure 8.5A. The main phosphate bearing mineral, apatite, occurs abundantly as rounded grains associated with magnetite, transparent gangue and sulphide minerals, as is shown in Figure 8.5B.

Olivine is also present in the sample, although partial replacement of olivine by serpentine has occurred. Much of the serpentine forms a fine grained aggregates which in some cases filles the fractures in magnetite and apatite.

Phlogopite, a Mg rich mica, occurs as flake-like aggregates associated with magnetite, sulphide minerals and transparent gangue. A small number of rock fragments consist mainly of plagioclase feldspar, pyroxene and skeletal magnetite - aggregates of which represents microgabbro - or dolorite - which is present as intrusions in the ore body (see Figure 8.2).

Other gangue minerals include ilmenite, which occurs along the grain boundaries of magnetite, which is abundant in this sample.

Apatite is the major phosphor bearing mineral in the ore and occurs mainly as rounded grains associated with magnetite, gangue and sulphide minerals. Many fractures are present within the apatite particles and these are commonly filled with magnetite and serpentine as illustrated in Fig 8.5D.

Magnetite occurs abundantly as discrete liberated grains and to a lesser extent as intergrowths with transparent gangue in this sample. A large number of magnetite grains also contain spinel inclusions. Micron sized inclusions of titanium rich bodies are spread throughout the magnetite grains. The nature of the magnetite and serpentine filling the fractures in the apatite is illustrated in Figure 8.5D.

The sulphide minerals, bornite and chalcopyrite, are usually intergrown and is present along the grain boundaries of magnetite as is shown in Figure 8.5C.

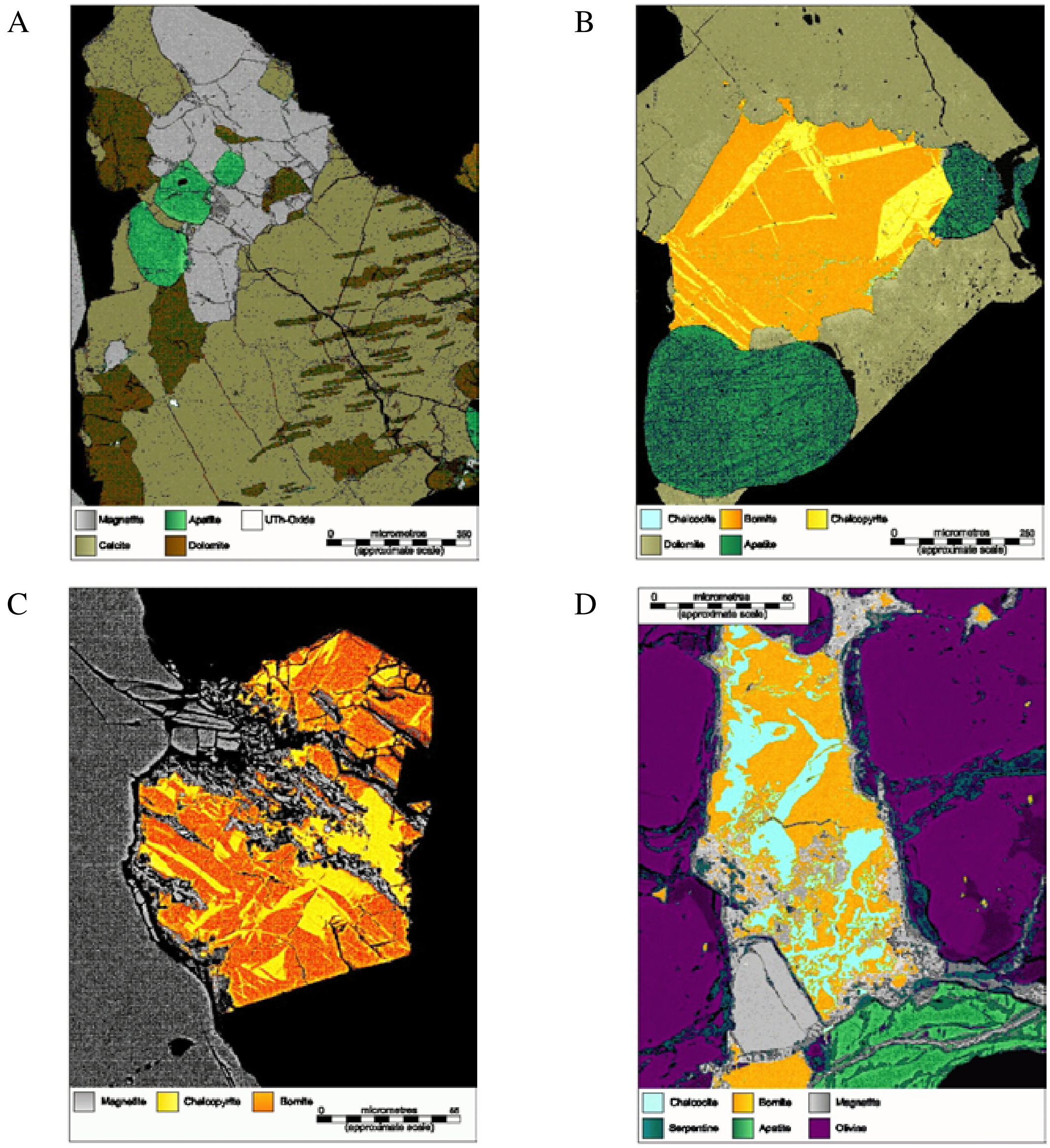


Figure 8.5 : False coloured backscattered electron images of untreated ore.

A) The textures of dolomite and calcite showing the intergrowths with magnetite and apatite.

B) Chalcopyrite lamellae within bornite as well as the rounded grains of apatite.

C) The nature of chalcopyrite and bornite along a magnetite grain.

D) The occurrence of a magnetite grain present along the margins and grain boundaries of a bornite and chalcocite aggregate. Magnetite is also present in the fractures in the apatite and olivine has been replaced by serpentine.

8.4) Experimental Procedure

8.4.1) Effect of Microwave Radiation on Mineralogy

As the mineralogy of an ore determines whether or not it can be successfully mined, it is important to know whether or not exposure to microwave irradiation affects the mineralogy. If microwave energy can be successfully utilized to reduce the cost of comminution, but at the same time change the mineralogy to such an extent that the valuables can no longer be extracted effectively, all the downstream processes will be adversely affected and processing of the ore might become uneconomical. Samples of Palabora copper ore were exposed to microwave radiation for different time periods in a 2.6 kW microwave unit, quenched and dried before being studied under a scanning electron microscope.

8.4.2) Effect of Microwave Radiation on Grindability

Previous work (Kingman et al, 2000), showed that Palabora ore responded well to microwave radiation with large decreases in work index possible. However, as all the studies were conducted on laboratory scale, it was decided to conduct a pilot scale study.

Initial trials were carried out on 5 kg batch samples in a 6.6 kW microwave unit at the Midlands Electricity Board (MEB) research facility in Halesowen, Birmingham. The microwave unit was fitted with a turntable as well as a Kevlar conveyer belt for continuous operation. The microwave unit is shown in Figure 8.6 :

Figure 8.6 : The pilot scale 6.6 kW microwave unit used for the pilot scale studies

Representative 5 kg samples have been made up of -19 600µm material. The different samples, have been treated for 60, 90, 120, 150, 180 and 300 s before being quenched in water. Initially samples were exposed to microwave radiation on the conveyer belt as well as on the turn table. Experiments were then conducted on 5 kg batches on the moving conveyer belt at different residence times, before moving onto “full scale”, pilot scale studies, exposing 165 kg/h of ore to microwave radiation before quenching the heated ore in water.

Figure 8.7 shows the dimensions of the continuous microwave unit as well as the position of the sample as it enters the cavity :

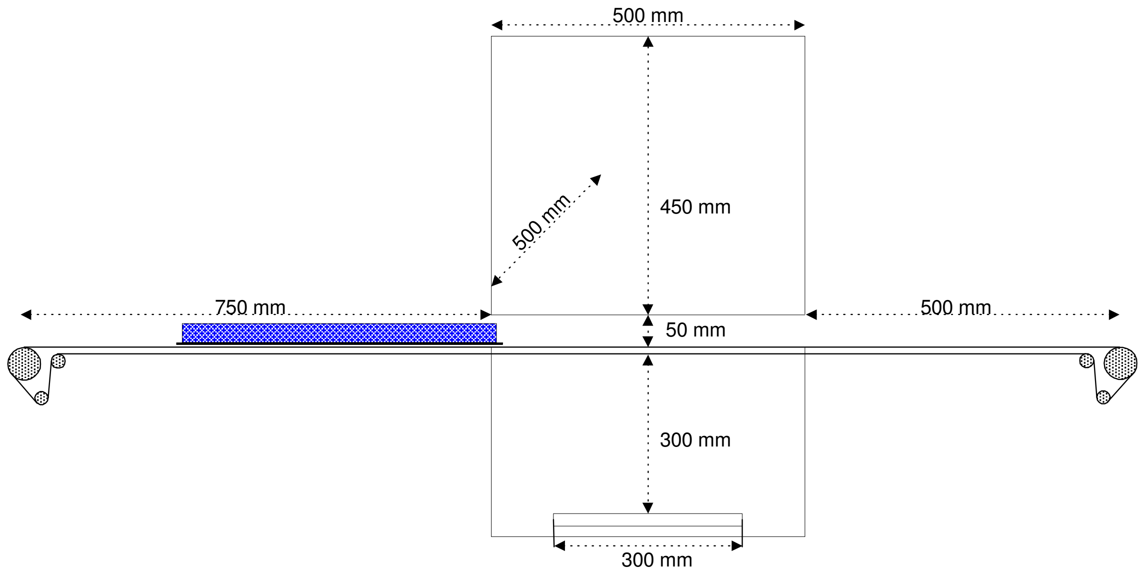


Figure 8.7 : Schematic of the microwave cavity, indicating dimensions

The relative grindability was determined using the Berry and Bruce method. Initially, a rod mill (diameter 155 mm, height 275 mm loaded with 5 rods; diameter 25 mm, height 265 mm) rotated at 90 rpm was used for determination of the relative grindability, using a 500g sub-sample. However, due to inaccuracies in obtaining the sub-samples, it was decided to use the whole 5 kg sample in a large rod mill (diameter 200 mm, height 310 loaded with 5 rods; diameter 32 mm, height 300 mm) rotated at 90 rpm for 5 min.

After continuously exposing 500 kg of ore to microwave radiation, pilot scale milling was carried out in a pilot scale rod mill :

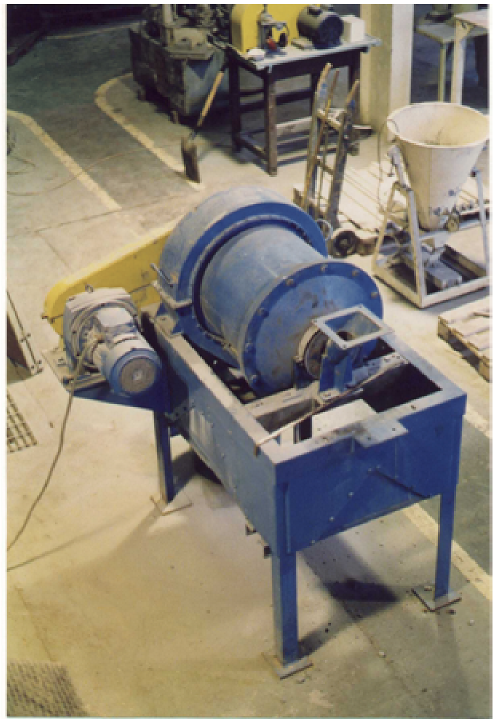


Figure 8.8 : Pilot scale rod mill used for pilot scale milling trials

Initially the rod mill was loaded with 83 rods and 30 kg of untreated ore. In order to mimic a continuous feed, batches of 2.1 kg each were fed to the mill over 5 min periods. This was continued for 65 min (25.2 kg/h), after which a sample was obtained from the output from the mill to determine the percentage passing 300μm. A separate sample was used to determine the full particle size distribution of the product. The experiments were repeated for mill feed rates of 50.4, 75.6, 100.8 and 126.2 kg/h.

The grinding trials were then repeated for ore exposed to microwave radiation (with mill fed rates of 126.2 and 100.8 kg/h). Representative 5 kg samples were made up from the treated and untreated material and grindability trials conducted using the large rod mill.

The effects of particle size on the reductions in work index due to microwave treatment was investigated. 500g of material consisting of a single size fraction were exposed to microwave radiation for 60s before being quenched and dried overnight. The relative grindability of each fraction (+1700-2360μm, +2360-3350μm, +3350-4750μm, +4750-6700μm, +6700-9500μm and +9500-13600μm) was then determined.

The power absorbed by the material in the microwave cavity is proportional to the square of the electric field strength according to Equation 2.8. The electric field strength of the 2.6 kW and 6.6 kW microwave cavities were then determined using the method explained in Chapter 2.

The effect of sample container were also investigated by exposing samples in different containers (pyrex, mortar, no container) and comparing the grindability. Different masses were also exposed to determine any mass loading effects. The effect of sample area on the effectiveness of microwave radiation have also been investigated by dividing the microwave unit into areas as shown in Figure 8.9 :

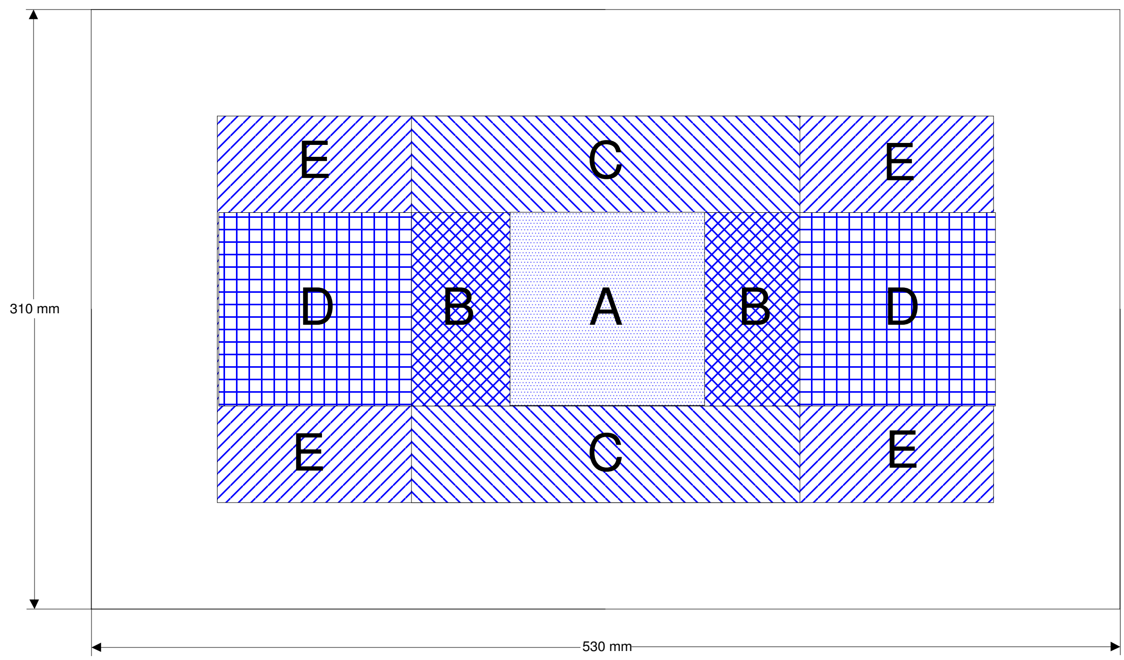


Figure 8.9 : The division of the 2.6 kW microwave into different areas

500g samples were placed on different areas (A, A+B, A+B+C, A+B+D and A+B+C+D+E) within the cavity, thus varying the thickness of the sample. The relative grindability was then determined to determine the optimum lay-out for sample treatment in the cavity.

The copper and iron content of different size fractions were also determined as this would affect the response of the material to microwave radiation. Additional experiments were conducted on the magnetic and non-magnetic sections of the material to quantify any possible effects on microwave response.

The dielectric constant and permittivity of the ore as well as the major individual components were determined at the Department of Electrical Engineering at the University of Nottingham (Chapter 2). Mixture equations were used to estimate the dielectric constants of the ore from its components and compare direct measurements.

In order to investigate alternative methods for the quantification of the effects of microwave radiation on Palabora ore, two types single particle breakage tests were conducted. Firstly, batches of 50 particles (-13600+9500μm) were exposed to microwave radiation (60s, 2.6kW) before being quenched. These particles were then individually broken on a load frame and the energy and work done for breakage recorded. Secondly the three point bend test (as described in Chapter 3) was used to determine particle strength. Core samples, 60mm in length of different diameters (19, 27 and 35mm) were obtained from R.O.M. ore. The cores were heated in a 1.3 kW microwave heater different exposure intervals before being quenched. The core samples were supported at their end-points on two mild steel plates. A third plate was used to apply a load diametrically. A 'load versus displacement' curve is continually recorded by a computer during the operation and results compared to the Berry and Bruce method for relative grindability.

8.4.3) Effect of microwave radiation on flotation and magnetic separation

Kingman et al, 2000, investigated the effects of microwave radiation on the flotability and magnetic separation of Palabora copper ore. Ore was exposed to microwave radiation at 2.6 kW before being ground and batch flotation trials conducted.

8.4.4) Alternative methods for the determination of rock strength

Three Point Bend Test

The three point bend test has been suggested as a possible method to overcome the problems associated with the Bond test and conventional single particle tests. The three point bend test is a method whereby a cylindrical specimen is subjected to loading by tensile, compressive and shear stress as illustrated in Figure 8.10. Tensile stress develops on the convex side of the beam and compressive stress on the concave side.

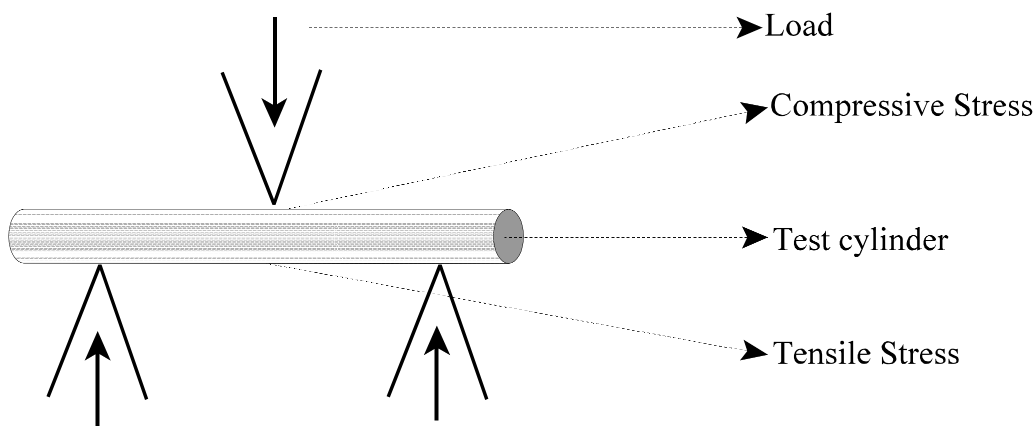


Figure 8.10 : Schematic description of the three point bend test

The test in this study is easily reproducible and due to it small scale, enables a large number of samples to be obtained per volume of material.

Palabora core samples, 60mm in length and 19, 27 and 35mm in diameter were also tested. Batches of 15 such cores were heated in a 1.3 kW microwave heater at 2.45 GHz for different exposure intervals. Upon removal from the microwave the specimens were quenched. The strength values (work required to induce catastrophic fracture) of the sample cores, were measured using a three-point bend test, as shown in Figure 8.10. A 'load versus displacement' curve is continually recorded by a computer during operation.

Drop Weight Tests

A drop weight test rig has been constructed and is shown schematically in Figure 8.11 :

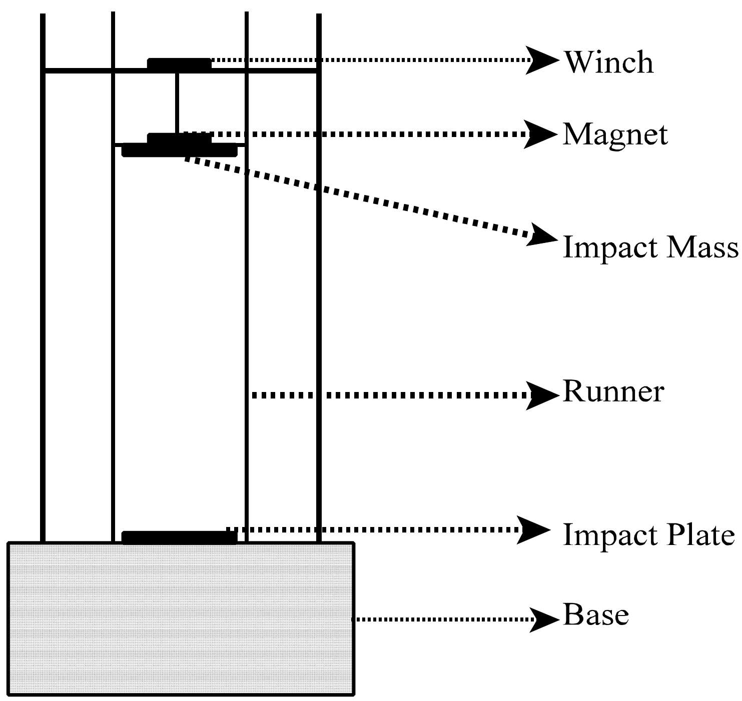


Figure 8.11 : Diagram of the drop weight tester

Fifty particles of similar size (in the same size class in a standard sieve series) comprised one sample. Each sample of 50 particles were weighed. A single particle was placed on the impact plate, the impact mass raised or lowered to the specified height and the impact mass released. After the drop, the impact plate was cleared of debris and the process repeated from the same height for all 50 particles. Five samples were used for each size class, the material in each sample was crushed at different energy levels (see Eq 3.14).

After breakage the debris was sized, the particle size distribution plotted and the value of t10 (the fraction passing a tenth of the input size) calculated for each energy input level (Eq 3.15 defines t10 in terms of ore parameters A and b). Using the graph of t10 versus specific energy input the values of the ore parameters may be calculated and the mode one fracture toughness (Kic) may be calculated from the parameters (using Eq 3.16).

Tests were conducted on three different particle sizes to determine the effect of particle size on results. In addition green granite and Yorkstone was used to determine the accuracy of the results. One size fraction of Palabora ore was exposed to microwave radiation for 60s at 2.6 kW to determine the whether or not the drop weight test is sensitive enough to detect changes in strength due to microwave pre-treatment.

The t10 values were calculated automatically in Mathcad by use of the cubic spline curve fitting function and A and b were calculated from the t10 and Ecs values by the least squares method.

8.5) Results and Discussion

8.5.1) Effect of Microwave Radiation on Mineralogy

Seven samples, subjected to microwave treatment for various time periods have been investigated for mineralogical changes. The samples of material treated for 10 and 30 seconds exhibit no evidence of changes due to microwave treatment. The majority of grains in the sample treated for 60 seconds are also unaffected, although a minute quantity of magnetite have been oxidised to hematite (according to Reaction 8.1) along the grain boundaries (Figure 8.12A) :

$$2\ Fe_3O_4 + ½\ O_2 \rightarrow 3\ Fe_2O_3 \qquad \text{Reaction 8.1}$$

Some, of the sulphide grains also show a small degree of oxidation on the grain boundaries. Metallic copper has formed on the grains boundaries of a few of the copper containing grains. Microwave treatment for 90 seconds did not affect the mineralogy of the majority of the grains. Oxidation did occur along a few grain boundaries - notably that of magnetite to hematite, although the number of grains affected are still very small. Some of the copper bearing particles have been oxidized along the grain boundaries (Figure 8.12B and D). A small number of glass rich particles have also formed (Figure 8.12B). After 120 seconds of microwave heating, the number of grains affected by microwave heating have increased. The mineral grains affected most obviously are the bornite and chalcocite particles, which have metallic copper and hematite on the boundaries. Some of the pyrite grains have also been oxidized according to Reaction 8.2 :

$$2\ FeS_2 + 2\ O_2 \rightarrow 2\ Fe_S + SO_2 \qquad \text{Reaction 8.2}$$

Partial melting along the grain boundaries is proof of the high temperatures reached. The partial melting of the magnetite typically results in the formation of bleb like and dendritic (skeletal) magnetite. The fine grained melted magnetite exhibits a difference in chemical composition compared to the original magnetite, in that the reflectivity is less - implying the presence of magnesium enrichment from the magnesium spinels. Glass rich fragments are abundant in this product, shown in Figure 8.12B.

After 180 and 240 seconds, the number of particles affected by microwave radiation has increased significantly. Partial melting along the grain boundaries of magnetite grains can be observed and oxidation to hematite can be more readily observed. Glass rich fragments have also increased in these samples. It was also noted that fractures are abundant in the non-microwaved samples. This made the recognition of extra fractures in the microwaved samples very difficult.

Figure 8.12A shows the partial oxidation and replacement of a magnetite grain (medium grey) by hematite (light grey/white). Oxidation is most prominent along the margins but also toward the core regions of this grain. The partial melting of magnetite results in the development of bleb-like dendritic magnetite that occur within the Ca-Si-rich glass (Fig 8.12B).

A large amount of carbonate in this sample have been altered to CaOH. These porous aggregates occur as liberated grains, intergrowths with the ore or as porous spheroids associated with intergranular calcium ferrite, which may form as a result of the reaction between carbonates and magnetite during microwave treatment.

Some copper bearing sulphide minerals have been altered as a result of microwave treatment. For instance, pyrite may be decomposed to a porous iron-sulphur phase as illustrated in Figure 8.12C. Certain sulphide minerals, in particular bornite, chalcopyrite and chalcocite, exhibit corroded grain boundaries with hematite and copper formation along these boundaries (Fig 8.12D).Valleriite, a copper bearing mineral with low flotability, occurs along the margins and as intergrowths with the more abundant ore and gangue and is largely unaffected by microwave radiation.. However, a small proportion shows an increase in porosity. Sulphates are fairly common in this sample and may represent the alteration products of former sulphide and carbonate aggregates.

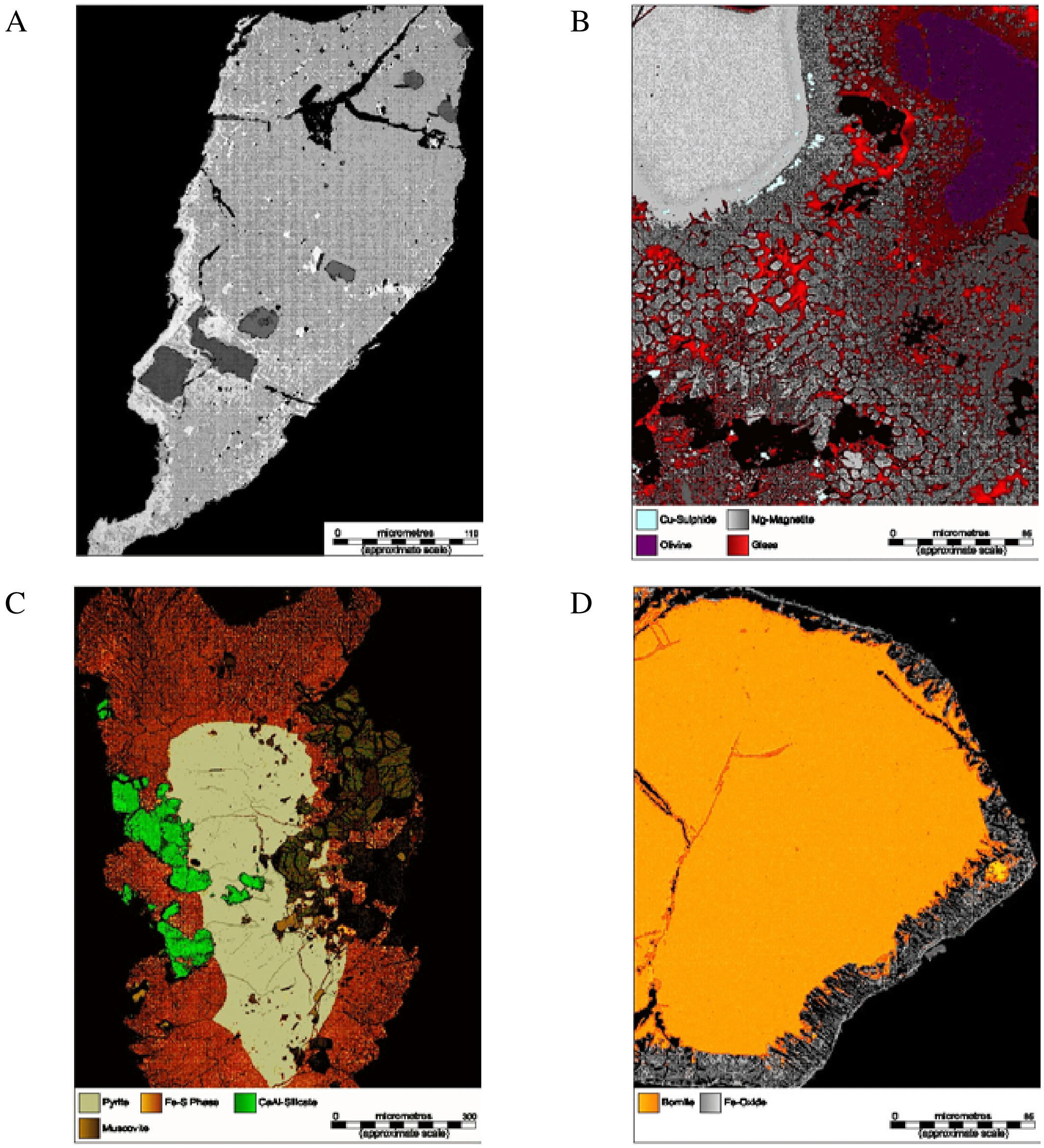


Figure 8.12 : SEM images of ore exposed to microwave radiation

A) Monochrome reflected light photomicrograph showing the partial oxidation of magnetite to hematite after 240 s @.2.6 kW.

B) Glass fragments and the skeletal magnetite formed (240s @ 2.6 kW).

C) Partial decomposition of a pyrite grain to a highly porous Fe-S product (240 s @.2.6 kW).

D) Nature of a bornite grain that has been partially oxidised to hematite.

8.5.2) Effect of Microwave Radiation on Grindability

It has been shown (Kingman et al, 2000) that microwave radiation effectively reduces the work index of Palabora ore, as is shown in Figure 8.13 :

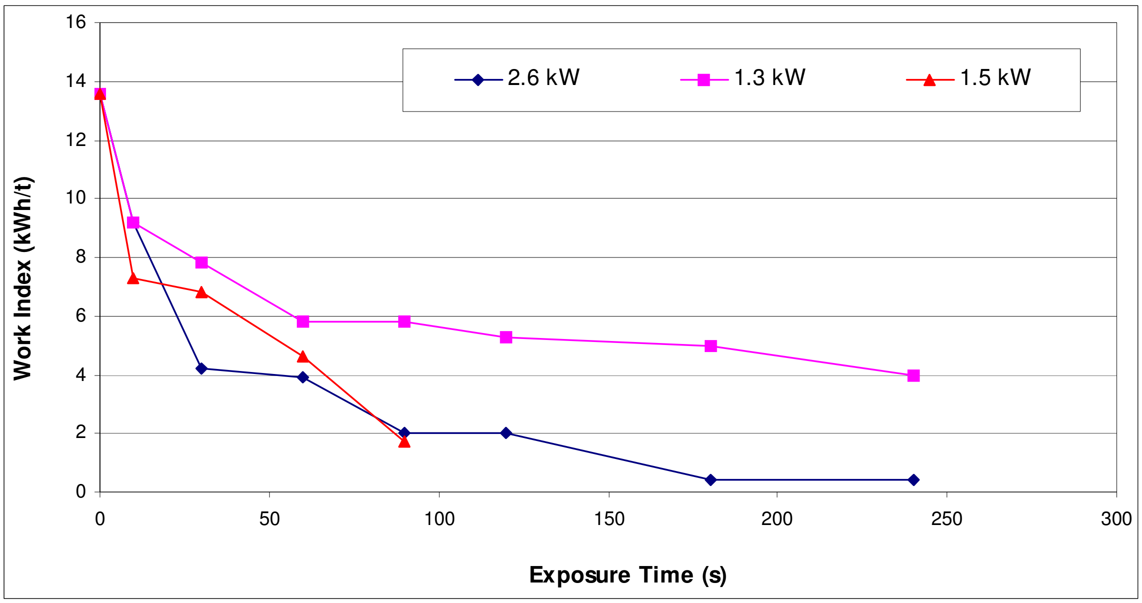


Figure 8.13 : The reduction in work index achieved in different microwave units as a function of time (after Kingman et al, 2000), using 1 kg batches

It should be noted from Figure 8.13, that long microwave exposure times are not necessarily equivalent to larger reductions in work index. This is because the rapid differential heating during the first few seconds of radiation is sufficient to cause intergranular fracture. Longer exposure times only serves to melt the ore, as was shown from the mineralogy report, which has detrimental effects on down stream processing.

All of the above trials were conducted in batch microwave units. This would be impractical in industry, where large tonnages need to be treated quickly and efficiently. Trials were therefore conducted in a 6.6 kW microwave unit, fitted with a conveyer belt, at the MEB research facility at Halesowen, Birmingham. The particle size distribution of the material used for the initial series of trials are shown in Appendix D1. Initial results showed only very small reductions in work index - a maximum of 40% reduction in work index opposed to in excess of 90% from previous work (shown in Appendix D2). The -1.7mm material was removed from the sample and microwave samples irradiated on thermal insulating sheets instead of the pyrex contained. The reduction in work index achieved for different residence times is shown in Figure 8.14. The results in the 6.6 kW microwave unit are also compared to the results by Kingman et al (2000) :

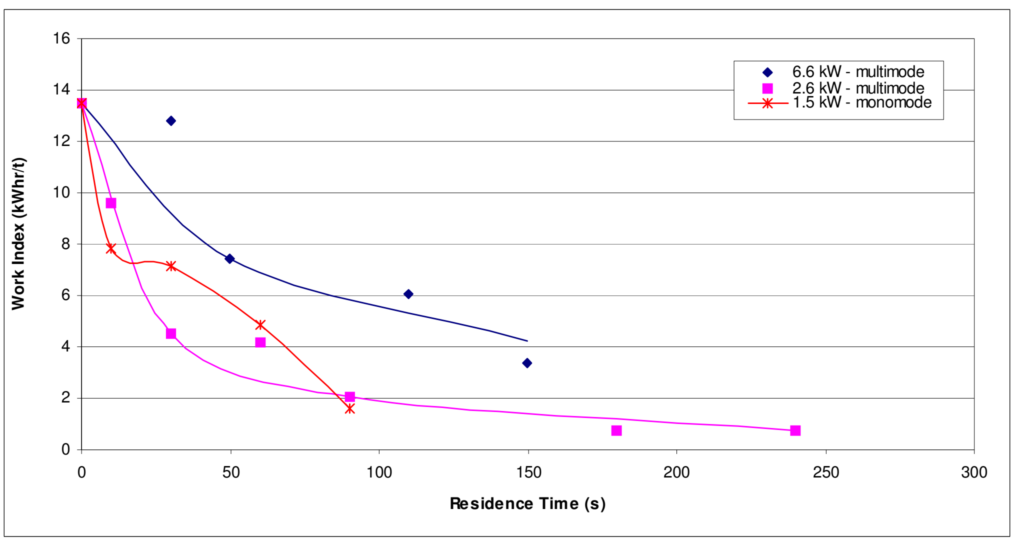


Figure 8.14 : Diagram comparing the reduction in work index in the 6.6 kW unit with previous results (after Kingman et al, 2000). The full set of results may be viewed in Appendix D3.

As can be seen from Figure 8.14, although the reduction in work index is greater than indicated in the initial trials, it is still less than the reductions achieved in the 1.5 kW and 2.6 kW units. However, the curve of work index versus time follows the same trend. Palabora copper ore were then treated continuously in the 6.6 kW microwave unit before being milled in a continuous pilot scale rod mill at Svedala Ltd in Derby.

A plot of %passing 300µm versus the feed rate (kg/h) was then plotted for untreated material as well as for material exposed to microwave radiation :

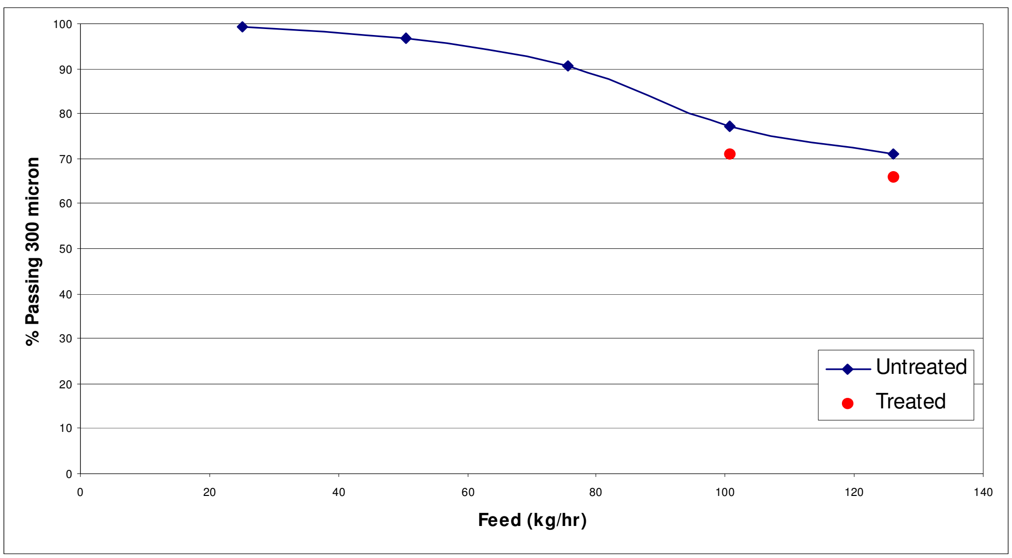


Figure 8.15 : Plot of %passing 300µm for treated and untreated Palabora ore

The blue curve in Figure 8.15 indicates the percentage mill product passing 300 micron for untreated ore. The two red points at 100.8 and 126 kg/h indicate the percentage mill product passing 300 micron for the treated ore.

It was expected that the percentage passing 300 micron for the treated ore (i.e. the red points) would be much less than for the untreated ore. Although there is a difference between the points, it could be due to experimental inaccuracies rather than any effect of the microwave pre-treatment (Personal Communication : G Brown, 1999). No further pilot scale grinding studies were conducted, as it was considered that the microwave pre-treatment was not effective.

To confirm the results in Figure 8.15, the particle size analysis of all the products were (Appendix D4) determined :

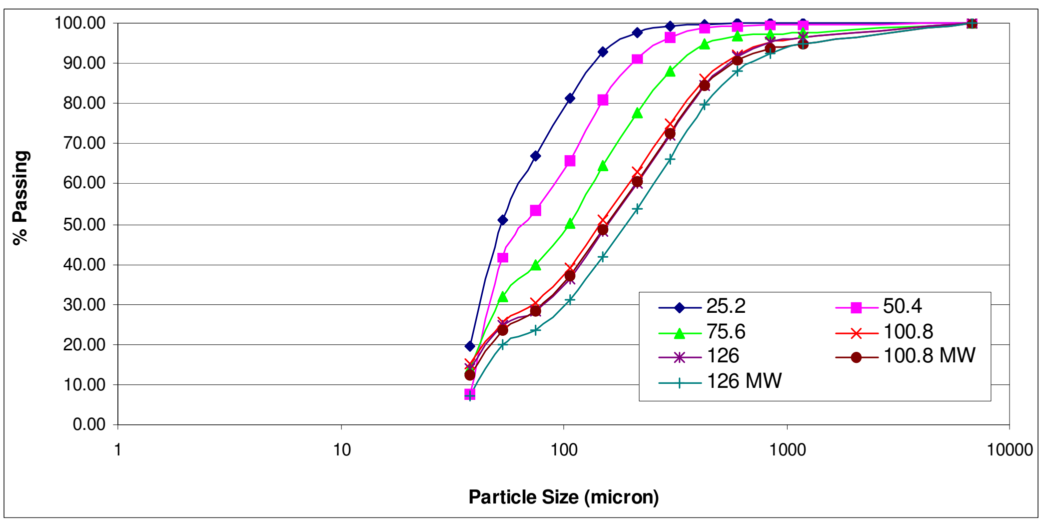


Figure 8.16 : Graph showing the size distribution for various mill throughputs (in kg/h). The results for 100.8 kg/h and 126 kg/h were duplicated using material that have been treated in the 6.6 kW microwave unit.

In Fig 8.16 the particle size distributions of the pilot scale mill products (different throughputs in kg/h) are shown. For the 100.8 and 126 kg/h runs, no significant difference between the microwaved ore (100.8 MW and 126 MW) and untreated ore is observed.

In order to confirm these results, laboratory scale grindability trials were also conducted on 5 kg samples of treated (in the 6.6 kW microwave unit) and untreated material. No significant effect of microwave radiation could be detected on the grindability of the ore (Appendix D5) as is illustrated in Figure 8.17 :

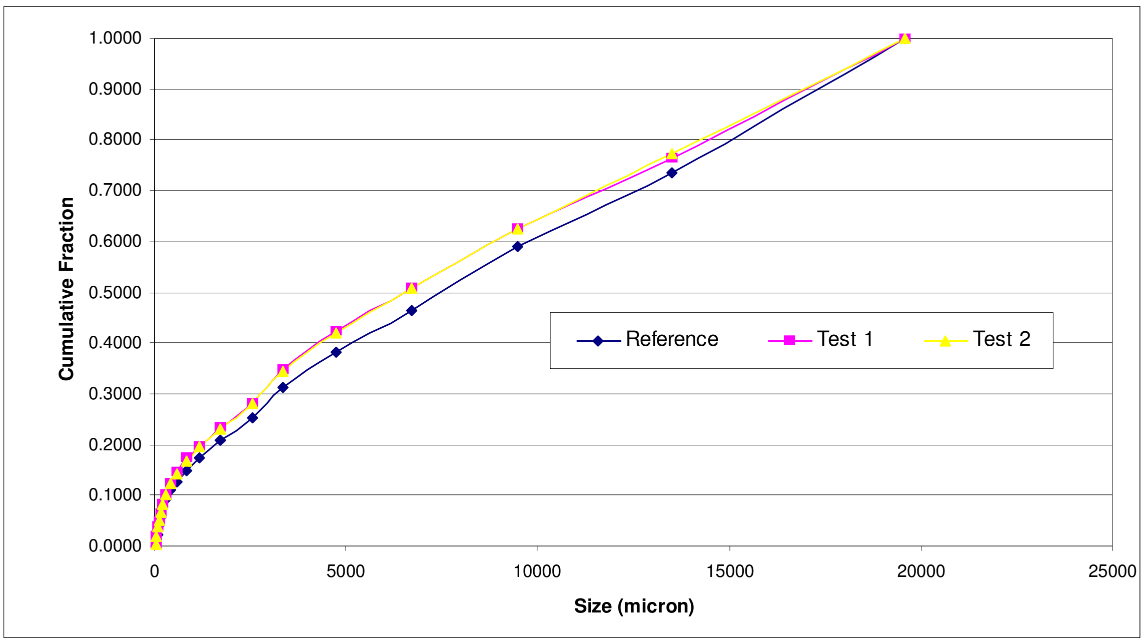


Figure 8.17 : No significant change in 80% passing size of the treated ore

The electric field strength of both microwave units were calculated (full results in Appendix D6).

The root mean square electric field strength of the 2.6 kW and 6.6 kW units have then been calculated. The field strength in the 2.6 kW and 6.6 kW units have been found to be approximately the same and equal to about 10 000 $V.m^{-1}$. As the field strength of the two units are approximately the same, one would expect the reduction in work index in the two units to be similar.

The difference between the continuous trials and semi-continuous initial trials were the mode of energy application. During the initial trials, 5 kg batches were moved through the cavity as shown in Figure 8.7 for a residence time of 110s (the maximum loading being 5 kg). Assuming that 6.6 kW energy is available to achieve the reduction in work index, the power to mass loading ratio change as shown in Fig 8.18 :

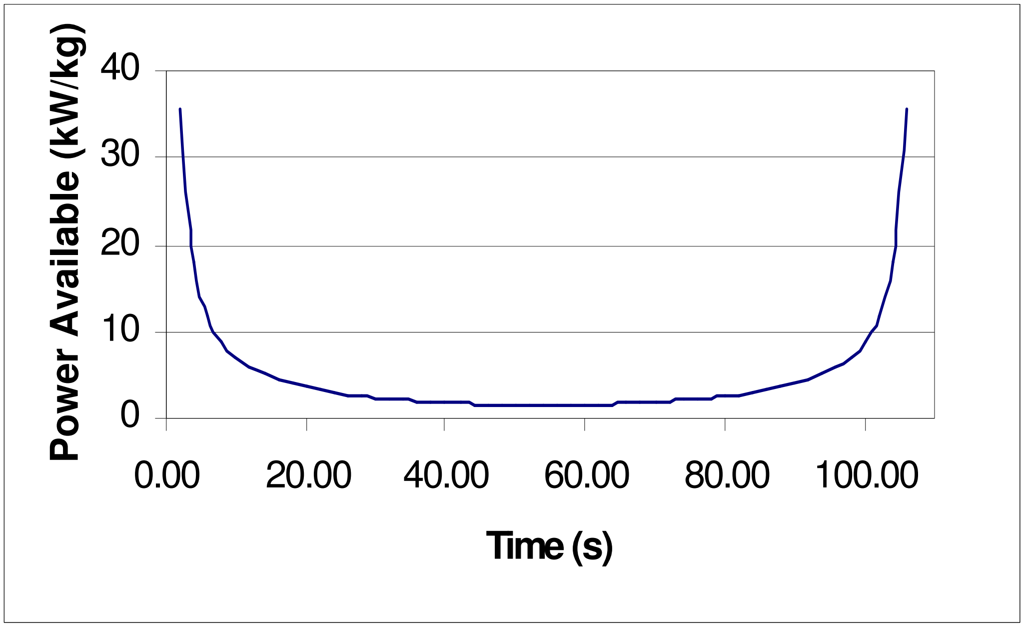


Figure 8.18 : The change in mass loading has on the available power for work index reduction for the initial semi continuous trials

For the continuous trials, the mass of material exposed to microwave radiation at any one time was 5 kg. The weighted average of available power for the semi-continuous trials is 5.2 kW/kg as compared to the minimum value (the value for the continuous trials) of 1.32 kW/kg (calculations in Appendix D7).

Particle size effect of microwave treatment

Shown below is a plot of reduction in work index for a variety of initial particle sizes (particle size distributions and calculations in Appendix D8) :

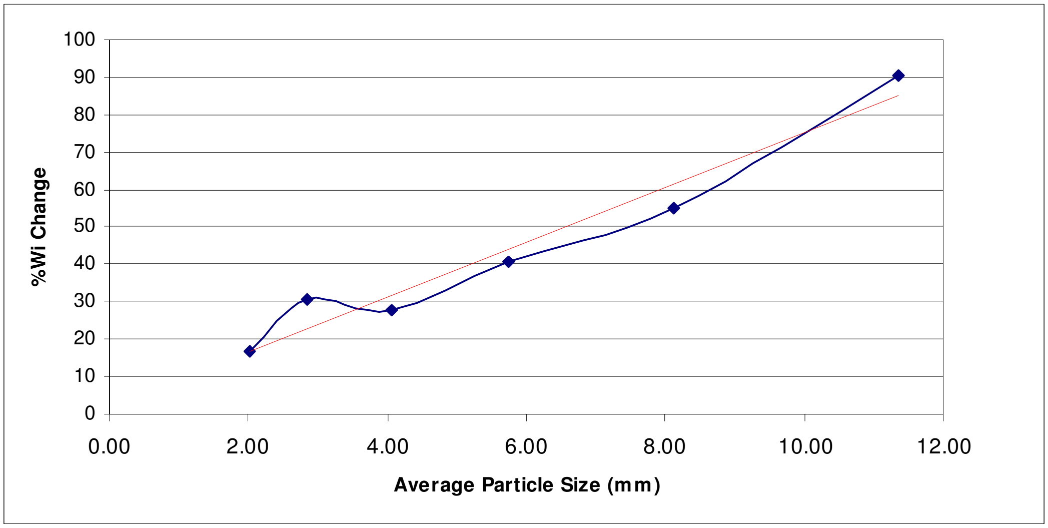


Figure 8.19 : Graph showing less reduction in work index for progressively smaller particles.

This graph shows that the smaller the particle size of the material the less effective microwave pre-treatment of the ore becomes. This may be due to the large grain size of the material. As particles decrease in size, the number of individual grains in a particle decrease. The major mechanism causing inter- and intra-granular fracture is differential expansion. Neighbouring grains (different minerals) heat (due to microwave radiation) and expand (due to heating) at different rates. As the fewer grains there are in a particle, the smaller the probability that they will be different minerals. Therefore the effect of radiation on grindability will be reduced.

The first property to be studied was the effect of sample area exposed to microwave radiation. The microwave cavity (2.6 kW) was divided into different areas as shown in Figure 8.9. Samples of 500g material were placed on the different areas and exposed to microwave radiation for 60s before being quenched and dried overnight. The relative grindability was then determined (results and calculations are shown in Appendix D13) :

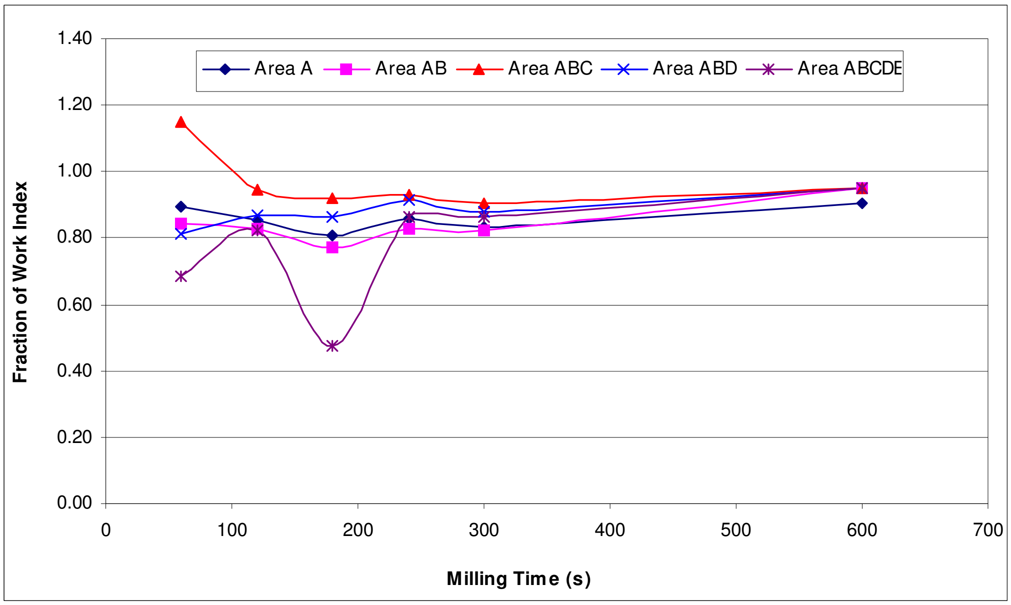


Figure 8.20 : Fraction of work index versus milling time for samples exposed to microwave radiation on different areas in the cavity

Figure 8.20 illustrates a few important characteristics. It confirms that the reduction in work index is independent on the milling time (within reason). Generally it shows that the larger reduction in work index occur on area AB followed by area A with the least effect on area ABC.

It will appear that the optimum area for microwave treatment is on Area ABCDE. However, the points at 60s and 120s are suspect. It was discovered after the trials were conducted that one of the sieves used for particle size analysis, was torn. This explains the large reduction in work index at 120s (more material at smaller size).

A small sample area seem to be more advantageous to microwave radiation than a spread out sample. This makes sense intuitively - a small sample area results in heat being kept in the sample whereas a large area facilitates heat transfer away from the sample.

In order to determine the effect of sample mass on work index reduction, different mass samples (250g, 500g, 750g, 1000g and 1250g) were then exposed to microwave radiation for 30s, 60s and 90s before being quenched, dried and milled for different times (results in Appendix D14). All samples occupied the same area in the microwave cavity (Area AB in Figure 8.9). It was found that 750g samples gives the largest reduction in work index - 28% - and seemed to be the "optimum" mass for treatment (in this case). The 250g sample gives larger reductions (up to 50%) but due to the small sample mass, large parts of the sample melts, making grindability trials non-repeatable. In addition, as experiments were conducted on +13.6mm-19mm material, 250g corresponds to approximately 15 particles - 1 particle of dolorite will affect the grindability trials - which is the reason for the apparent increase in particle strength for Area ABC at 60s in Figure 8.20.

This batch of material gives much lower reductions in work index than reported by Kingman et al (2000) and earlier work on the 6.6kW microwave unit. This is ascribed to a new batch of material from Palabora which was used for the test work. The new batch of material consisted in part of ore dug from underground during the construction of the mine shaft. It also contains a higher and more variable dolorite content.

The effect of Cu and Fe content in different size fractions was studied as well. It was postulated that higher Cu and Fe content in some size fractions may affect the response of different size fractions (Appendix D15). To investigate, 50g samples of +1.7mm-19.6mm were pulverised and analysed for iron and copper (by SGS Laboratories) :

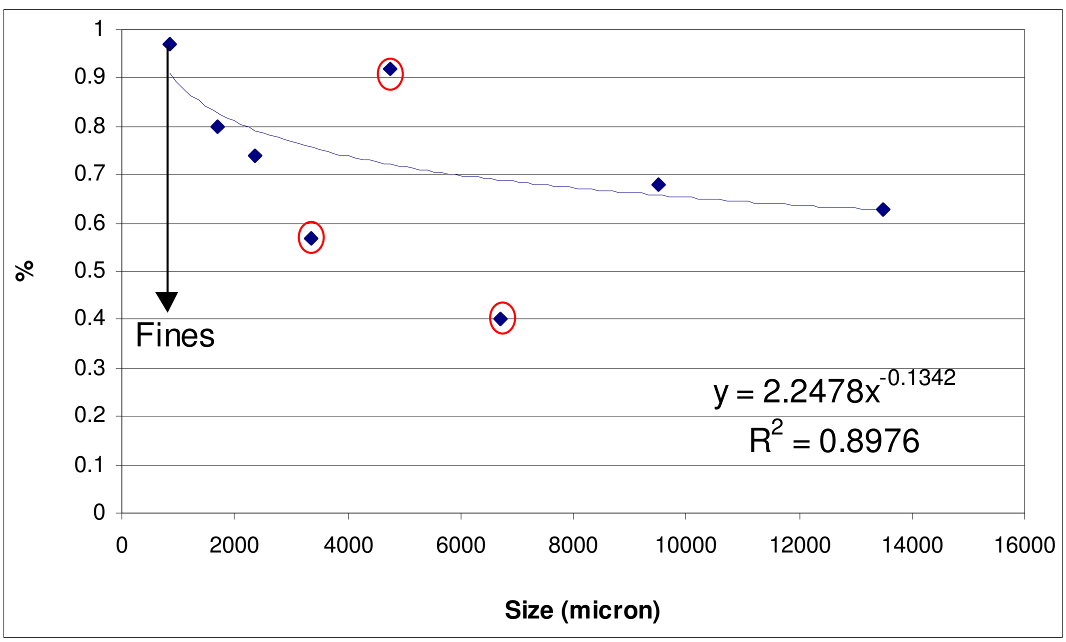


Figure 8.21 : The copper content of the Palabora ore

The average iron content is 19% and is not indicated on this graph (see Appendix D15). Assuming that the three points circled in red are due to inaccurate sampling (50g from 500kg), it can be seen that there is a decrease in copper content with increasing particle size. This makes intuitively sense as the milling size on site is 300µm (liberation size for the copper for flotation). The fines (-1.7mm) would therefore register more copper.

The effect of magnetic and non-magnetic fractions in different size fractions was also studied. It was postulated that the magnetic fractions would respond far better to microwave radiation as the bulk of magnetite (an extremely good microwave receptor) should be in the magnetic fraction. The magnetic and non magnetic fractions were separated in different particle sizes, using a rare-earth magnet.

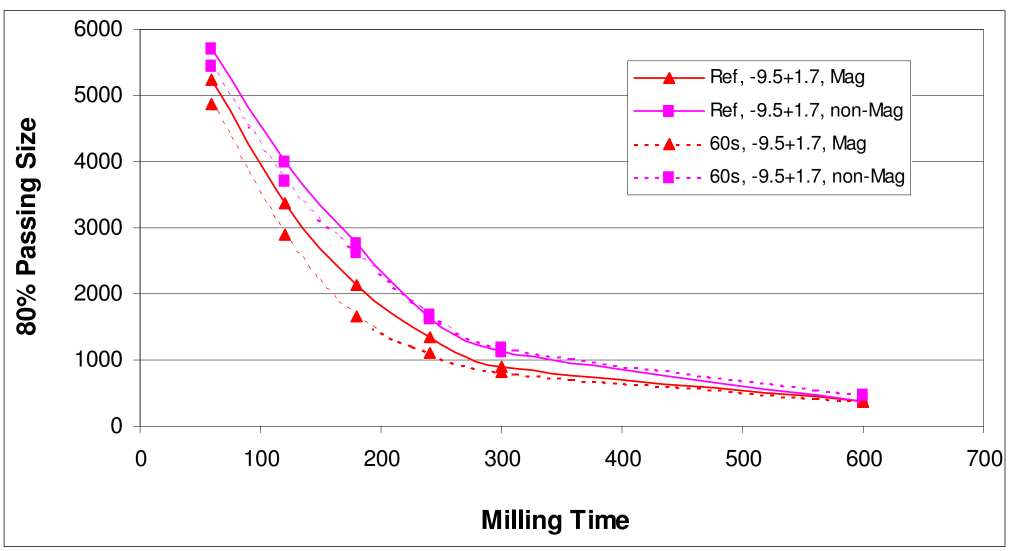


Figure 8.22 : 80% passing size versus milling time for references (unexposed) and microwave irradiated ore for magnetic and non magnetic fractions of the ore

From Figure 8.22 it can be seen that there is no great difference in microwave response for the magnetic and non-magnetic fractions. The non-magnetic sample has a lower grindability (possibly due to dolorite) than the magnetic sample. A new sample was received (which was used for this test work). As this sample comes from a different location (far away from the original sample locations in the open pit), it is thought that this material contains material susceptible to microwave radiation - explaining the small reductions in work index.

The dielectric properties of Palabora ore, as well as that of magnetite and calcite, were estimated using a TM_{010} cavity at the University of Nottingham (as described in Chapter 2). The values are tabulated in Table 8.1 :

Table 8.1 : Dielectric data of two samples of Palabora ore, quartz and magnetite

Mineral	**Dielectric Constant**	**Mineral**	**Dielectric Constant**
Palabora 1	3.100 - 0.0379·i	Quartz	2.862 - 0.216·i
Palabora 2	2.76 - 0.0424·i	Magnetite	6.418 - 0.150·i

Arai et al, 1995, proposed that the dielectric properties of a binary mineral mixture may be estimated from the dielectric permittivities of the individual components and the following equations :

$$\epsilon * = \left\{ \frac{\rho}{\rho_A} m_A \left(\epsilon_A * \right)^{\frac{1}{2}} + \frac{\rho}{\rho_B} m_B \left(\epsilon_B * \right)^{\frac{1}{2}} \right\}^2 \quad or$$

$$\epsilon * = \left\{ \frac{\rho}{\rho_A} m_A \left(\epsilon_A * \right)^{\frac{1}{3}} + \frac{\rho}{\rho_B} m_B \left(\epsilon_B * \right)^{\frac{1}{3}} \right\}^3$$

Using these equations, the permittivity of the Palabora ore may be estimated assuming that the ore consists of 75% quartz and 25% magnetite. The two estimates are 3.157 + 0.031·i and 3.139 + 0.03·i, which correspond well with the measured values in Table 8.3 (full calculations and results in Appendix D17).

The heterogenous nature of the Palabora ore made single particle breakage tests on the load frame nearly impossible. Results are shown in Appendix D18. The average value of the force required for breakage was 1.625 kN with a standard deviation of 1112. No further tests were done on single particles using the load frame.

Figure 8.23 shows the percentage decrease in work index to induce catastrophic fracture of different diameter cores versus microwave exposure time. This graph indicates that the strength of Palabora ore is significantly affected by exposure to microwave radiation and that the three point bend test is able to register this change (results and calculations are shown in Appendix D19).

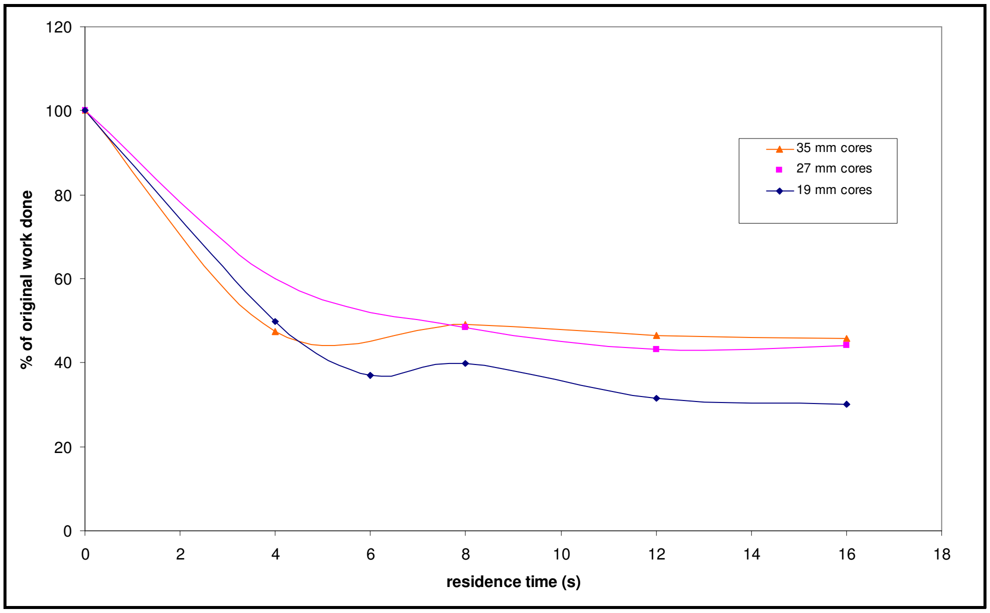


Figure 8.23 : Plot of relative work done versus microwave exposure time

The major strength reduction occurs after 8s microwave radiation. Prolonged microwave exposure does not result in significant further reductions the strength of the cores. Exposure times in excess of 16s resulted in the destruction of the cores upon quenching.

Comparing Figure 8.23 with the reductions achieved during initial grinding trials (Figure 8.14), it can be seen that the shape of the reduction versus exposure curves are similar. The main difference being that the exposure times are longer in the case of the grindability trials (Figure 8.14 and 8.23) - mainly because of the mass loading (1 core versus 500g samples).

However, it should be noted that trend in both graphs are similar - the largest reductions occurs after short exposure times while longer exposure times does not result in significantly higher reductions. The main mechanism responsible for the reduction in work index is differential heating and differential stresses at the grain boundary. To generate temperature differentials, long exposure times are unnecessary - short exposure times are sufficient - as long as high temperatures within some species are generated in the time.

8.5.3) Effect of microwave radiation on flotation and magnetic separation

The results of the investigation into the effect of microwave radiation on the flotability and magnetic separation of Palabora copper ore by Kingman et al, 2000, is shown below. For the ore exposed to microwave radiation in an oxygen rich environment (i.e. air) the percentage recovery of copper versus flotation time is shown in Figure 8.24 :

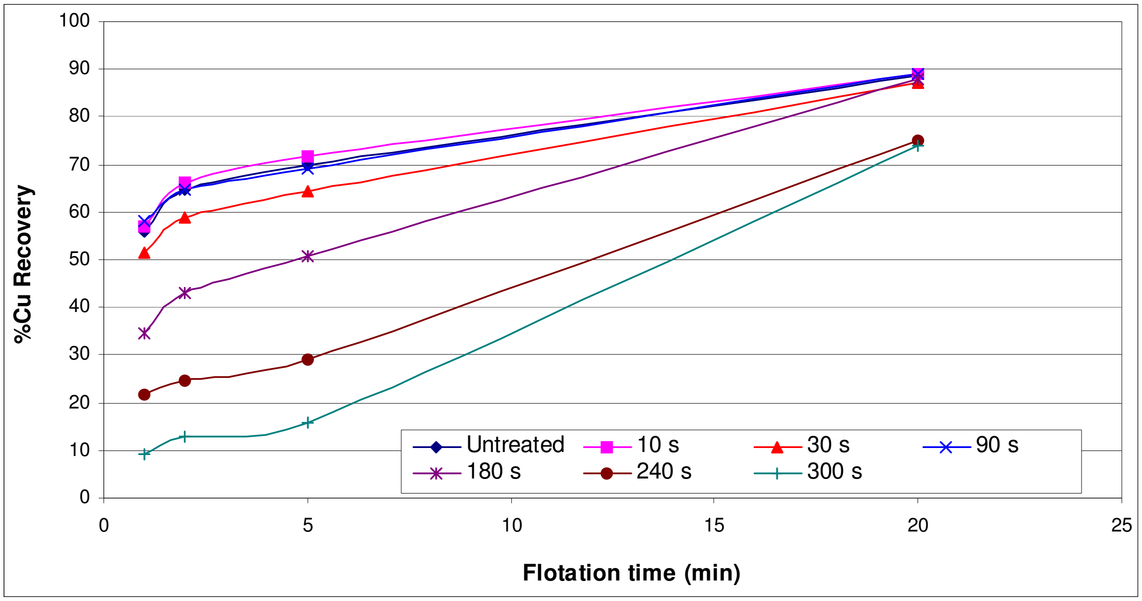


Figure 8.24 : Effect of microwave radiation on copper recovery (air environment - after Kingman et al, 2000)

Figure 8.24 illustrates the significant effect of microwave radiation on copper recovery during froth flotation.

For the sample exposed to microwave radiation for 10s, the recovery after 5min is 69.7% compared to a recovery of only 68.8% for material not exposed to microwave radiation. A possible reason for the increase in recovery could be that the copper minerals have been liberated more effectively from the gangue minerals.

For short exposure times (approximately 10s), no detrimental effects on copper recovery are observed. For increased exposure times, however, copper recovery is reduced due to oxidation of copper sulphide species and/or partial melting (as illustrated in Fig 8.12A and Fig 8.12B). Exposing the ore to microwave radiation in an inert atmosphere (such as nitrogen) will not result in the ore oxidizing. Figure 8.25 shows the copper recovery for ore treated in a microwave heater in an inert atmosphere.

Slight increases are evident in copper recovery for the ore treated in a nitrogen atmosphere for 10s and 30s. Longer exposure time result in a decrease in copper recovery. For 10s treatment after 5min flotation, the copper recovery was 72.7% compared to the untreated recovery of 68.7%.

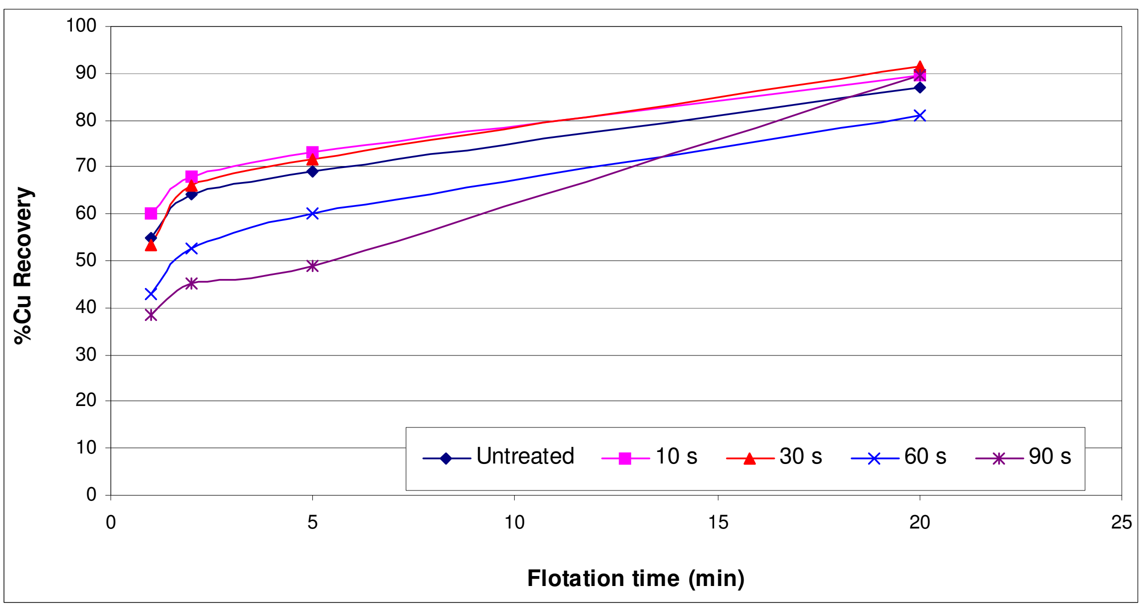


Figure 8.25 : Effect of microwave radiation on flotation (inert atmosphere - after Kingman et al, 2000)

Figure 8.26 illustrates the effect of microwave radiation on recovery of ferrous Fe during magnetic separation trials.

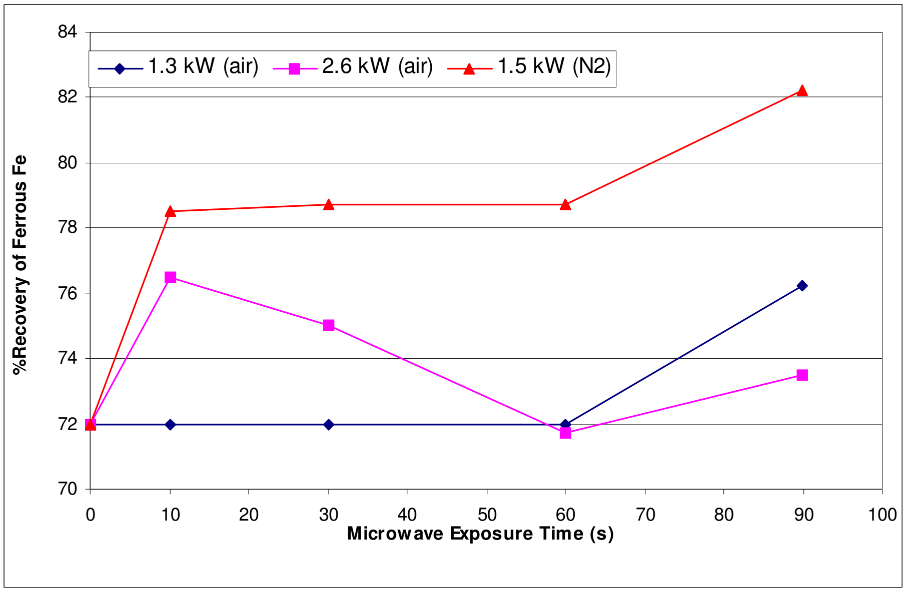


Figure 8.26 : Effect of different power level microwave radiation treatment on the recovery of ferrous Fe (after Kingman et al, 2000)

Figure 8.26 illustrates that microwave radiation had significant effects on the recovery of iron during magnetic separation trials. Within 10s of microwave exposure (at 2.6kW) the recovery is 77% compared to only 72% for untreated ore. For prolonged microwave radiation (in excess of 90s) the recovery of iron would have decreased due to the oxidation and partial melting of mineral species in the ore. Increased recoveries are most likely due to increased mineral liberation due to microwave exposure.

8.5.4) The use of the Drop Weight Testing Rig to quantify rock strength

The results and calculations may be seen in Appendix D20. The plot of t10 versus for the Palabora ore is shown in Fig 8.27 :

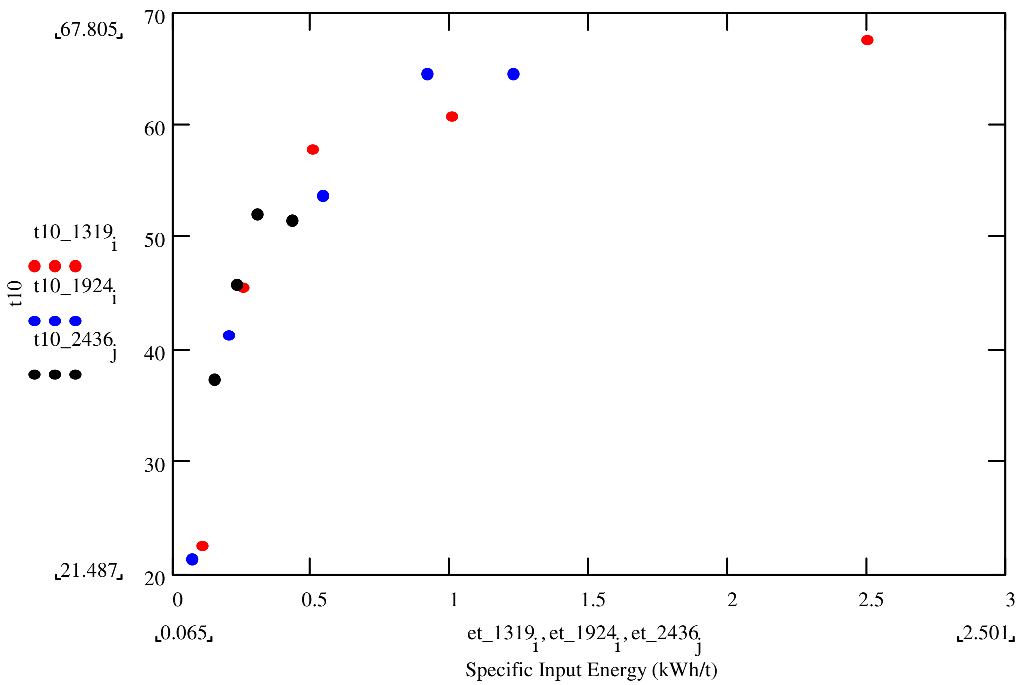


Figure 8.27 : Plot of t10 vs Ecs for 3 size classes of Palabora ore (red 13-19mm, blue 19-24mm, black 24-36mm)

Using the method of least squares to fit the curve (described by Eq 3.15, see Chapter 3) :

$$t10 = A\left(1 - e^{-b\,E_{cs}}\right) \qquad \text{Equation 3.15}$$

A brief summary of the results (values of A and b as well as the calculated value for the mode one fracture (Kic) are given in Table 8.2.

Table 8.2 : Summary of A, b and Kic values

Material		**A**	**b**	**Kic** (MN.m1.5^{-1})
Palabora	Combined	63.133	5.306	0.614
	13-19 mm	64.940	4.577	0.663
	19-24 mm	62.735	5.478	0.604
	24-36 mm	54.459	8.306	0.500
Microwaved Palabora		56.437	4.587	0.687
Yorkstone		59.735	2.353	0.987 (1.04 - Nott)
Green Granite		59.969	1.678	1.194 (2.11 - Nott)

An interesting trend that may be observed from Table 8.4 is that the value of Kic decrease with increasing particle size. This is to be expected as larger particles are generally weaker than smaller particles.

In order to test the accuracy of the drop weight results, tests were conducted on Yorkstone and green granite to compare the Kic values with those that were calculated from tests conducted at Nottingham University. As can be seen from Table 8.4 the results for Yorkstone compare extremely well whereas those for Green Granite are not as close (although still in the same order of magnitude).

Tests were also conducted on Palabora ore (13-19mm) treated in a microwave heater at 2.6 kW for 60s. Previous work have indicated that large reductions in work index are possible for Palabora ore. However, tests conducted using the drop weight tester, indicates that the microwaved ore is stronger than the untreated ore. As the ore was not exposed to such an extent that partial melting should have occurred - it was expected that the ore would have been weakened. However, Palabora ore consists of large proportions of non-microwave receptive dolorite which are extremely hard. It is conceivable that the 50 particles used for microwave treatment and subsequent testing, consisted of slightly more dolorite particles and would therefore not be weakened by the pre-treatment.

In addition, it is also possible that the mass was being released from an excessive height, i.e. an excessive amount of energy was being used to crush the ore, making small changes in strength impossible to detect.

8.6) Conclusions

Experimental work has shown that the mineralogy of Palabora ore may be affected by microwave radiation, the extent of the change are dependant on the time of exposure. Extended exposure times do not necessarily yield larger reductions in work index, with the main reduction in work index taking place in the initial period.

It has been shown that treatment of up to 165 kg/h of Palabora ore is possible on a pilot scale microwave unit. No reduction in work index was noticed during pilot scale milling or laboratory trials after continuous microwave treatment. This is attributed to the constant load in the microwave heater.

It is postulated that a semi-continuous system of treating batches of 5 kg (continuously changing the load in the cavity) will overcome this problem, although it will result in only half the feed (i.e. 83 kg/h) on the current system. A pulsating power source might also be used to achieve the same effect.

Various experiments regarding the area and mass loading in the microwave cavities, were conducted. These showed that each microwave cavity has an optimum area of treatment as well as an optimum mass load. These should be found for any microwave unit before full scale operation will be possible.

Thus far all microwave units were basically domestic microwave units - designed with the dielectric properties of water. More care will have to be taken in future design of cavities - to adapt to the specific properties of the ore.

The mixture equations of Arai et al, 1995, has been shown to hold for the Palabora ore. This ore is “ideal” in that it consists of mainly 2 components - magnetite and gangue (assumed to be calcite). It has also been shown that the three point bend test may be used as an alternative method to quantify the effect of microwave radiation on the ore.

It has also been illustrated that, in order to do a full economic evaluation of the feasibility of microwave pre-treatment of Palabora ore, attention will need to be paid to possible increases in recovery in downstream processing, in addition to the benefits regarding comminution energy.

Overall this study has shown the three-point bend test to be as reliable as the Berry and Bruce grinding method, in terms of its ability to quantify a decrease in rock strength due to thermal pre-treatment. Furthermore the Three-point bend test surpasses the Berry and Bruce method in that it is easily reproducible, requiring little sample preparation and considerably less time to conduct. In addition a large number of specimens can be produced from a relatively small volume of rock material, be it bulk sample or expensive drill core.

Initial trials conducted on Palabora ore, Yorkstone and green granite indicates that the drop weight test rig is capable of detecting different material strengths. It detects slightly different values for different particle sizes in the case of Palabora (increased particle size corresponding to lower values for Kic). This makes intuitively sense as larger particles are generally weaker than larger ones. Acceptable mode I fracture toughness values have been calculated for Yorkstone and green granite.

Further work is required to assess definitively the affect of various factors (particle size, ore strength, input energy etc) on the accuracy of the drop weight test procedure.

The following papers regarding work in this chapter have been published/accepted for publication :

1) "The effect of microwave radiation on the processing of Palabora copper ore" by Kingman, Vorster and Rowson in the Journal of SAIMM, May/June 2000 vol 100, no 3
2) "The influence of mineralogy on microwave assisted grinding" by Kingman, Vorster and Rowson, Minerals Engineering, 2000, vol 13, no 3, pp 313-327, Pergamon
3) "Applications of microwave radiation for the processing of minerals" by Vorster, Kingman and Rowson in the Proceedings of the 9th SAICHE National Meeting, 2000
4) "The use of the three point bend test to quantify the effects of thermal pre-treatment on rock strength" by Campbell, Vorster, Merchant and Rowson in Minerals Engineering

These papers have been included in the back of this thesis, under the "Papers"-section.

In addition two presentations have been delivered regarding this work (both of these presentations have been included on the CD at the back of this thesis) :

1) "Applications of microwave radiation for the processing of minerals" was delivered at the 9th SAICHE National Meeting in October 2000 (saiche00.exe)

CHAPTER 9

ECONOMIC IMPLICATIONS OF MICROWAVE PRETREATMENT OF MINERALS

(Appendix E : Usimpac Simulations is on the CD at the back of this thesis)

9.1) Introduction

Esk (1986) conducted a study on the energy requirements of mineral processing circuits. Of particular interest is the breakdown in energy consumption for a copper sulphide concentrator :

Operation	**Electrical Energy Usage (kWh/t)**	**Operation**	**Electrical Energy Usage (kWh/t)**
Crushing	2.04	Filtration	0.21
Grinding	9.8	Regrinding	0.26
Flotation	1.28	Water Pumping	2.8

Table 9.1 : Energy requirements of a copper sulphide concentrator (after Esk, 1986)

Table 9.1 illustrates that 62% of the energy in the concentrator is consumed by the grinding stages alone. It has been estimated (Napier-Munn et al, 1999) that 1.5% of the total energy consumption in the U.S. is utilized in the comminution processes of mineral concentrators alone. Accurate prediction of process energy requirements for laboratory microwave treatment is difficult. Scale factors (surface to volume ratio and heat loss effects), difficulties in calibration of power sensors, non-optimal applicator designs with high reflected powers and low magnetron efficiency contribute to the difficulty in scale-up estimations.

In this section the effect of microwave radiation on Neves Corvo and Palabora copper ores on the comminution circuit are determined by simulation on the mineral processing package, USIMPAC. Actual plant data (of Somincor and Palabora) was used to configure and test the models prior to running iterations.

9.2) USIMPAC Simulations

9.2.1) Neves Corvo Flowsheet Simulation (flow sheets and results in Appendix E1)

The software was used to model the comminution circuit at Somincor Ltd. The flow sheet is shown in Figure 9.1 :

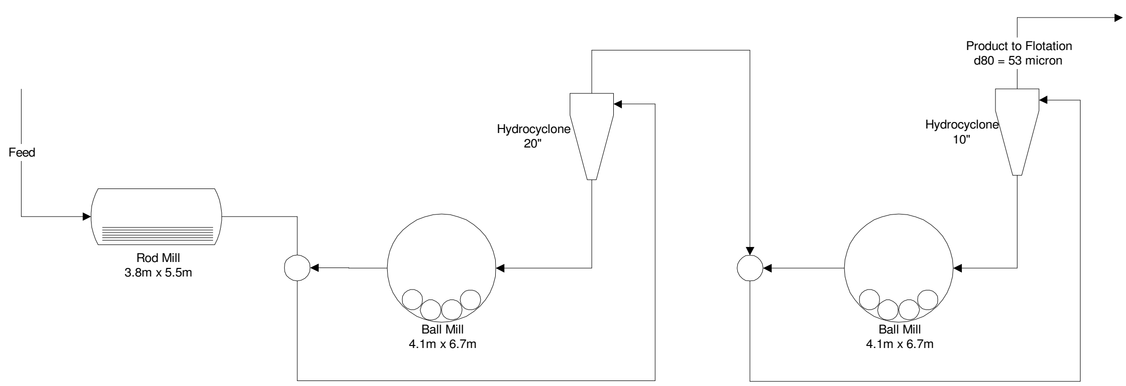


Figure 9.1 : The comminution circuit at Somincor Ltd (Anon, 1994)

The following information was required for the simulation :

	Diameter and length	Rod/ball SG
Mills :	% Critical Speed	Work Index of material (kWh/t)
	Volumetric loading of rods/balls	Discharge method
Cyclones :	Cut point (μm)	% Solids in underflow

The model was configured, using actual plant data supplied by the mine, and simulated results compared well with site data. The effects of microwave treatment upon the ore was investigated by changing the parameter of Bond Work Index (10 kWh/t, 9 kWh/t, 7 kWh/t, 5 kWh/t and 3 kWh/t) in the first closed circuit ball mill and calculating the circulating load. .

Figure 9.2 shows a plot of circulating flow rate versus Bond work index :

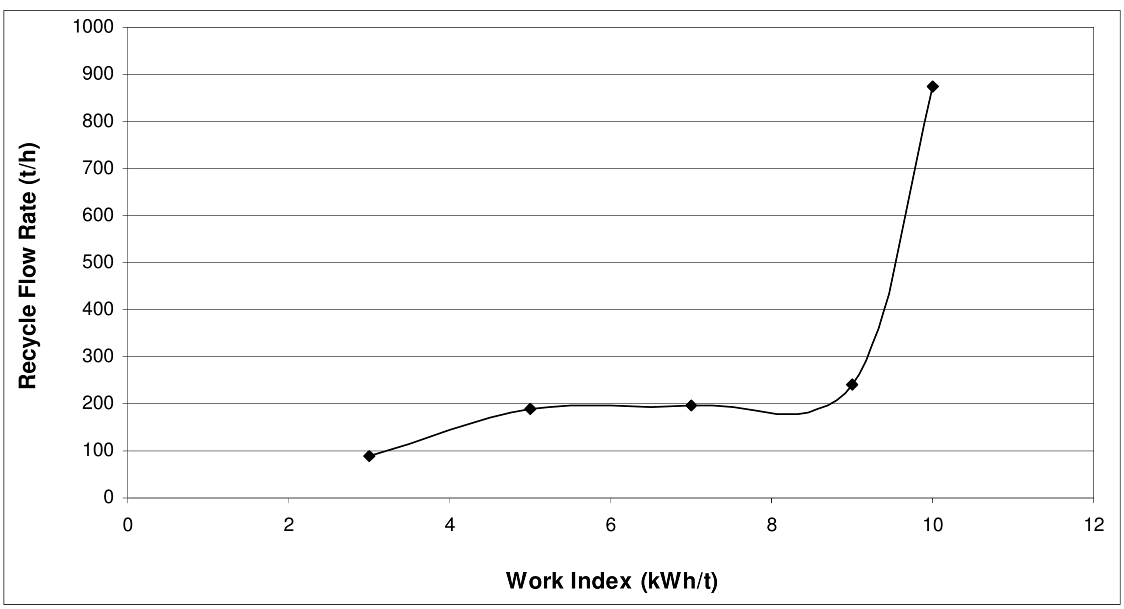


Figure 9.2 : The circulating load vs Work Index for the closed circuit ball mill

As Bond work index is decreased, a dramatic initial decrease in the circulating load is observed. In fact if the work index is reduced as far as 2kWh/t (as is limited by USIMPAC) the circulating load is virtually removed. This has significant implications for the energy requirements for the processing of this ore.

A plot of the particle size distribution of the product stream of the first section versus work index is shown in Figure 9.3 :

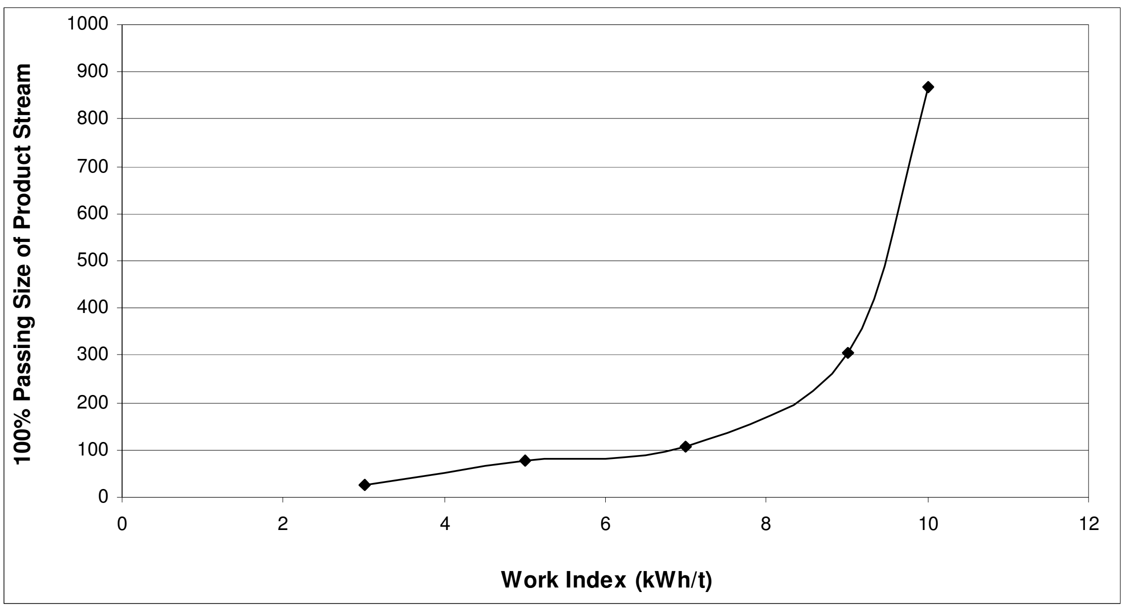


Figure 9.3 : 100% passing size of the product stream from the first closed circuit section versus work index.

The practical implication of reducing the Bond Work Index of the ore as low as 2kWh/t is to (according to the simulation) allow the same product size distribution at the same plant throughput with just the first section of the current comminution circuit, and a dramatically smaller closed circuit ball mill. In practise, reducing the Bond Work Index of the ore will significantly increase the plant throughput for a set flowsheet.

9.2.2) Palabora Flowsheet Simulation (flowsheets and results in Appendix E2)

USIMPAC software was used to model the conventional comminution circuit at Palabora. A simplified flowsheet is shown below :

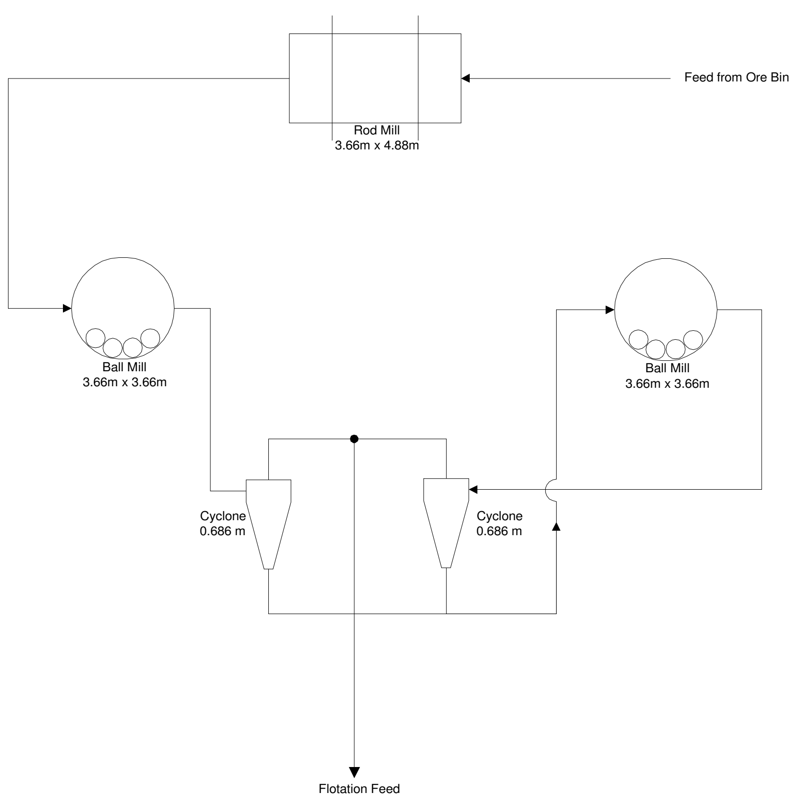


Figure 9.4 : Simplified flowsheet of the conventional milling circuit at Palabora

The accuracy of the model was determined by comparing the predicted circulating load with the actual circulating load on plant. The model predicted the circulating load to be 277% assuming a d_{80} input size of 16mm. The actual circulating load on plant is approximately 250%. It was therefore assumed that the model described the comminution process accurately enough for an economic analysis.

The effect of microwave treatment on the ore was quantified by changing the parameter of Bond work index and calculating the circulating load. The values of Bond work index used for the tests were obtained from previous work by Kingman (1999). The values of work index were input into the model for each mill and the model run as for the configuration test. Figure 9.5 hows a plot of circulating load versus Bond work index :

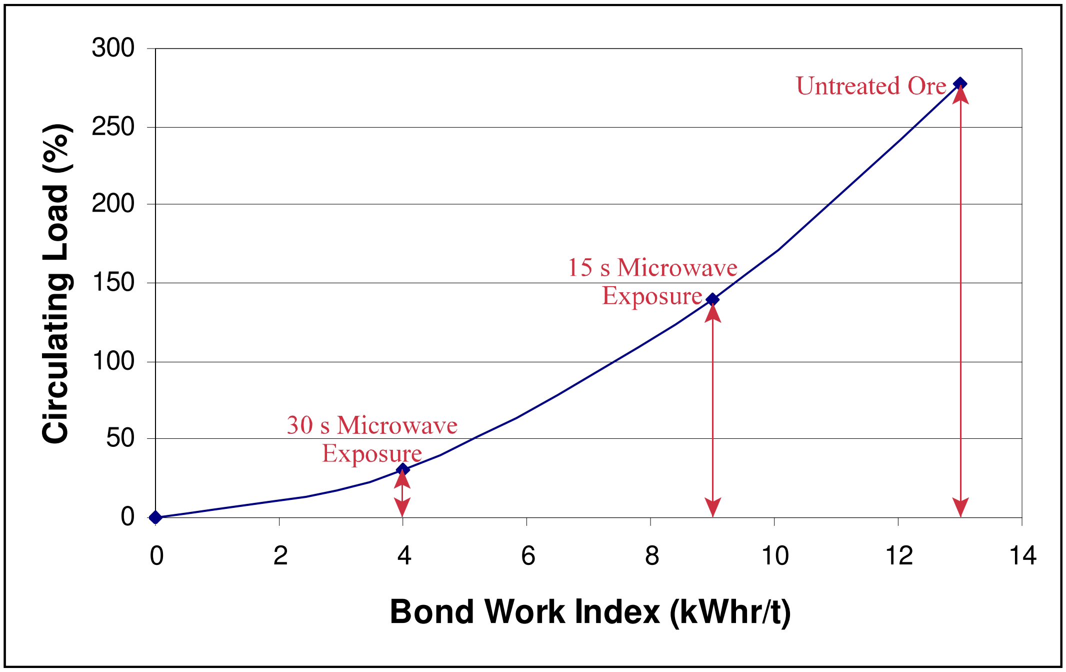


Figure 9.5 : Plot of work index versus %circulating load

As Bond work index is decreased so does the circulating load. In fact if the work index is reduced as far as 2 kWh/t (as is indicated possible by the experimental tests) the circulating load is removed.

9.3) Discussion and Conclusion

The effect of reducing the work index of both ores on the comminution circuits are illustrated. In the case of the Neves Corvo material, the second closed circuit ball mill may be removed after microwave treatment of the ore. The conventional grinding circuit at Palabora, currently consisting of 1 rod mill, 2 ball mills, the second ball mill with a recycle, can be reduced to only 1 mill in closed circuit, clearly having favourable economic implications. The removal of a mill from the current flowsheet is, however, impractical. Increased plant throughput (and hence increased production) is possible however.

However, one of the main protests against ongoing research in the application of microwave assisted thermally assisted liberation is that longer grinding times to increase the fineness of grind will also improve the metallurgical characteristics by increasing liberation. Exposing mineral ores to microwave radiation is at best simply application of energy in a different form which (it is argued) should rather be added to current comminution processes.

However, grinding minerals for extended time periods has the disadvantage of causing the production of slimes and therefore losses in down stream processing (gravity, magnetic separation and froth flotation being particularly sensitive to the presence of slimes).

Microwave pre-treatment of the ore has the effect of causing liberation at a higher particle d_{80} therefore giving rise to less slimes production. It is important to note that increases in recovery after microwave treatment are not solely due to the enhanced liberation of particles but also to liberation at a higher particle $d_{80.}$ Increased grinding times would also give rise to lower plant throughput (for the same flowsheet) and increases in wear to mill, media and liner. All these items are significant operating costs in any mineral processing plant. In contrast with increased grinding periods, microwave pretreatment increases plant throughput.

In the case of the Palabora ore the work index may be reduced from 13.1 kWh/t to 1 kWh/t in 180s at 2.6 kW, which corresponds to 130 kWh/t. For an exposure time of 10s, and a reduction in work index of 4.1 kWh/t, the energy input is reduced to only 7.2 kWh/t. Apart from the fact that 130kWh/t is extremely uneconomical, exposure for 180s is pointless as the micro structure as well as the mineralogy is destroyed. However, an exposure time of 10s gives a reduction in work index of 4.1 kWh/t compared to an microwave energy input of 7.2 kWh/t.

For an industrial concentrator, 62% of the total energy input is utilized in the comminution circuit (see Table 9.1). If a microwave system may be devised to increase the grindability of the ore by 50%, the energy requirements of the comminution circuit is reduced to 44% of the total energy cost. A total of 5 kWh/t is saved in comminution energy - which may be used for the microwave units.

The benefits of microwave treatment of ores do not extend solely to potential reductions in required grinding energy. Consideration must be given to increases in grade and recovery of valuable minerals, increases in plant throughput, liberation of valuable minerals at higher grind sizes therefore reducing slimes production, and also reductions in plant wear (particularly media and liner). In carrying out a full techno-economic assessment, however, the following points must also be considered. This test work was carried out using laboratory scale equipment. Such equipment is unlikely to have an energy efficiency of much above 45%. Industrial scale microwave systems have the advantage that they operate at 915MHz, with typical magnetron efficiencies of 85%. Penetration depths at 915MHz are also superior to those at 2.45GHz which would also provide greater flexibility in the design of the system.

Most of the industrial applications of microwave technology, excluding those in the telecommunications industry, are based on heating of water molecules. However, as Roddy (1986) states that “ovens and heaters for industrial applications generally have to be custom made for the particular application”.

Large scale microwave heaters may be adequate for drying purposes as they have been specifically designed for the dielectric properties for water, but the dielectric properties of minerals differ vastly from that of water. Industrial scale microwave equipment is available that will produce about 75kW in continuous operation. For mineral processing plants such as copper concentrators, many generators would required and the capital cost of such a systems will undoubtedly be significant. It will be vital, therefore, that energy input to the system be minimized

Although microwave technology is well established for domestic applications [Kingman et al, 2000], various obstacles hinders its acceptance for industrial applications. The practical application of the technology on the scale required to treat many thousands of tonnes per day - continuously - is one of the most difficult obstacle to overcome. Coupled with the safety aspects of operating such a piece of equipment makes for a daunting project. However, with the ever decreasing grades and reserves of ore, microwave technology may offer a unique solution.

CHAPTER 10

CONCLUSION AND FURTHER WORK

10.1) Conclusion

Experiments were conducted on four different types of material - Palabora carbonatite copper ore, Neves Corvo massive copper and massive copper-zinc ore, Mambula iron-titanium ore and vermiculite from Palabora. The following conclusions has been drawn from the series of experiments conducted on these materials.

10.1.1) Grindability

The work index of the Neves Corvo massive copper ore has been determined to be 9.7 kWh/t.

Of the grindability trials, which were conducted on Palabora, Neves Corvo and Mambula, the Palabora ore showed the largest reduction in work index - up to 80% (in a 6.6kW microwave cavity with a load of 5kg and exposure of 110s followed by quenching). The maximum reduction in work index achieved in the case of Neves Corvo was 70% (for 500g massive copper-zinc material) after 90s exposure at 2.6kW, whereas the Mambula material only showed a reduction in work index of 26% after 90s exposure at 2.6kW.

This highlights the importance of the mineralogy of the ore on the effectiveness of microwave pre-treatment of minerals (as is reported in Kingman et al, 2000). Ores of consistent mineralogy containing a good microwave absorber in a transparent gangue matrix are most responsive to microwave treatment, whereas ores containing small, finely disseminated particles in discrete elements respond poorly and yields only small reductions in work index.

The reduction in work index has been shown to be due to extensive intergranular fracture which occurs when different species within a particle respond (i.e. heat up) at different rates to the applied field. This causes differential expansion which generates internal stresses at the grain boundaries, weakening the mineral structure. In the case of the Mambula ore, none (or very little) structural and mineralogical changes due to microwave exposure occurred. Partial melting and a small degree of oxidation did occur, but these changes were not significant enough to affect the grindability.

10.1.2) Flotation

Flotation trials have only been carried out in the case of the massive copper and copper-zinc ore form Neves Corvo. Although extensive intergranular fracture occurred (which might have led to increased mineral liberation) as well as some changes to the mineral surfaces (bornite formation, oxidation and partial melting) no change was detected in the recovery of copper during laboratory froth flotation trials.

10.1.3) Magnetic Susceptibility

No significant change in Fe-recovery between samples exposed to microwave radiation and the reference samples can be observed in the case of Mambula ore. There is some improvement in Ti-recovery in the first concentrate (unwanted). However, this would need to be confirmed by extensive further test work. The Ti content in the second concentrate (0.8T concentrate) appears to drop. This may be due to changes in magnetic susceptibility of the ilmenite phase.

This has been confirmed by the mineralogy. Oxidation did occur to a small extent, as did partial melting and the formation of gas bubbles. But these changes are not frequent throughout the sample and do not affect the magnetic susceptibility of the Mambula ore.

10.1.4) Vermiculite Exfoliation

It was discovered that the Palabora vermiculite has the unique (amongst micaceous minerals) property of being responsive to microwave exposure. When exposed to microwave radiation of sufficient power level, rapid exfoliation occurs.

It was found that the large and medium grade material respond best (in comparison to the fine, superfine and micron grade material). The volumetric expansion of the large and medium garde vermiculite obtained in the 2.6kW microwave cavity correspond well to values quoted in literature.

In the case of the finer grades of vermiculite, the volumetric expansion is much less than the values quoted in literature. However, comparative tests in a conventional furnace regarding the volumetric expansion, showed that microwave exposure yielded a larger volume per unit mass of vermiculite in a shorter period of time.

From experimental work, it was determined that large grade vermiculite may be exfoliated continuously, utilizing a total 352 MJ/t without the need for additional cooling. In the case of current industrial microwave exfoliation equipment the energy consumption ranges between 1.24 and 1.99 GJ/t and yields a product which needs to be cooled down from 1200°C to 35°C utilising an additional 1.2 GJ/t.

10.1.5) Cavity Effects

Experimental work revealed that the design of the cavity constitutes an important section in the economical application of microwave technology. The way in which the material is presented to the electric field also plays an important role.

There seems to exist a correlation between percentage reduction in work index and the particle size of the material. The larger the particles, the higher the reduction in work index. This makes sense intuitively, as the larger material will have a greater probability of consisting of a mixture of microwave receptors and insulators which would assist in the reduction in work index. There would be an upper limit to the particle size, related to the penetration depth and exposure time required for heating.

For smaller particles, there is a higher probability that the particles will consist of only a single phase and therefore differential heating will not occur, and subsequently the reduction in work index will be lower.

Therefore it may be deduced that there will be an optimum size for microwave pre-treatment of minerals to ensure a maximum reduction in work index.

The mass and area of the material in the microwave cavity also plays an important part in the effectiveness of microwave pre-treatment. Under similar exposure conditions (residence time) a small sample mass would respond faster than a large mass of material. In addition there is an optimum area within multimode microwave cavities where the best response of the minerals to the applied electric field is achieved.

It has also been illustrated that the size of the microwave cavity and the electric field strength has a large effect on the efficiency of microwave pre-treatment. A high power, large volume cavity may be less efficient than a lower power, small cavity with a higher electric field strength. This is due to the fact that the absorbed power is proportional to the square of the electric field strength.

10.1.6) Flowsheet Simulation

It has been illustrated that the simulation package USIMPAC, may be used to accurately model an existing plant operation as well as predict changes to the flow sheet as a result of reduced Bond Work Index.

In the case of the Palabora flowsheet, the plant consisting of a rod mill, ball mill and a second closed circuit ball mill, may be reduced to a single closed circuit mill to achieve the same final particle size distribution after a reduction in work index. In practise, microwave pre-treatment would not be used to reduce the work index of the material to such a great extent (as this affects downstream processing), but plant throughput may be increased.

10.2) Further Work

The following areas have been identified for further investigation regarding the economic and industrial application of microwave pre-treatment of minerals.

10.2.1) Dielectric Property Calculations and Modelling

The dielectric properties of a material determines its response to microwave radiation with respect to heating rate, penetration and skin depth. Accurate data regarding the dielectric properties of minerals are required to calculate these fundamental parameters.

However, ores seldom consist of single mineral species. In reality, ores may contain dozens of mineral species as is the case in Palabora copper ore which contains at least 30 different minerals.

Calculation of the dielectric properties of the bulk of the ore is extremely difficult. The main problem lies with finding a representative sample of the ore to be used to calculate the bulk dielectric properties. Arai et al, 1995, developed a model which may be used to estimate the dielectric properties of a binary mixture of mineral species.

This work may be extended to include more species, which would make the calculation of the dielectric properties possible using the composition of an ore and the dielectric properties of the individual components. It should also be noted that when using the dielectric mixture equations (for powders) that there is a third component - air - in between the solid particles. This will affect the dielectric properties. It would be advisable to determine the dielectric properties of pure, solid minerals in the form of drill cores.

Once data has been collected for a certain ore type, calculation of the heating rate, penetration depth and skin depth may be carried out. Data on the penetration depth and skin depth may be combined with experimental data to determine the optimum particle size for microwave pre-treatment. Data regarding the heating rate may be used to predict temperatures of different species within the ore material after exposure for different time periods. If the heating rates of the individual species are known then it may be predicted to what extent the constituents of the ore will expand on heating and contract upon quenching.

Once the volumetric response of the mineral species have been determined, the magnitude of the stresses induced by the expansion and contraction on the grain boundaries may be evaluated (possibly by the finite element method). This may assist in determining the susceptibility of a certain ore body to microwave radiation and whether or not a significant reduction in work index is possible or not - without the need for experimental work.

The thermal and volumetric response of the material may also be used to calculate the effects of thermal pre-treatment due to microwave exposure, on the surface properties of the material, which affects processes like flotation and magnetic separation.

10.2.2) Scale-up

Before any process can be applied to an industrial process it need to be tested at pilot scale. Therefore pilot scale testing need to be conducted on the ore types from Palabora, Neves Corvo and Mambula with regard to microwave exposure. This should be done at high power level and electric field to ensure the shortest possible exposure times (for economic benefits).

After microwave exposure, these materials need to be milled in pilot scale milling equipment en results (particle size distribution, throughput, energy consumption etc) compared to unexposed material, milled in the same equipment.

Tests also need to be conducted to ensure that downstream processing such as flotation and magnetic separation are unaffected (or affected positively).

10.2.3) Other Work

- Walkiewicz (1991) claimed that microwave pre-treatment will result in reduction in plant wear and increased mineral liberation. Thus far, work mainly focussed on the economical application of microwave assisted grinding.

  Work need to be carried out to investigate and quantify the effects of work index reduction on both plant throughput and plant wear. Increases in plant throughput may be quantified using the simulation package USIMPAC.

  Experimental work and modelling will be required to quantify the effects on plant wear due to a reduction in work index.

- The structural differences between vermiculite exfoliated in a microwave cavity and those exfoliated in conventional furnaces need to be further investigated.

- The use of the drop weight test rig need to be further investigated. In particular the effects of the release height, friction losses, rebound energy impact and particle size need to be further investigated before in order to be used to effectively determine the effect of microwave radiation on ore weakening.

It should be noted that just because an ore is judged unresponsive to microwave radiation (such as in the case of Mambula ore), this does not necessarily imply that the ore will never be affected by microwave radiation. In the case of Norwegian Ilmenite (not tested in this thesis) the composition is very similar to that of the Mambula ore. However, the ilmenite responds extremely well to microwave treatment - giving large reductions in work index. However, investigating the mineralogy of the ore reveals that included more unresponsive gangue materials in the matrix (whereas Mambula consists predominantly of good microwave receptors).

In the case of Norwegian Ilmenite, differential heating and expansion thus occurs readily, whereas the Mambula material heats up rapidly, but no differential expansion occurs. It may therefore be quite feasible that as the grade of the material decreases that the amount of gangue in the matrix increases and the mineralogy gets closer to that of Norwegian Ilmenite and microwave treatment becomes a viable option once again.

Personal Communication

1
Mr K Pietersen, 2000
Richards Bay Minerals Ltd
Richards Bay, South Africa
Topic : *The effect of microwave treatment on the grindability and magnetic susceptibility of Fe-Ti ore from Richards Bay, South Afric*
Email : Kevin.Pietersen@rbm.co.za

2
Mr G Brown, 1999
Then at Svedala Ltd
Derby, UK
Topic : *Continuous pilot scale milling of Palabora copper ore in a rod mill*

3
Dr NA Rowson, 1998-2001
Department of Chemical Engineering, University of Birmingham
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Topic : *General discussions of microwave pre-treatment of mineral ore*
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4
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