

THE EFFECT OF HEAT TREATMENT ON CHALCOPYRITE

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The influence of conventional heat treatment and microwave radiation on chalcopyrite was investigated. There was a significant increase in the proportion of material recovered to magnetic fraction and magnetic susceptibility with conventional heating time. XRD analysis detected phase changes in conventional heat-treated chalcopyrite, which increases the magnetic susceptibility of the ore and enables its effective magnetic separation, which is impossible to achieve in its original state.

With microwave treatment, the magnetic susceptibility increases and the proportion of material recovered to magnetic fraction on the induced rolls is also increased. However, XRD analysis failed to detect any phase changes. A possible explanation for this observed behaviour is that the more magnetic component that has been formed by microwave treatment is below the threshold of detection of the XRD analyser. Taking into account that results from the froth flotation tests indicated that % weight fraction in the concentrate decreases with microwave exposure time, the change might be due to a surface effect.

Keywords: Microwave energy; Chalcopyrite; Magnetic separation; Thermal decomposition; Magnetic susceptibility; Froth flotation

INTRODUCTION

Chalcopyrite (CuFeS_2) is an industrially important mineral which forms an essential component of high quality copper ores. In view of its weak paramagnetism, the specific magnetic susceptibility of CuFeS_2 varies within the interval from 0.8×10^{-12} to $4.5 \times 10^{-12} \text{ m}^3 \text{ kg}^{-1}$, and the magnetic separation of this mineral requires a very strong magnetic field, which leads to low effectiveness of the treatment. The intensification of the magnetic properties of CuFeS_2 by means of heat treatment enables its effective dry separation in industrial magnetic separators [1].

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THERMAL BEHAVIOUR OF CHALCOPYRITE

Differential Thermal Analysis of CuFeS_2 shows a single oxidation reaction marked on thermal curves by a sharp exothermic effect. On the DTA curve, the beginning of the reaction is recorded at about 300°C . The curve becomes abrupt at about 340°C , having a maximum exothermic effect at about 450°C , the end of the reaction is at $600\text{--}650^\circ\text{C}$. The data [2] indicate that, at elevated temperatures, an endothermic effect appears at about 770°C , but the nature of this effect is not clear. It is believed to arise because the product remaining after oxidation attacks the crucible, when this is made of metal, or the thermocouple, when this is in direct contact with the sample [2].

EXPERIMENTAL PROCEDURE

Two separate batches of 1.5 kg lumps of Norwegian CuFeS_2 were obtained from a commercial mineral supplier (Gregory, Bottley and Lloyd) in order to carry out the described test programme. The lumps were jaw-crushed and then rolls-crushed with the gap set at the minimum aperture size possible in order to obtain a size fraction ranging from -500 up to $+250\ \mu\text{m}$.

The material obtained from the first batch was riffled in order to obtain approximately 100 g samples for conventional treatment tests. The material obtained from the second batch was riffled in order to obtain 150 g samples for microwave treatment tests.

Conventional Heat Treatment of Chalcopyrite

Representative 100 g samples of ore were placed in a furnace and heated at 250, 350, 450, 550, 650, 750 and 850°C . An untreated sample was used to provide baseline data. The samples were placed in the furnace for a period of 1 h after which they were taken out of the furnace and allowed to cool.

The samples were fed into an induced roll magnetic separator and separated at a magnetic field of 1.2 T. The field strength between the poles of the separator was set to 1.2 T using a calibrated gaussmeter. The feed rate and position were kept constant at all times.

After separation, the percentage weight of the magnetic and non-magnetic fractions at 1.2 T was noted. The magnetic susceptibility of the material was then measured using an Oxford Instruments vibrating sample magnetometer (VSM). The magnetic susceptibility of a material (χ , volume susceptibility) is dimensionless and is defined as the ratio of induced magnetisation to magnetic field intensity. It is expressed as [3]:

$$\chi = \frac{M}{H} \quad (1)$$

where χ is the magnetic susceptibility (dimensionless), M is the magnetisation (A/m) and H is the magnetic field strength (A/m).

A sample of 10 g from the magnetic fraction was TEMA milled and taken for X-ray diffraction to see any phase changes or decomposition that occurred. Powder diffractometry was carried out on the samples of CuFeS_2 to monitor any phase changes that might have occurred.

Microwave Treatment

For the microwave treatment tests, a Panasonic II 2600 multimode microwave oven was used. The microwave reactor could be operated at 650, 1300 and 2600 W power levels and at a frequency of 2.45 GHz. Representative 150 g samples of the ore were irradiated at a fixed power level of 2.6 kW. Irradiation times of 10, 30, 60 and 90 s were used. All samples were irradiated in a 14.7×8.5 cm fused silica dish, the position of which was kept constant in the microwave cavity. An untreated sample was used to provide baseline data.

The samples were then riffled in order to obtain 100 g for induced rolls tests and 50 g for froth flotation tests. The 100 g samples were fed into the induced rolls and separated at a magnetic field of 1.2 T. The field strength between the poles of the separator was set to 1.2 T using a calibrated gaussmeter. The feed rate and position were kept constant at all times.

After separation the percentage weight of the magnetic and non-magnetic fractions at 1.2 T was noted. The magnetic susceptibility of the material was then measured using an Oxford Instruments VSM.

A sample of 10 g from the magnetic fraction was TEMA milled and taken for X-ray diffraction to see any phase changes or decomposition that occurred in the magnetic fraction.

Flotation tests were then carried out on the 50 g samples of ore. All flotation tests were conducted in a 1 L Denver laboratory flotation cell with a constant impeller speed of 1500 rpm. Sodium isobutyl xanthate was used as a collector and was added at a dosage of 1000 g/t. Senfroth was used to stabilise the froth and was added at a dosage of 360 g/t.

The ore was added to 1200 mL of water and 2 min were allowed for conditioning to ensure total wetting of the particles before the addition of the collector. The collector was added and a further 2 min were allowed for conditioning the pulp. The frother was then added and 30 s of further conditioning were allowed. The air was then turned on fully and the scraping of the froth was continued for 5 min. After separation the percentage weight fractions of the concentrate and tails were noted.

RESULTS AND DISCUSSION

Conventional Treatment of Chalcopyrite

Heat treatment enables effective magnetic separation of CuFeS_2 , which is not possible to achieve in its normal state. It can be seen from Fig. 1 that as conventional heating time increases, the proportion of material recovered to magnetic fraction also increases significantly.

The thermal decomposition of minerals resulting from the thermal effect of heat treatment forms the basis of modification of magnetic properties of minerals. It produces phase changes in heat-treated minerals and gives rise to new phases, first of all on the surface of the grains. These newly developed phases differ from the original mineral by considerably higher susceptibility and for this reason increase the average value in individual grains of the irradiated mineral [4]. It can be seen from Fig. 2 that significant changes in magnetic susceptibility are apparent with CuFeS_2 exhibiting a significant increase in magnetic susceptibility after heat treatment.

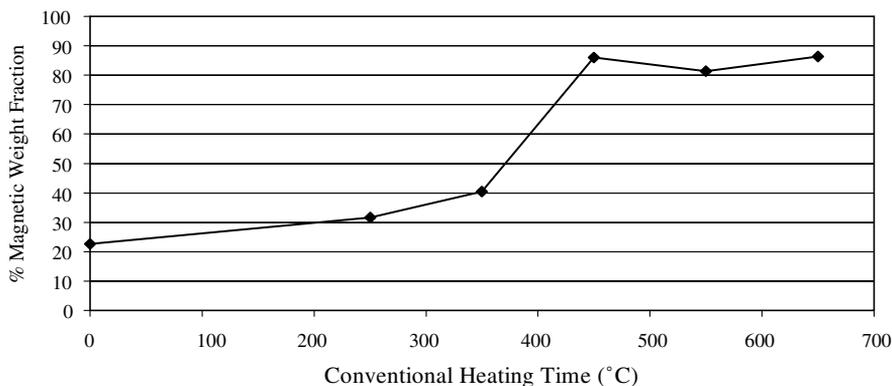


FIGURE 1 Plot of percentage magnetic weight fraction *versus* conventional heating time (°C).

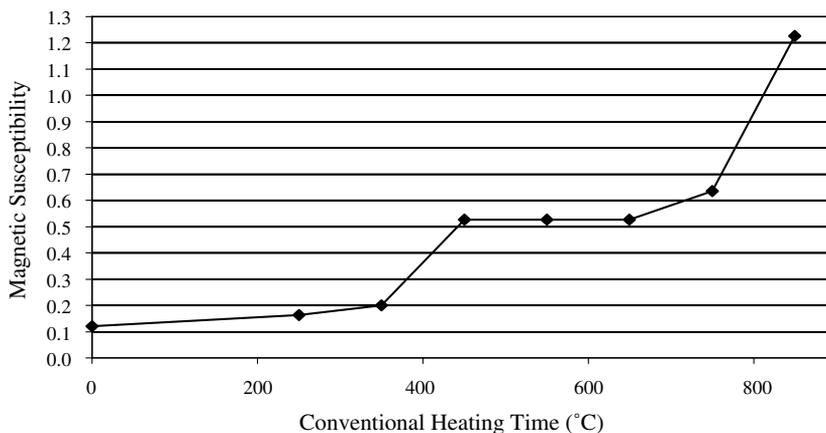


FIGURE 2 Plot of magnetic susceptibility *versus* conventional heating time (°C).

The X-ray diffraction analysis provided a qualitative estimation of the mineralogy of the samples, which is listed in Table I.

It can be seen from the table that in the non-heated sample pyrrhotite is present. However at 250 and 350°C pyrrhotite is replaced by troilite. This is due to a crystallographic change. It is clear that the elemental make-up of the sample is exactly the same. However, the relative proportions of each element have changed.

At 450 and 550°C troilite is replaced by haematite due to the oxidation process that is taking place. The oxidation processes that occur on the surface of grains result in formation of haematite, the magnetic susceptibility of which is approximately 100 times higher in comparison with that of CuFeS_2 [1].

At 650 and 750°C sulphurisation occurs as shown in the equation below [4]:

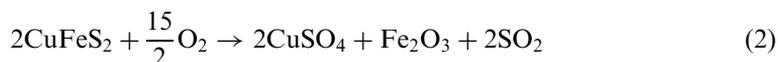


TABLE I Mineral phases identified by XRD

<i>Heat treatment (1 h)</i>	<i>Mineral phases identified</i>	
Non-heated	Azurite	$\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$
	Chalcopyrite	CuFeS_2
	Pyrite	FeS_2
	Pyrrhotite	FeS
250°C	Azurite	$\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$
	Chalcopyrite	CuFeS_2
	Pyrite	FeS_2
	Troilite	FeS
350°C	Azurite	$\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$
	Chalcopyrite	CuFeS_2
	Pyrite	FeS_2
	Troilite	FeS
450°C	Azurite	$\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$
	Chalcopyrite	CuFeS_2
	Haematite	Fe_2O_3
	Pyrite	FeS_2
550°C	Azurite	$\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$
	Chalcopyrite	CuFeS_2
	Haematite	Fe_2O_3
	Pyrite	FeS_2
650°C	Azurite	$\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$
	Chalcocyanite	CuSO_4
	Chalcopyrite	CuFeS_2
	Haematite	Fe_2O_3
750°C	Azurite	$\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$
	Chalcocyanite	CuSO_4
	Chalcopyrite	CuFeS_2
	Haematite	Fe_2O_3
850°C	Azurite	$\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$
	Chalcocyanite	CuSO_4
	Chalcopyrite	CuFeS_2
	Maghemite	Fe_2O_3

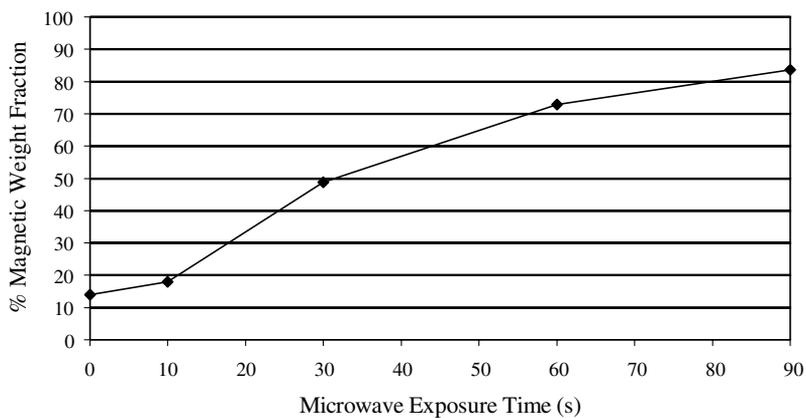
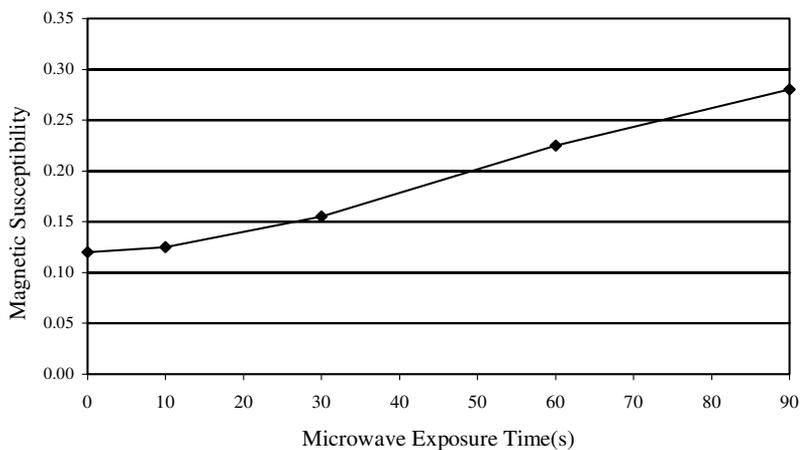
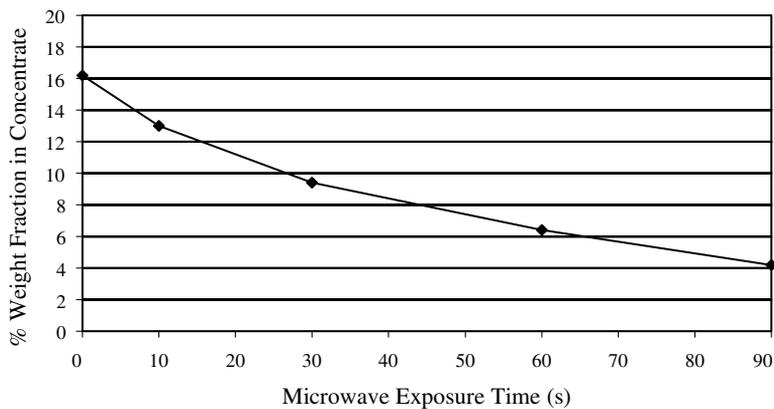
At 850°C haematite is replaced by maghemite due to another change in the crystalline structure.

Microwave Treatment of Chalcopyrite

It can be seen from Fig. 3 that as microwave exposure time increases, the proportion of material recovered to magnetic fraction increases significantly.

Moreover, it can be seen from Fig. 4 that changes in magnetic susceptibility are apparent with CuFeS_2 exhibiting an increase in magnetic susceptibility after treatment.

After microwave treatment, the magnetic susceptibility increases and the proportion of material recovered to magnetic fraction on the induced magnetic rolls is also increased significantly. However, XRD analysis failed to detect any phase changes and the only minerals detected by XRD before and after microwave treatment were quartz and CuFeS_2 . A possible explanation for this observed behaviour is that the more magnetic component that has been formed by microwave treatment is below the threshold of detection of the XRD analyser. Taking into account the results from

FIGURE 3 Plot of percentage magnetic weight fraction *versus* microwave exposure time (s).FIGURE 4 Plot of magnetic susceptibility *versus* microwave exposure time (s).FIGURE 5 Plot of percentage weight fraction in concentrate *versus* microwave exposure time ($^{\circ}$ C).

the froth flotation tests, the change might be due to a surface effect as flotation is a physico-chemical process, which depends on complex phenomena occurring at the surfaces of mineral particles and air bubbles in water [5].

It can be seen from Fig. 5 that the percentage weight fraction in the concentrate decreases with microwave exposure time.

CONCLUSIONS

It can be concluded that modification of magnetic properties caused by decomposition processes in CuFeS_2 enables a considerable increase in the effectiveness of its magnetic treatment. The modification of magnetic properties can be applied only to minerals that contain iron or other elements capable of forming strongly magnetic compounds. Heat treatment increases the magnetic susceptibility of CuFeS_2 ore and enables its effective magnetic separation, which is impossible to achieve in its original state.

The influence of conventional heat treatment on CuFeS_2 showed that there was a significant increase in the proportion of material recovered to magnetic fraction and magnetic susceptibility with conventional heating time. XRD analysis detected phase changes in conventional heat-treated CuFeS_2 , which increases the magnetic susceptibility of CuFeS_2 ore and enables its effective magnetic separation, which is impossible to achieve in its original state.

With microwave treatment, the magnetic susceptibility increases and the proportion of material recovered to magnetic fraction on the induced magnetic rolls is also increased significantly. However, XRD analysis failed to detect any phase changes. A possible explanation for this observed behaviour is that the more magnetic component that has been formed by microwave treatment is below the threshold of detection of the XRD analyser. Taking into account the results from the froth flotation tests, which indicated that the percentage weight fraction in the concentrate decreases with microwave exposure time, the change might be due to a surface effect.

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Caline Sahyoun graduated from the School of Chemical, Environmental and Mining Engineering at the University of Nottingham in 1999 during which she won the Institution of Mining Engineers prize in 1998 and 1999. She is currently completing her Ph.D. studies on the influence of microwave radiation on the liberation of minerals from ores. Ms Sahyoun's research is sponsored by Rio Tinto Technical Services and the Overseas Research Scholarship.