

MAGNETIZING ROAST FOR CHALCOPYRITE: EFFECT OF PARTICLE SIZE AND DILUTION

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Abstract In excess oxygen, 0-37 μm CuFeS_2 became magnetic at a temperature of 280°C . The magnetic product is copper ferrite, $\text{CuO}\cdot\text{Fe}_2\text{O}_3$. The 'magnetizing' temperature increased with particle size apparently following the trend reported for the ignition temperature. Dilution increased the magnetizing temperature; 80% dilution by PbS , MoS_2 or SiO_2 increases the temperature from 280°C to over 420°C . Sintering was a concurrent reaction. Nevertheless, a chalcopyrite/galena mixture after roasting and gentle grinding was readily separated on a Davis tube.

INTRODUCTION

De-coppering galena and molybdenite concentrates can pose severe difficulties, especially at fine particle sizes. One approach is high intensity, high gradient magnetic separation, appealing to the weak paramagnetism of CuFeS_2 , the principal Cu-mineral¹⁻². Some success is reported, but field strengths are high (2T) and capacities low. The magnetic susceptibility of CuFeS_2 can be greatly increased by gentle roasting³, which offers the prospect of reducing the field strength and increasing capacity. Such a magnetizing roast was used commercially at Ain Barbar, Algeria early this century in the separation of coarse (+ 3mm) chalcopyrite from sphalerite and galena⁴. Heating was at 525°C for 15-20 min.; the magnetic product was considered to be hematite ($\alpha\text{-Fe}_2\text{O}_3$).

Heating of CuFeS_2 to about 180°C is one of the most effective methods of de-activation in flotation. At the temperature

reached, no oxidation is expected. The magnetizing roast of chalcopyrite appears to be little studied, but is worth re-considering for fine sulfide separation. This note reports the effect of particle size and dilution on the magnetizing temperature and examines the reaction products.

EXPERIMENTAL PART

Chalcopyrite concentrate from Duval's Sierrita mill was employed. Material passing a 400 mesh screen ($37\ \mu\text{m}$) was isolated and sized fractions obtained by Cyclosizer. The dilution materials were galena (Pine Point Mines), molybdenite (Sierrita) and quartz.

A laboratory muffle furnace was employed. After many trials a procedure was developed. A two to three gram sample was placed in a clay crucible and spread evenly to a maximum thickness of 1 mm. The crucible was slowly heated, with the furnace door open, to the required temperature and held for 10 mins. Temperature was monitored by thermocouples placed on the crucible. The temperature did fluctuate (partly due to having an open door) but could be controlled manually to within 5°C . This was repeated at $5\text{-}10^{\circ}\text{C}$ temperature increments until the sample became magnetic. Three tests were devised to judge if the sample was magnetic: capture on the Davis tube after slurring; response on the Frantz Isodynamic Separator of $+15\ \mu\text{m}$ particles at 100 mA and a side slope of 20 deg. (the unreacted chalcopyrite responded at about 1000 mA); or simple response to a hand magnet. All gave the same result within experimental error. The hand magnet was the most convenient and was used for most of the work. The temperature was termed the 'magnetization temperature' and was repeatable within 10°C .

The procedure was important. For example, keeping the furnace door closed increased the magnetization temperature by about 100°C .

The separation tests were conducted on the Davis tube. The roasted sample was gently ground in a mortar and pestle for 1 min., slurried and passed through the tube at 0.6 cm/s water flowrate. Products were weighed and assayed by atomic adsorption spectrophotometry.

RESULTS

On heating, the chalcopyrite changed from green to black to brown. It was at the brown stage that the sample became magnetic, and also sintered. For example, the 0-37 μm sample was black by 250°C, then brown and magnetic by 280°C.

The effect of particle size is shown in figure 1, where results for Cyclosizer cone 2 (28 μm) and cone 5 (11 μm) are plotted along with the ignition temperature vs. particle size data of Volsky and Sergievskaya⁵. The present results appear to follow the same log-linear trend.

Figure 2 shows that increasing dilution increases the magnetization temperature. From 280°C the temperature rises to over 420°C for 80% w/w diluent, regardless whether it is SiO_2 , PbS or MoS_2 .

In all cases the sample sintered at the magnetizing temperature. The sinter could be readily broken, however. After roasting and grinding of $\text{CuFeS}_2/\text{PbS}$ mixtures separation was typically 50% Cu recovery to mags and 95% Pb recovery to non-mags.

X-ray diffraction (XRD) on magnetized CuFeS_2 identified $\alpha\text{-Fe}_2\text{O}_3$ and $\text{CuO}\cdot\text{Fe}_2\text{O}_3$ as the only reaction products. Electron microprobe analysis showed this was a thin, approximately 4 μm , surface layer. XRD on the roasted mixture of CuFeS_2 and PbS also revealed Pb_2O_3 and PbSO_4 .

DISCUSSION

The important magnetic phase is copper ferrite, $\text{CuO}\cdot\text{Fe}_2\text{O}_3$. Pure $\text{CuO}\cdot\text{Fe}_2\text{O}_3$ has a high saturation magnetization (about 1/3 that of

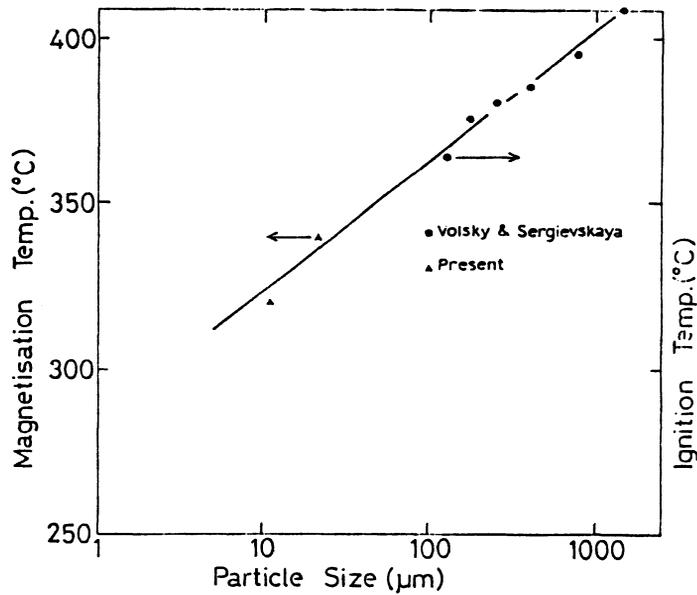


Figure 1. Magnetization (present work) and Ignition (Volsky and Sergievskaya (5)) Temperature vs Particle Size. (Scale on ignition axis is the same)

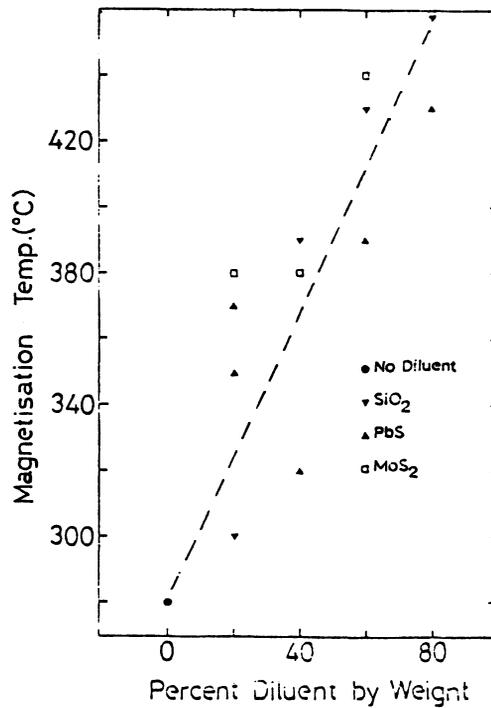
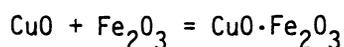
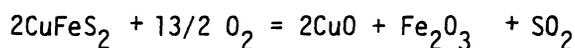


Figure 2. Magnetization Temperature vs Dilution.

magnetite, Fe_3O_4 ⁶); consequently relatively small amounts will impart strong magnetic properties. (The susceptibility increase indicated by the Frantz was over 1000 times). It can be speculated that this ferrite was also responsible for the success of the Ain Barbar process, rather than just $\alpha\text{-Fe}_2\text{O}_3$. The $\alpha\text{-Fe}_2\text{O}_3$ is responsible for the brown colour.

In the considerable literature on thermal oxidation of chalcopyrite there is relatively little on ferrite formation. The reaction is probably the following, which is strongly exothermic⁷:



The temperatures reported for ferrite formation are much higher than those here ranging from 500°C to 900°C. Thermo-dynamic analysis supported these higher temperatures. The ferrite here may be an intermediate product retained for kinetic reasons or a result of the exothermic reaction raising local temperatures to values at which ferrite forms. In agreement with the literature is the need for a plentiful supply of oxygen⁸, hence the need to keep the furnace door open.

Particle size and dilution are important variables. The effect of size appears similar to that reported for the effect on ignition temperature⁵. (No experimental details on that work were given, so further comment is not warranted). The size effect accounts for the low magnetization temperature of the 0-37 μm sample (280°C); the fines react first producing sufficient heat to cause the coarser particles to react. The heat generated is also responsible for the sintering.

The effect of diluents is a disadvantage, especially as removing small levels of chalcopyrite impurity was the target. The reason is presumably because heat is conducted away by the diluent preventing the temperature from reaching the level

necessary for ferrite formation. A method of roasting where the particles are not left in undisturbed contact may reduce the diluent effect. The diluent effect has been patented in a process to burn off adsorbed flotation reagents from Cu/Mo concentrates at elevated temperatures after first mixing with coarse quartz⁹.

Sintering is a recognized problem in roasting sulphides. Despite the sintering, a clean separation was readily achieved, however. Schemes to minimize sintering are clearly desirable.

As the apparent limit to selective separation by flotation is reached, around 1-5 μm ¹⁰, alternative schemes will become attractive. Low grade concentrates could be collected at a central facility for processing. In the present process the main difficulties would be controlling the roast conditions to maximize ferrite production while minimizing sintering and the effect of diluents. Also, a dry magnetic separation would be preferable.

CONCLUSIONS

1. 0-37 μm CuFeS_2 has a magnetization temperature (with excess O_2) of 280°C.
2. The magnetic phase is $\text{CuO}\cdot\text{Fe}_2\text{O}_3$.
3. Magnetization temperature increases with particle size apparently continuing the trend reported for the ignition temperatures.
4. Magnetization temperature increases with dilution.
5. Magnetization is accompanied by sintering.
6. After gently breaking the sinter produced by a $\text{CuFeS}_2/\text{PbS}$ mixture, 50% of the Cu could be removed from Pb as the magnetic product on a Davis tube.

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