

The Effect of Oxidation on the Flotation Behaviour of Nickel-Copper Ores

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ABSTRACT

Surface oxidation is one of the most important factors that influence the flotation selectivity and recovery in the processing of complex sulphide ores. It is an inherent part of surface chemical reactions that determine whether the surface film formed on a sulphide mineral will act as a hydrophobic or hydrophilic one. Because of the highly complex surface chemistry and mineralogy involved the results of flotation tests may often be difficult to interpret. In this paper, the flotation behaviour of nickel-copper ores processed in the Sudbury area is discussed with emphasis on the effect of oxidation on individual minerals, primarily pyrrhotite. In general, moderate oxidation of a freshly ground ore promotes the flotability of all principal sulphides in the ore leading to a bulk flotation concentrate. The ease of individual mineral flotability under normal aeration conditions, which is chalcopyrite > pentlandite > pyrrhotite, changes to pentlandite > chalcopyrite > pyrrhotite under oxygen-deficient conditions. This behaviour is correlated with the catalytic activity of these minerals for oxygen reduction.

On the other hand, preferential oxidation of pyrrhotite may lead to a selectivity increase in flotation. Such a behaviour has been noted in the flotation of a pyrrhotite-rich process stream. The results have been discussed in the light of fundamental electrochemical interactions involving pentlandite, chalcopyrite, pyrrhotite, pyrite and grinding media and a mechanism has been proposed to explain the selectivity.

INTRODUCTION

The nickel-copper ores of the Sudbury basin contain pentlandite, Pn (~36 per cent Ni), chalcopyrite, Cp (~34.5 per cent Cu) and nickeliferous pyrrhotite, Po (0.3-1.2 per cent Ni in a solid solution form) as the principal sulphides along with pyrite and other sulphides in relatively small and variable amounts. Non-sulphide gangue minerals consist of mainly quartz and feldspar along with minor quantities of tremolite, biotite, magnetite and talc. Pyrrhotite which typically represents 20 - 25 per cent of the ore, is intimately associated with other minerals, primarily pentlandite. It contains only about ten per cent of the nickel in these ores while containing over 75 per cent of the sulphur. Therefore, efforts to reduce sulphur dioxide emissions from the smelting process have included the rejection of pyrrhotite in the milling stage as an alternative means of treatment.

In the flotation process, a number of reagents are seen as a means of depressing pyrrhotite. However, the complex surface chemistry of the nickel-copper ores appears to challenge the effectiveness and consistency of these reagents; in many cases, testing indicates that pyrrhotite continues to float when it is not supposed to. Some of the contradictory results may be attributed to the superficial oxidation which is an integral part of the surface chemical reactions in sulphide flotation.

Sulphide minerals are subject to increasing surface oxidation during various milling stages. Naturally, this oxidation is minimal in a grinding process using steel - commonly mild steel - media, where the minerals experience mostly reducing conditions. The oxygen content and redox potentials of dense slurries at mill discharge point are lowest (Klassen and Mokrousov, 1963; Woodcock and Jones, 1969). Many concentrators in the Noranda group have practised aeration prior

to flotation to help depress pyrite and pyrrhotite and promote chalcopyrite and galena (Konigsmann, 1985). The effect of oxidation, however, can be quite complicated. For example, it has been reported to be beneficial for selective flotation of pentlandite in one case (Qun and Heiskanen, 1990) and detrimental in another case (Volkov *et al*, 1991). During a reagent testing program involving a pyrrhotite-rich stream, it was found in Falconbridge laboratories that none of the reagents tested produced consistently superior results with respect to pentlandite-pyrrhotite separation. However, a significant trend appeared to link better pyrrhotite depression with longer residence time in the flotation head tank, prior to any reagent addition. This flotation behaviour has been subsequently confirmed without using any specific reagent. The objective of this paper is to discuss the results of the latter study in comparison with the usual flotation behaviour observed in plant practice as well as in laboratory studies.

STRATHCONA MILL FLOWSHEET

The process flowsheet of Strathcona plant, as already reported elsewhere (Holmes *et al*, 1990), comprises two parallel nickel circuits, referred to as 'A' and 'B' circuits, processing about 200 stph each. 'C' circuit is fed from the Deep copper zone of Strathcona mine, which contains very little nickel, and treats about 40 stph high grade copper ore. The primary rougher concentrate feeds the copper/nickel separation column (column #1), producing copper concentrate and nickel-rich tailings. The latter reports to the final nickel-copper concentrate. The copper concentrate from column #1 is combined with the copper concentrate originating from 'C' circuit (Column #2) and sold to external processors. Secondary rougher concentrate reports directly to the final nickel-copper concentrate. Magnetic separators are used on scavenger concentrate to remove pyrrhotite. This magnetics fraction is reground to liberate pyrrhotite/pentlandite middlings, followed by flotation to selectively recover pentlandite. The non-magnetics fraction is thickened and floated in a single stage. Magnetics and non-magnetics concentrates report to the final nickel-copper concentrate which constitutes the feed to the smelter complex.

Currently, various options in the development of a new mill flowsheet are to be evaluated. The new flowsheet will enable increased amount of pyrrhotite rejection.

TEST SAMPLES AND PROCEDURE

A nickel-copper ore sample from rod mill feed was used in the first part of the testwork. In this case, 1.8 kg representative ore samples (-10 mesh) were ground using closed stainless or mild steel mill and rods under air or oxygen-deficient conditions (provided by using nitrogen) to obtain about 55 per cent -200 mesh. The samples used assayed 1.22 - 1.58 per cent Ni, 0.73 - 0.75 per cent Cu, and 9.7 - 12.1 per cent S. Standard flotation procedure, which simulated the primary rougher, secondary rougher and scavenger stages in Strathcona mill practice, involved the use of a four-litre Denver cell, air or nitrogen as the gas, sodium isobutyl xanthate as the collector, Dow froth 250 as the frother and lime as the pH modifier (for a value 9 to 9.5).

The second series of tests were performed on the process stream from the magnetics fraction of scavenger concentrate after

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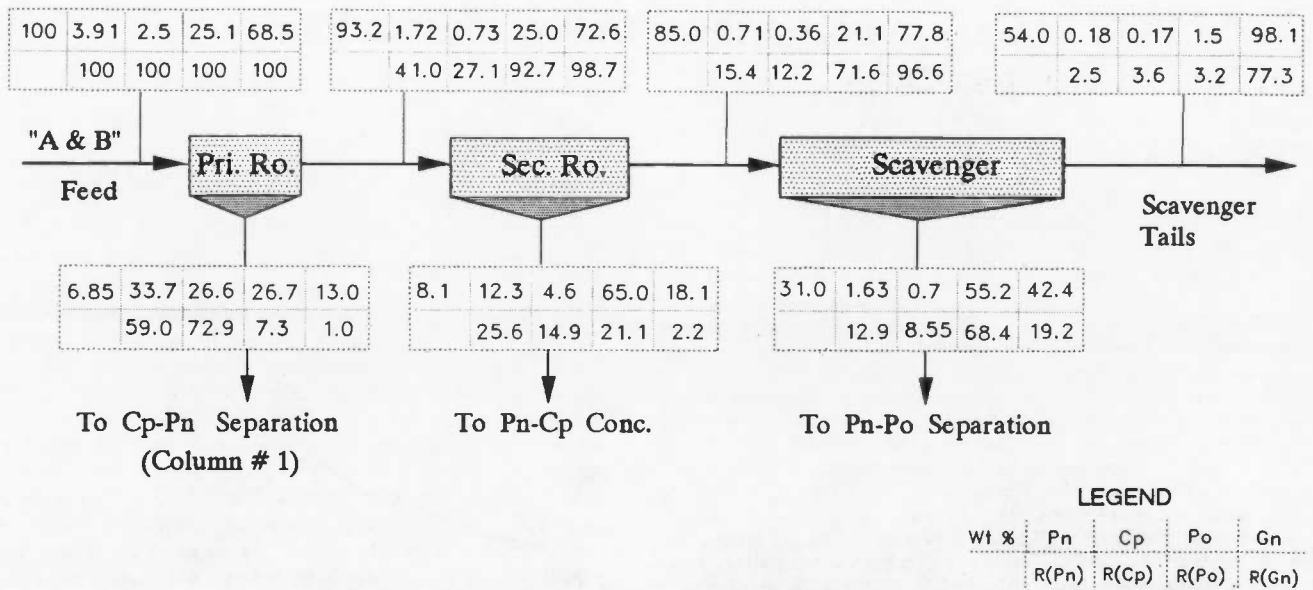


Fig 1 - A typical mineral balance of the rougher-scavenger circuits of Strathcona Mill - average performance based on two plant surveys (Note that, in near future, the mill flowsheet will be changed for increased amount of pyrrhotite rejection).

regrinding to a nominal size of 75 per cent -325 mesh. The slurry sample from regrind cyclone overflow was cut into plastic pails, brought to the testing facility and mixed in a 65 litre head tank in order to have a homogeneous and single source of flotation feed (pH: 9.5). The average composition of the flotation feed was 0.94 per cent Ni, 0.10 per cent Cu, 31.8 per cent S. Redox potential, dissolved oxygen, pH and temperature were measured in the stirred tank as well as in the four litre flotation cell. Slurry samples were periodically withdrawn from the head tank into the flotation cell and floated. For each test, the slurry was conditioned with about 10 g/ton of sodium isobutyl xanthate for five minutes before collecting the concentrates consecutively at 0.5, 1, 2, and 4.5 minutes during a total flotation time of eight minutes. The tests involved no pH adjustment and no frother usage due to the presence of residual reagents.

RESULTS AND DISCUSSION

Flotation behaviour in plant practice

A typical mineral balance of the rougher-scavenger circuits is given in Figure 1, based on the average of two plant surveys conducted in 1990. Currently, rougher service is provided by large flotation cells (ie 2.8 cubic metre cells replaced by 38 cubic metre cells). Pyrrhotite recoveries are cumulatively plotted in Figure 2 with respect to each flotation stage. At the primary rougher stage (first bank of six small cells), pyrrhotite recovery is only about seven per cent and it is not unusual in plant practice to see pyrrhotite recoveries even lower than five per cent. The pyrrhotite recovery nearly triples after about a two-fold increase in retention time (secondary rougher stage). After this stage, pH is lowered to about pH 8 by sulphuric acid addition. This is followed by copper sulphate addition to facilitate the activation and flotation of remaining pyrrhotite and other sulphides. As may be noted from Figure 1, the flotation rates of chalcopyrite and pentlandite are much higher than that of pyrrhotite. Thus, with its marginal pyrrhotite content, the primary rougher concentrate constitutes a suitable feed for the copper-nickel separation which is accomplished by column flotation in a highly alkaline pulp using lime (pH: 12) and sodium cyanide.

Since most chalcopyrite and pentlandite particles recovered in the primary rougher stage are liberated from pyrrhotite, the relative ease of individual mineral flotability in the processing of nickel-copper ores at Strathcona may be written as follows:

chalcopyrite > pentlandite > pyrrhotite

The same is seen in laboratory flotation with and without xanthate (Kelebek and Huls, 1991)

LABORATORY FLOTATION BEHAVIOUR

Primary flotation case

Results of the bench scale tests simulating plant's rougher-scavenger performance are given in Figure 3 which shows pyrrhotite recoveries obtained under the conditions summarised in Table 1. Grinding ore samples under air using

TABLE 1a

Typical conditions of the testwork using stainless steel/air.

Process	pH		Dissolved O ₂ (% sat)		Redox Potential, mV-SHE	
	before	after	before	after	before	after
Grinding	7.7	8.1	75-80	4-8	334 (H ₂ O)	221
Flotation	9.2	8.3	80-85	99-100	215	240

TABLE 1b

Typical conditions of the testwork using stainless steel/nitrogen.

Process	pH		Dissolved O ₂ (% sat)		Redox Potential, mV-SHE	
	before	after	before	after	before	after
Grinding	7.8	8.1	<1	<1	298	-55
Flotation	9.2	8.1	<1	<1	20	-80

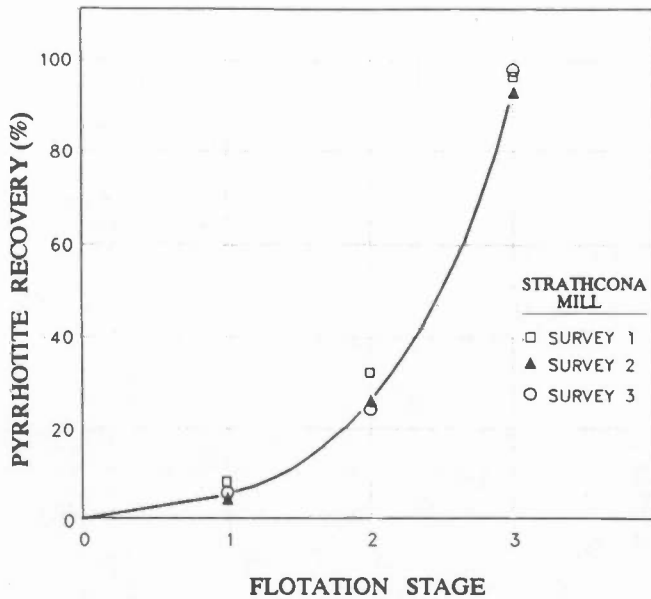


FIG 2 - Flotation of pyrrhotite in primary rougher (1), secondary rougher (2) and scavenger stage (3) in plant practice.

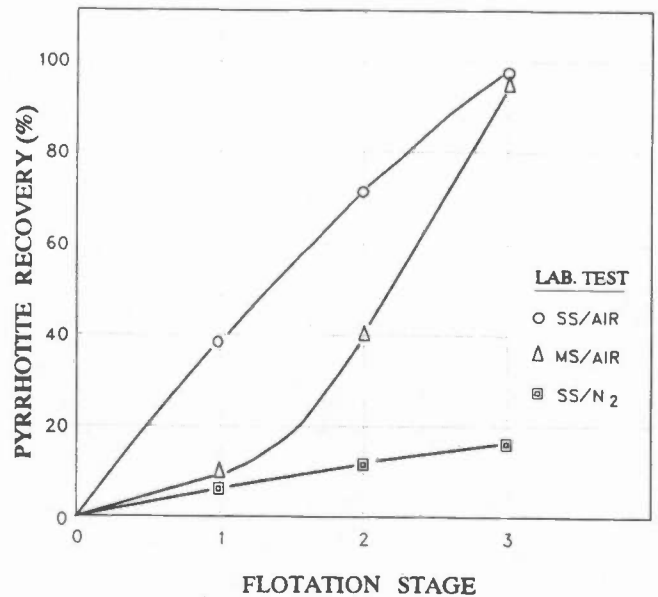


FIG 3 - Flotation of pyrrhotite in primary rougher (1), secondary rougher (2) and scavenger stage (3) in laboratory tests simulating the plant practice.

stainless steel mill/media generates relatively oxidising conditions (Table 1A) which promotes the formation of hydrophobic surface species. Based on a previous work by Allison *et al* (1972), the dominant hydrophobic species for pyrrhotite flotability is assumed to be dixanthogen (X₂) which is an oxidation product of xanthate (X⁻) and forms according to the following reaction:



Accompanying this oxidation half reaction is the reduction of dissolved oxygen adsorbed



on sulphide minerals. The formation of dixanthogen may be preceded by that of ferric xanthate and hydroxy xanthates which is believed to be responsible for the initial hydrophobisation of iron sulphide minerals (Wang, Forssberg and Bolin, 1989). Conditions that lead to the formation of dixanthogen in polysulphide slurries usually produce a bulk flotation concentrate showing poor selectivity.

Under oxygen-deficient conditions (the SS/N₂ case, Table 1B), the formation of hydrophobic surface species on pyrrhotite are restricted or at least retarded because of the impact on the kinetics of these surface reactions. The apparent redox potential obtained under these conditions (eg -55 mV, SHE) is lower than the reversible potential for the formation of dixanthogen. A comparison of all three sulphide recoveries obtained in a typical laboratory primary rougher flotation is given in Table 2. Tabulated values are the averaged recoveries from two tests. It is seen that pentlandite recoveries are practically the same in both cases. However, the recovery of chalcopyrite, like that of pyrrhotite, is substantially lower under oxygen-deficient conditions.

TABLE 2

Comparison of pentlandite, chalcopyrite and pyrrhotite recoveries (primary rougher stage).

	Pn	Cp	Po
Stainless steel/ nitrogen	60	26	10
Stainless steel/ air	62	77	26

There is a striking similarity between the shape of recovery curves obtained in plant practice (Figure 2) and laboratory tests (Figure 3) with the mild steel grinding media in both cases. Considering the laboratory test results, one may note that in the primary rougher stage the pyrrhotite recovery observed in the mild steel grinding-air flotation case is practically the same as in the stainless steel grinding-nitrogen flotation case. It is evident that the mild steel grinding media retards the surface oxidation of sulphide minerals which is essential for hydrophobic layer formation. Upon galvanic contact between the grinding media and the sulphide mineral the latter will act as a cathode while the former as the anode. Such a contact was shown to reduce the rest potential of pyrrhotite to a level close to that of iron (Rao, Moon and Leja, 1976). Such a situation may even inhibit the adsorption of xanthate on sulphide minerals. In the laboratory, pulp potentials as low as -300 to -500 mV (SHE) are observable after grinding with mild steel mill/media. The dissolved oxygen level in these pulps is usually as low as 1 ppm because of preferential consumption by the media. Thus, the corrosion of the grinding media will play a major role in determining the kinetics of flotation, especially in the early stages of flotation (eg primary rougher stage).

The above considerations apply also to chalcopyrite. The electrochemical aspects of chalcopyrite flotation are the most published of any mineral (eg Heyes and Trahar, 1977; Trahar, 1984; Luttrell and Yoon, 1984; Pang and Chander, 1990). Recently, it has been suggested that chalcopyrite can undergo a cathodic decomposition under oxygen-free conditions. The reaction proposed by Li and Iwasaki (1992) is as follows:



The iron hydroxide thus formed, may be one of the surface species explaining the poor flotability. However, a common observation is that chalcopyrite regains its relatively strong hydrophobicity upon sufficient aeration of the pulp.

Note that the order of flotability under oxygen-deficient conditions becomes:

Pentlandite > Chalcopyrite > Pyrrhotite

This is in the same order as the catalytic activity for oxygen reduction on these minerals (Rand, 1977). The relative values of

TABLE 3
Summary of experimental variables as a function of residence time.

Elapsed Time (hr)	Redox Potential (mV, SHE)		Dissolved O ₂ (saturation)		pH	Temp (°C)
	tank	float cell	tank	float cell		
0.3	-70	-50	21.7	19.6	9.5	16.7
3.5	+38	+13	11.9	14.1	9.1	23.6
4.0	-	-	7.2	14.3	9.0	-
6.0	+67	+68	6.0	8.3	8.9	24.2
6.5	+78	+78	-	7.1	8.8	24.2

Tafel slopes associated with the oxygen reduction on these minerals have been confirmed by Hodgson and Agar (1988a). There is a significant difference between the catalytic activity of pentlandite and those of pyrrhotite and chalcopyrite. This is offered as an explanation for the origin of difference in the flotation kinetics and selectivity observed under these conditions. Martin *et al* (1989) observed that pyrite in complex sulphides can float preferentially using nitrogen. The explanation noted above also applies to the pyrite case. Owing to its high catalytic activity, pentlandite, like pyrite, can develop hydrophobicity even under oxygen-deficient conditions and float at relatively low potentials under which dixanthogen formation is not very likely. More details on these features of complex sulphide flotation will be presented elsewhere.

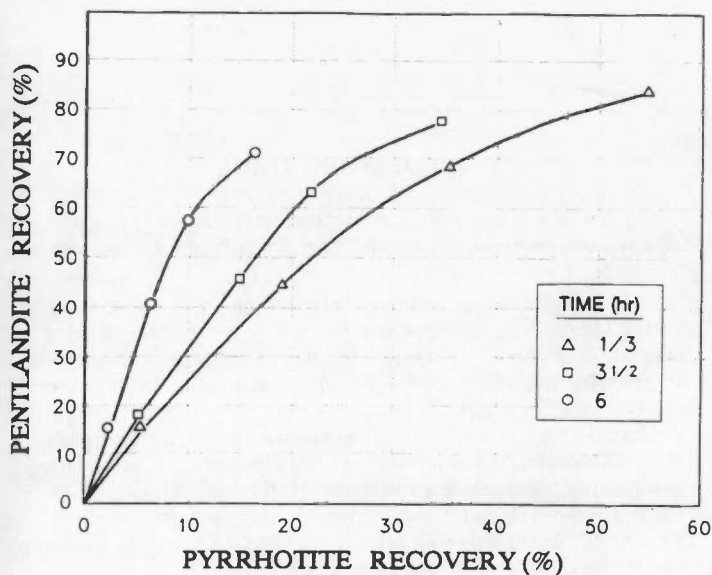


FIG 4 - Variation of flotation selectivity between pentlandite and pyrrhotite with respect to oxidation time.

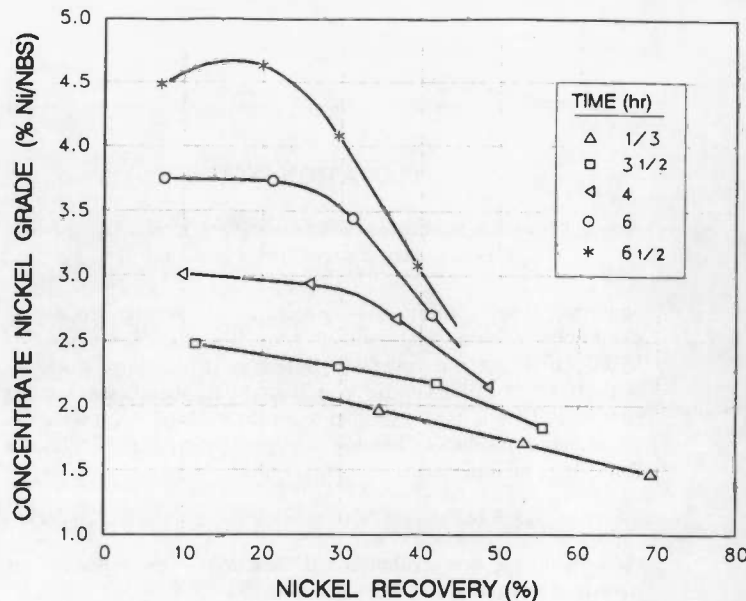


FIG 6 - Nickel grade (as per cent Ni in nickel bearing sulphides)/ recovery relation for various oxidation periods.

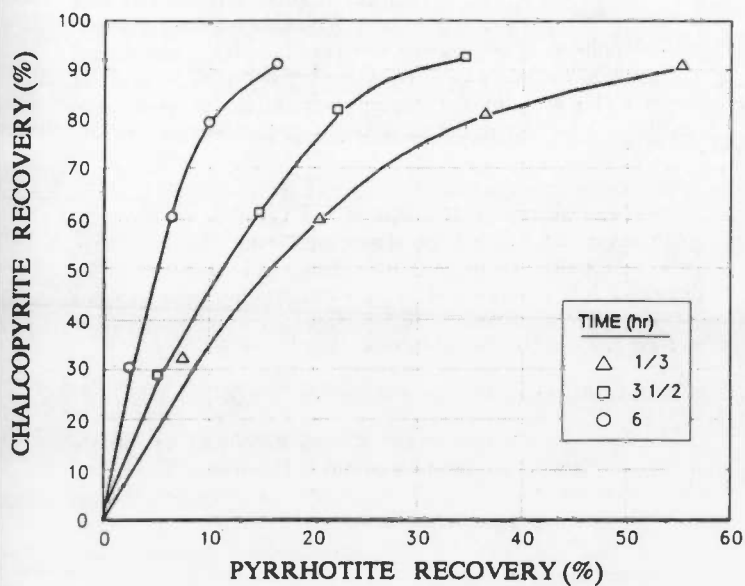


FIG 5 - Variation of flotation selectivity between chalcopyrite and pyrrhotite with respect to oxidation time.

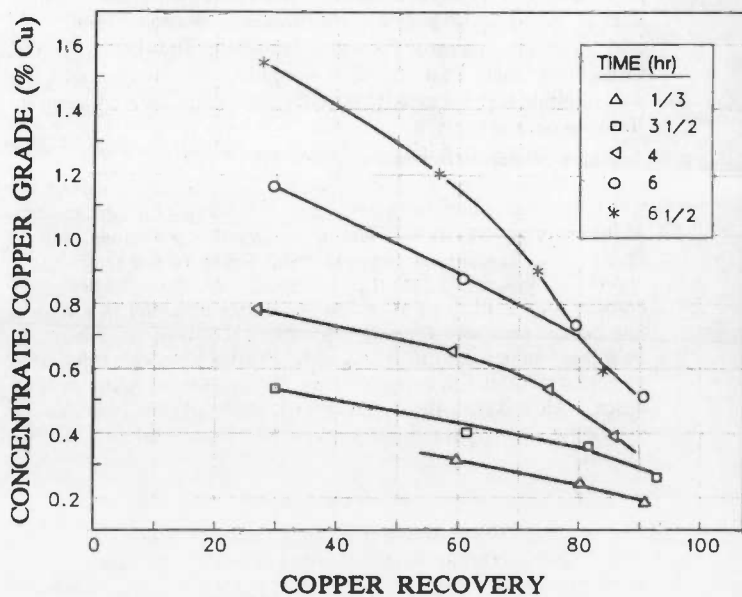


FIG 7 - Copper grade/ recovery relation for various oxidation periods.

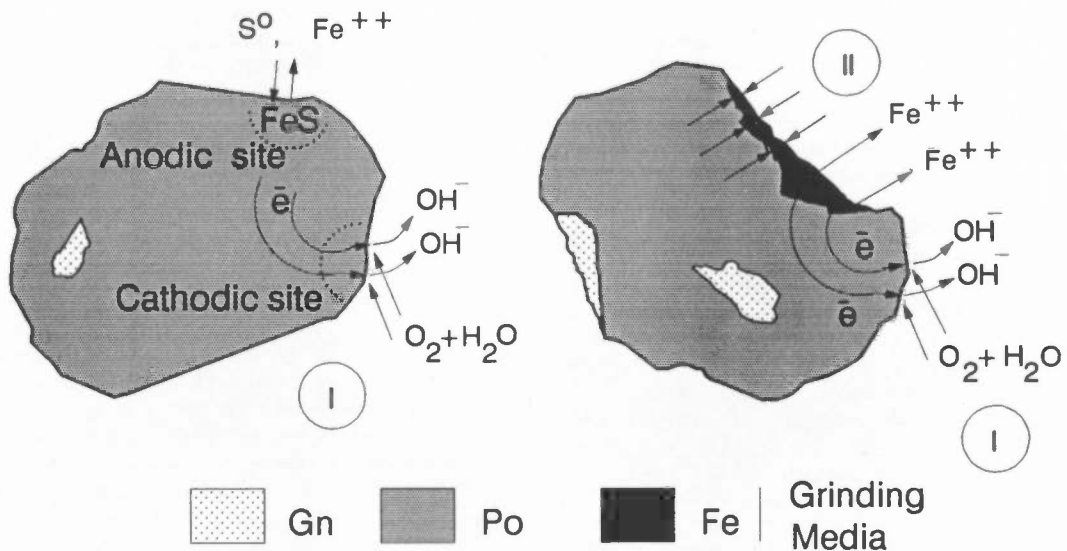


FIG 8 - Electrochemical (I) and magnetic (II) interactions on pyrrhotite.

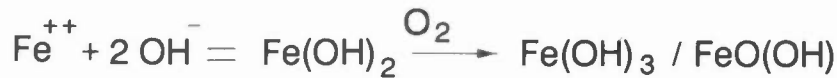
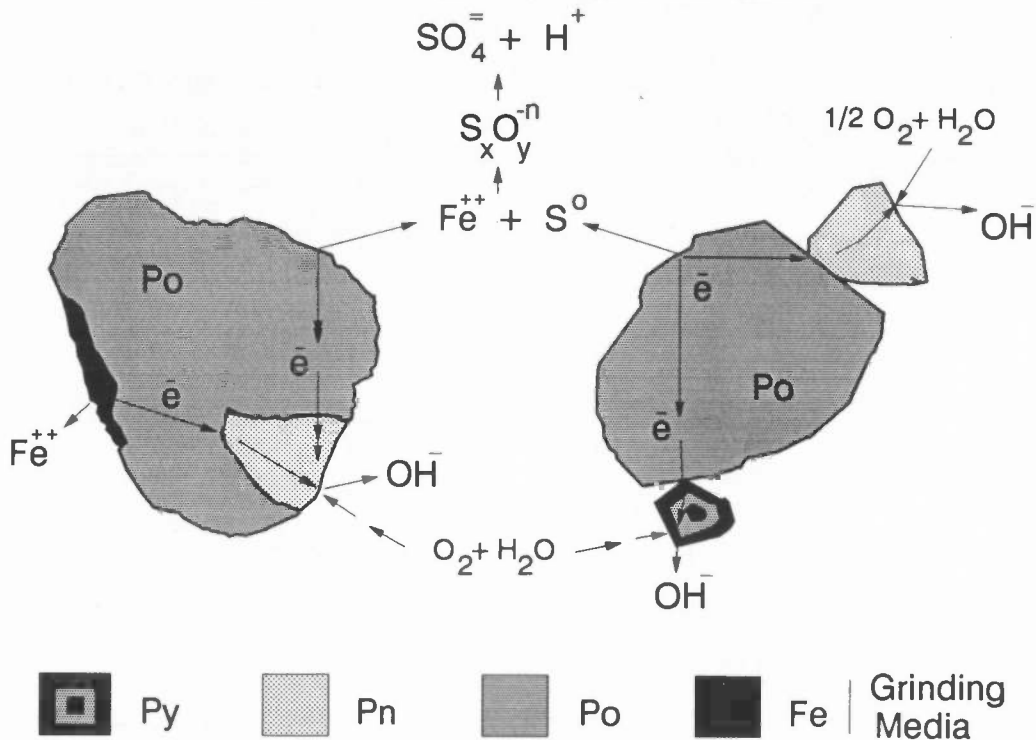


FIG 9 - Electrochemical interactions and the surface reactions involved in preferential oxidation of pyrrhotite and grinding media.

Behaviour of reground process magnetics

Test conditions. The stream tested consists mainly of monoclinic Po (~80 per cent), the remaining fraction being mostly gangue in addition to Pn (~1.2 per cent) and some Cp (~0.3 per cent). Prior to regrinding, almost all of Pn is considered to be locked with Po.

The image analysis indicated the presence of significant amount of pyrite in the magnetics flotation feed.

The relationship between the test variables is summarised in Table 3 as a function of pulp's residence time in the stirred head tank.

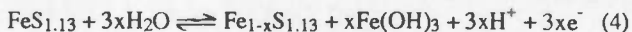
Both the redox potential and temperature increase with increasing agitation time. Simultaneously, a reduction in the dissolved oxygen content and pH of slurry is apparent.

Relative flotabilities. The flotability of pentlandite versus that of pyrrhotite is shown in Figure 4. For the sake of clarity, only three sets of data are plotted here. There is an apparent shift in the curves towards higher pentlandite recoveries demonstrating the increase in flotation selectivity with increasing agitation time. These results suggest that pyrrhotite particles undergo a selective oxidation which renders this mineral relatively more hydrophilic. The oxidation period for nearly six hours (ie with no deliberate aeration) accounts for about 70 per cent decrease in pyrrhotite

recovery. Figure 5 shows the same form of plot for chalcopyrite and pyrrhotite recoveries. The same trends are notable in relation to the effect of oxidation on chalcopyrite-pyrrhotite selectivity.

Grade and recoveries. Increases in concentrate grade as a result of Po-Pn separation are shown with respect to the recoveries of nickel in Figure 6. The nickel grade in this figure is expressed in terms of percentage Ni in nickel bearing sulphides, NBS (ie $100 \cdot [\text{Ni per cent}] / [\text{Pn per cent} + \text{Po per cent}]$). The longer the oxidation period the more efficient the separation of Pn from Po and the higher the nickel grade in the concentrate. The data plotted in Figure 7 indicate a similar copper grade-recovery improvement with longer periods of oxidation.

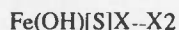
Mechanism of selectivity. Pyrrhotite requires some initial oxidation in order to exhibit significant flotability, as the surface reactions leading to its hydrophobisation are more favourable at relatively high redox potentials. However, beyond a certain degree of oxidation, changes occur in the chemistry of the pulp. Some of the changes affecting the selectivity in flotation include degradation of xanthate into a variety of products such as dixanthogen, perxanthate, etc. Sulphide minerals produce surface species such as elemental sulphur (or an incipient metal-deficient sulphide), a variety of oxy-sulphides (S_xO_y)ⁿ, metal hydroxides such as $\text{Fe}(\text{OH})_3$. Buckley, Hamilton and Woods (1988) proposed the following oxidation reaction on pyrrhotite and emphasised the formation of iron-deficient sulphide.



The kinetics of surface oxidation on sulphide minerals may be accelerated by a set of galvanic interactions (Majima, 1969; Kocabag and Smith, 1985). This type of interaction has been the subject of a number of investigations which involved pyrrhotite as one of the principal minerals (Pavlica and Iwasaki, 1982; Nakazawa and Iwasaki, 1985; Cheng and Iwasaki, 1992). As noted previously, the catalytic activity of pentlandite for oxygen reduction is greater than that of both chalcopyrite and pyrrhotite. On the other hand, pyrite, which exists in the same sulphide system, is more cathodic than the previous three (Rand, 1977). In view of these electroactivity differences, some galvanic interactions are expected to take place among these sulphide minerals. Pn will tend to accelerate the anodic reactions on Po while itself hosting the oxygen reduction reaction. The effect of Py on Po will be similar in nature. At earlier stages (eg roughers), this type of interaction may help in the hydrophobisation of Po during the anodic processes such as:

1. generation of surface site for xanthate adsorption,
2. generation of S^0 through a metal-deficient surface and
3. oxidation of xanthate to dixanthogen.

However, with an increasing period of Pn-Po (and Py-Po) contact under the influence of oxygen, the hydrophobic/hydrophilic balance on Po surfaces will shift in favour of hydrophilic surface products. The transition to hydrophilicity will be facilitated especially with the lack of xanthate in the slurry. Because the Po-rich feed under study was previously floated (ie part of scavenger concentrate) the Po is partially hydrophobic after regrinding. The hydrophobic surface product on Po would consist of:



(Hodgson and Agar, 1988b). Here, X and X₂ represents xanthate and dixanthogen respectively, the latter being physically adsorbed on the surface. Once the Po in slurry is free of the galvanic influence of the grinding media it will be the main target for oxidation which would be catalysed by the more cathodic minerals in galvanic action.

As a result of such interactions, the Po particles will experience a selective oxidation with increasingly stable hydrophilic surface

species gradually dominating its surface. Pentlandite surfaces, on the other hand, would be expected to retain more of their original surface chemistry having been in a locked state prior to regrinding and being cathodically protected by preferential anodic reactions on Po (and metallic iron from media after grinding). Thus, the difference in the surface states of Po and Pn constitutes the origin of observed selectivity in these tests. Reactions postulated by Pavlica and Iwasaki (1982) are applicable for the proposed mechanism which is illustrated in Figures 8 and 9. The former shows the fundamental electrochemical reactions (schematic, not balanced) on a liberated pyrrhotite particle and also the magnetic/electrochemical influence of a residual metallic iron from grinding media. The latter shows the reactions likely to be taking place as a result of the Pn-Po and Py-Po contact. The ferrous hydroxide that initially forms will subsequently be converted to more stable ferric hydroxide and/or oxy-hydroxide form consolidating the hydrophilic nature of Po.

Note that other sulphide minerals in the pulp can experience surface oxidation despite their relative nobility. Oxidation can and does cause a reduction in the flotability of Pn and Cp as these minerals may also be rendered hydrophilic by iron hydroxide formation under extended agitation, especially in the presence of pyrite.

SUMMARY AND CONCLUSIONS

Oxidation taking place during the processing of nickel-copper ores influences the flotation selectivity. This effect could produce different results depending on factors that include the type of grinding media, reagent dosage, flotation stage and mineralogy and susceptibility to natural activation. In general, moderate oxidation of the freshly ground ore promotes the flotability of all principal sulphide minerals in the ore leading to a bulk flotation concentrate. In laboratory studies, favourably low redox conditions prevail during the initial period of flotation. As a consequence, pyrrhotite/pentlandite selectivity is limited to the early stages of flotation following a grinding operation. As the flotation progresses, pyrrhotite develops more and more hydrophobic sites; it becomes more floatable or relatively more difficult to depress. The ease of flotability under normal aeration conditions, which is $\text{Cp} > \text{Pn} > \text{Po}$, changes to $\text{Pn} > \text{Cp} > \text{Po}$ under oxygen-deficient conditions. This behaviour is correlated with the catalytic activity of these minerals for oxygen reduction.

The selectivity in the flotation of magnetics may improve as a result of a preferential oxidation of pyrrhotite. The observed flotation selectivity has been discussed in the light of fundamental electrochemical interactions involving pyrrhotite, pentlandite, chalcopyrite, pyrite and metallic iron from grinding media. A mechanism was proposed on the basis of electrochemical reactions postulated by Iwasaki *et al.*

The two flotation behaviours discussed in this paper with emphasis on the flotability of pyrrhotite, may be rationalised within the context of redox potential dependency of the sulphide flotation; increased activation and flotation with increasing potential (within the range of moderate oxidation) followed by a suppression of flotation again with increasing potential (excessive, beyond moderation). Examples of such behaviour have been seen for a variety of sulphide minerals (Trahar, 1984).

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