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A ToF-SIMS investigation on correlation between grinding environments and sphalerite surface chemistry: Implications for mineral selectivity in flotation

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Changes in mineral surface properties during grinding play a key role in flotation performance. ToF-SIMS surface chemical analytical studies have shown that flotation separation of sphalerite from chalcopyrite is significantly affected by the oxidation of metal species on the surface of sphalerite. The intensity of iron oxyhydroxyl species on the surface of sphalerite has a positive correlation with poor recovery of sphalerite. Given the link between the presence of oxide species on the surface of sphalerite and a lower recovery during Cu flotation, a laboratory study was initiated to evaluate the potential for sphalerite surface oxidation control and improve recovery through grinding. For the investigation, a ball mill which allowed for monitoring pulp chemistry during grinding was utilized to study the impact of grinding conditions on selective flotation of sphalerite. ToF-SIMS was used to identify and measure the variability in sphalerite surface species as a result of the different test parameters. Variable mill parameters include 2 types of grinding media, aeration conditions, addition of $FeSO_4$, and altering the pyrite content in the feed ore. TOF-SIMS analyses of mill discharge samples identified higher intensities of iron oxyhydroxyl species on sphalerite surface subsequent to grinding with mild steel balls, in condition of aeration, use of $FeSO_4$, and by increasing the pyrite content of the feed ore. The higher adsorption of iron oxyhydroxyl species on the surface of sphalerite should be consistent with the lower recovery. To verify this, bench scale flotation tests in the presence and absence of $FeSO_4$ were performed; results correlated iron oxyhydroxyl species with the poor sphalerite recovery.

KEYWORDS
flotation, grinding, sphalerite, ToF-SIMS

1 | INTRODUCTION

Copper is typically extracted from oxide and sulfide ores that only contain 0.5% to 2.0% copper. The refining technology employed by copper producers depends on the ore type, as well as other economic and environmental factors. Currently, approximately 80% of primary copper production comes from sulfide ores, but sulphidic copper ores are too dilute to be suitable for direct smelting and require enrichment. In a poly-metallic sulfide mineral processing plant, copper ores are milled then concentrated by physical separation, generally flotation, to give concentrates.[1] The enrichment process usually involves 2 or more stages. In the flotation process, the recovery of minerals is accomplished by the attachment of a hydrophobic mineral to bubbles introduced to the flotation chamber. The net result is to produce a concentrate of the particular value added mineral of interest and a tailings consisting of all other phases. In order to facilitate the recovery of a desired mineral, various reagents are introduced to induce selective hydrophobic surface development on the mineral of interest. For a Cu/Zn ore, consisting for example of chalcopyrite ($CuFeS_2$) and sphalerite (ZnS), the first stage involves selective chalcopyrite flotation and sphalerite rejection to produce marketable copper concentrate. In the second stage, the reject sphalerite is recovered by flotation to produce a Zn concentrate. In the case of chalcopyrite flotation, this is simply accomplished by the addition of surfactants (collectors) which attach to the mineral surface promoting hydrophobicity and enhance bubble mineral attachment. The flotation of sphalerite on the other hand is somewhat more challenging as there is a requirement of surface activation in order to promote collector attachment. The activation of sphalerite is generally accomplished by the adsorption and attachment of Cu; in sphalerite recovery, $CuSO_4$ is typically added to the flotation process to induce activation, collector attachment, and recovery. For Cu/Zn ores, inadvertent activation of sphalerite by dissolved copper ions results in sphalerite reporting to the Cu concentrate which ultimately represents an overall loss of Zn in the recovery process. Inadvertent activation of the sphalerite in the flotation of complex sulfide ores has been reported in many instances and requires a more complex metallurgical process to effectively separate the various mineral phases in the ore. For a detailed discussion regarding the activation of sphalerite, the authors are referred to the excellent review article by Chandra and Gerson.[2]

The following brief review on the effect of grinding media on mineral selectivity in flotation is by no means exhaustive. The presented materials were selected to illustrate the link between grinding media and improved flotation response. The authors encourage those whom are interested in this link to pursue the vast proportion of information available in the literature. An early review on the effect of grinding media on sulphide flotation by Martin et al[3] concluded that the type of media can indeed have an important effect on flotation and mineral selectivity. They identified that the most likely conditions to be effected by media type and hence flotation response are dissolved oxygen content, pulp potential, and galvanic interactions. Ye et al[4] showed that the surface chemistry of minerals, which impact optimum mineral separation, may change significantly during grinding or regrinding depending on the type of mill and grinding media used. Bruckard et al[5] and Rabieh et al[6] separately reviewed the effects of grinding environments on the flotation of copper sulfides and pyrite. Kinal et al[7] studying the effect of grinding media on zinc depression in a lead cleaner circuit identified that a change in chrome grinding media produced a significant impact on galena/sphalerite selectivity. They report that the improvements in sphalerite depression are directly related to changes in pulp chemistry. The study illustrates that the strong link between grinding media, mineralogy, and the resultant pulp chemistry significantly affects metallurgical performance and therefore necessitates evaluation on a site by site basis.

In a previous study on ores from Mine Matagami (Une Compagnie Glencore),[8] we reported that a difference of grinding regimes in the plant and in the laboratory is responsible for a significant difference in Zn recovery. Research by site metallurgists found that sphalerite was being activated during Cu/Zn flotation laboratory tests; however, when the same ore was tested in the plant, the activation was significantly reduced. At Surface Science Western, time of flight secondary mass spectrometry (ToF-SIMS) was used to analyze sphalerite surfaces from the 2 testing programs. The analyses identified that the variation of surface species on sphalerite was linked to the discrepancy in Zn recovery between the laboratory tests and flotation plant samples. Two important observations were found. First, there is considerably more hydrophilic iron oxyhydroxide on sphalerite surfaces from the plant samples relative to those from the lab. Secondly, comparing the surface chemistry on sphalerite and chalcopyrite from the laboratory and mill samples, the relative proportion of hydrophilic iron oxyhydroxide species is significantly greater on the surface of sphalerite grains relative to chalcopyrite. These results indicate that grinding in the plant produced a sufficient proportion of iron oxidation species resulting in sphalerite depression but apparently did not have a significant effect on chalcopyrite. This study illustrates that, as the grinding strategies were different in the 2 programs described earlier, there is a link between grinding conditions and the sphalerite flotation behavior. Optimization of grinding conditions thus has the potential to control the proportion of iron oxidation species on the surface of sphalerite and affect its flotation.

This paper evaluates various grinding strategies in order to optimize the selective depression of sphalerite during the Cu flotation stage in Cu/Zn flotation recovery process. The testing strategy involves grinding with 2 types of grinding media, by introducing aeration during milling, the addition of $FeSO_4$, and increasing the pyrite content in the feed ore. A ball mill that allows the pulp chemistry to be monitored during grinding was utilized to link the pulp chemistry to the selective depression of sphalerite. ToF-SIMS was used to identify differences in surface species occurring as a result of the different grinding parameters.

2 | MATERIALS AND METHODS

The sample ore was obtained from the Matagami mines (Glencore Canada Inc). It was crushed to 1.7 mm, homogenized, split, sealed, and frozen prior to use. Grinding was performed in a horizontal, cylindrical, rubber-lined laboratory ball mill, which has probes mounted in the grinding chamber to monitor pulp chemistry changes (pH, Eh, DO, and conductivity) during grinding. The grinding pulp density is 25%. Grinding time is 40 minutes to have a mill discharge of 80% passing 75 microns. After grinding, the pulp sample was immediately purged of oxygen using argon and frozen in liquid nitrogen.

Mineral surface chemistry analysis by ToF-SIMS was performed on sphalerite from the mill discharge. To analyze the outermost layer of samples, an ION-TOF, TOF SIMS IV™ secondary ion mass spectrometer was used. This technique allows for the analysis of the outermost 1 to 3 atomic layers of a surface by mass spectrometry. Each sample was mounted on indium foil substrate, introduced into the instrument, pumped down in the vacuum, and analyzed. From 6 regions on each sample, a minimum of 6 grains of each mineralogical type were examined providing a minimum of 36 grain analyses. This analysis provides a comprehensive survey of the surface species on the mineral grains in the various samples.[9]

The intensity of selected species detected on sphalerite surfaces as positive or negative ions is plotted in vertical box plots and illustrates relative changes in surface specie abundance for the mineral grain examined in the sample. In the vertical box plots, the median is plotted as the solid line across the box, whereas the mean is plotted as the dashed line (Figure 1). All TOF-SIMS data presented (counts) are normalized by the total ion intensity (counts of the recorded total mass spectrum) for the region of interest.

3 | RESULTS AND DISCUSSION

In order to evaluate and potentially identify opportunities to optimize the selective separation of sphalerite from chalcopyrite by flotation, 4 different testing strategies focused on pulp chemistry manipulation during milling were performed. The results of the testing programs are given later.

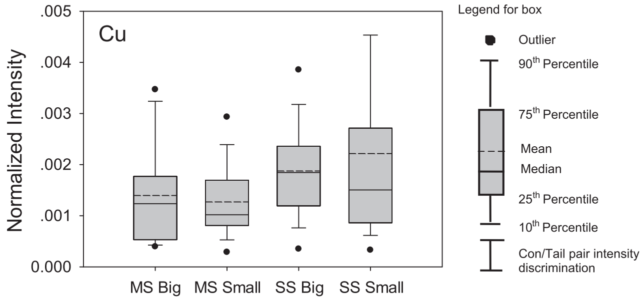

FIGURE 1 An illustration of a box plot showing the various components that are discussed throughout the test

3.1 | Two types of grinding media

In the laboratory ball mill, 200 g of homogenized sample was ground to 80% passing 75 microns with either mild steel or stainless steel balls. Balls used for the tests are both mild steel and stainless steel of 2 sizes: the big (1 inch) and the small (1/2 inch). For each test, regardless of size or composition, the weights were kept constant in order to produce the same grinding efficiency. Table 1 shows the experiments performed and the grinding media parameters.

The grinding discharge from each test was analyzed by ToF-SIMS to examine the sphalerite surface chemistry (Figures 2 and 3).The surface evaluation of sphalerite grains shows that Cu is significantly enriched on the surface of sphalerite when using stainless steel balls relative to the mild steel balls (Figure 2). Furthermore, Figure 3 reveals a higher degree of Fe, FeO, FeOH, and FeOOH on the sphalerite surface for the tests with mild steel balls relative to stainless steel balls. Galvanic interactions occurring during wet grinding of sulfide ores cause the dissolution of iron from the grinding media which leads to the formation of iron hydroxides. Electrons are transferred from grinding media to pyrite (0.66 V) which is the nobler in comparison to chalcopyrite (0.56 V, anomalous) and sphalerite (0.46 V). Rest potential of stainless steel (high Cr) is 0.206 V in DI water and 0.088 V in 0.5 M NaCl; correspondingly, the rest potential of mild steel is −0.336 and −0.396 V. The difference between rest potential of the grinding media and pyrite is more pronounced if mild steel used. Consequently, the oxidation of iron from mild steel would be much greater than from stainless steel. With sphalerite as the anodic mineral, its surface oxidation increases when the galvanic interaction becomes greater. The surface chemistry indicates that Cu ions from the dissolution of chalcopyrite attach to the surface of sphalerite grains during the milling. The testing identified a link between surface chemistry and the pulp chemistry in the mill environment. Mild steel balls readily

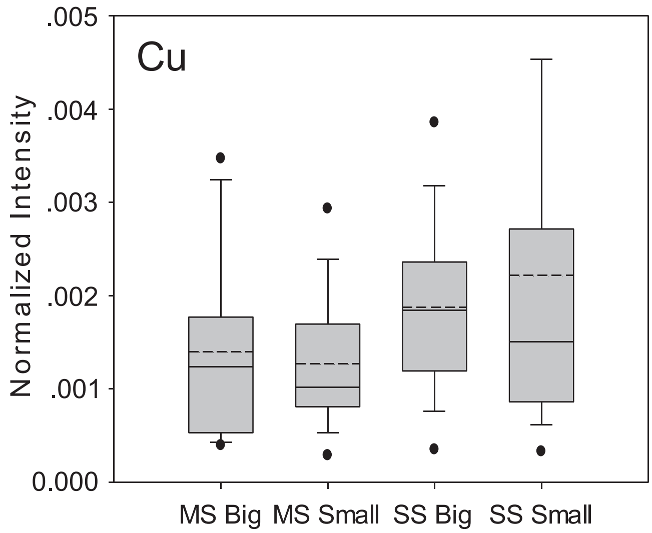

FIGURE 2 Normalized intensity of Cu on the surface of sphalerite as measured by TOF-SIMS. Abbreviations are given in Table 1

supply ferrous ions to the pulp during grinding, increasing the opportunity to generate a higher proportion of iron oxyhydroxyl species on sphalerite surfaces. The effect in the mill is that the generation of metal (Fe) oxides contributes to the rapid consumption of oxygen for milling with the mild steel balls relative to stainless steel balls.[8] As previously reported, other pulp chemistry parameters measured during grinding also indicated a less oxidizing environment when milling with mild steel balls and were reflected in lower pulp pH, ORP, and slurry conductivity.[10]

In the tests with different size of grinding media, the pulp chemistry details indicated that grinding with smaller balls (larger surface area) compared with bigger balls increased oxygen consumption. The ToF-SIMS surface evaluation of sphalerite unfortunately did not reveal any conclusive consistent species discrimination between the tests. In conclusion, grinding with mild steel balls in comparison to stainless steel balls, iron oxide species development on sphalerite surfaces may cover or compete for copper adsorption. In this fashion, the activation of sphalerite by copper is substantially reduced by the production or adsorption of iron oxide species.

3.2 | The action of aeration

Aeration of pulp in complex sulphide ores processing affects the flotation behavior of sulphide minerals as well as their selectivity.[11,12] Therefore, aeration tanks are used at many flotation plants after grinding in order to improve the selectivity between sulphide minerals.[13] From our lab mill tests, it was observed that oxygen consumption during grinding with mild steel balls is greater than with stainless steel

TABLE 1 Experiments performed using different types of grinding media

Testing no.	Labeled as	Grinding medium	Cr, %	Size of the balls	Total surface area of the grinding medium, cm^2
Test 1	MS big	Mild steel	0	Big (1 inch)	5837
Test 2	MS small	Mild steel	0	Small (0.5 inch)	11 644
Test3	SS big	Stainless steel	18.44	Big (1 inch)	5837
Test4	SS small	Stainless steel	18.44	Small (0.5 inch)	11 644

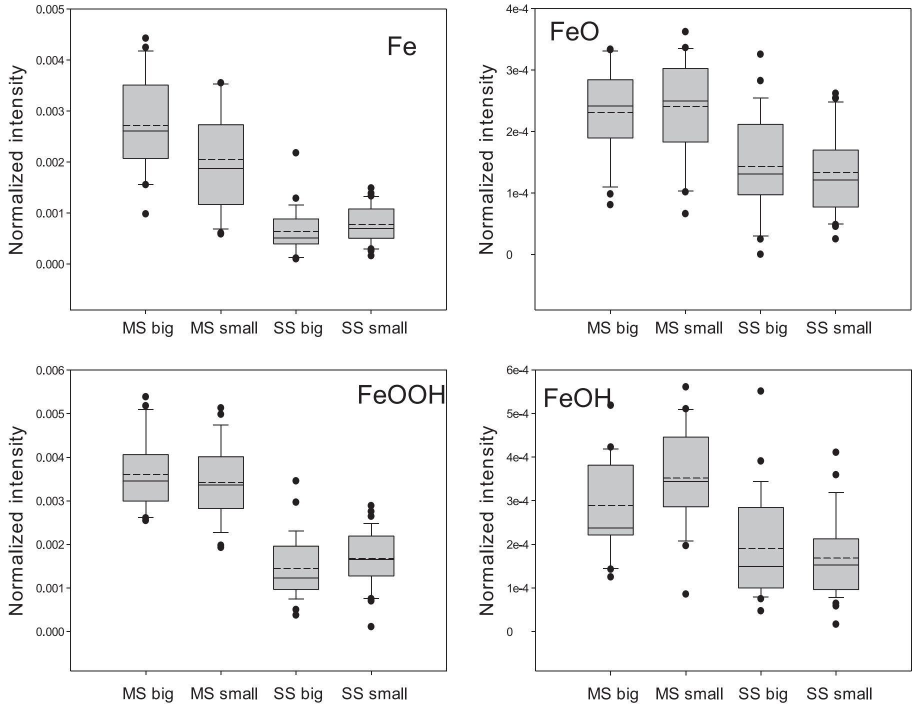

FIGURE 3 Normalized intensity of Fe and iron oxide and oxyhydroxide species on sphalerite surfaces as measured by TOF-SIMS. Abbreviations are given in Table 1

balls. Surface analyses of sphalerite showed a higher relative proportion of Fe, FeO, FeOH, and FeOOH for tests with mild steel balls and appear to be linked to rapid oxygen consumption in the mill. The significance here is that adjusting oxygenation of the pulp can better control the iron oxyhydroxyl species generated on the surface of sphalerite. In the following tests, the effects of aeration during grinding on sphalerite surface chemistry were studied. Milling tests were conducted with mild steel grinding balls and run at air sparged conditions of 1 mL/second.

An increase of oxygen in the pulp results in a reduction in the rate of oxygen consumption leading to a more oxidizing environment as shown the DO and ORP (Figure 4). Dissolved oxygen content is obviously higher with air sparging in comparison to the closed system baseline test. As a result of the electrochemical reaction of oxygen in the

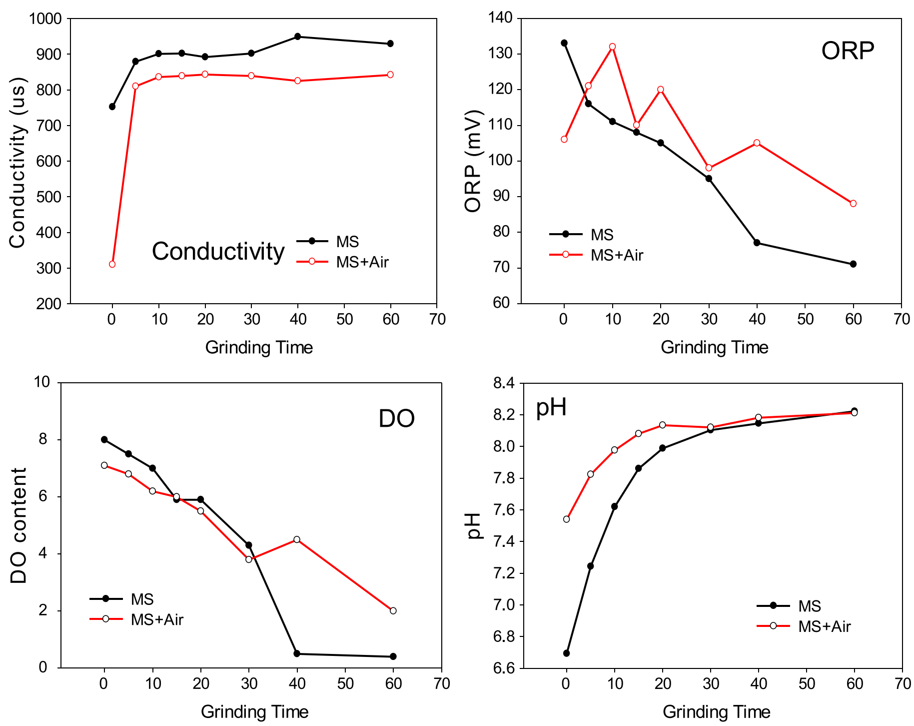

FIGURE 4 Pulp chemistry variations during grinding with air sparged ball mill (MS + Air) and without air sparging (MS)

pulp, an increase in the production of oxyhydroxyl ions results in an initial pH increase and slurry conductivity decrease. The later potentially reflecting a decrease in free Fe species in the pulp and the generation of iron oxyhydroxyl species as identified on the surface of sphalerite grains by ToF-SIMS (Figure 5).

3.3 | Addition of iron sulfate

Chemicals used in froth flotation are added to regulate pH, to control the surface charge on minerals, to complex ions, and to enhance or prevent collector adsorption. Reagents that are believed to be associated with increasing the iron oxyhydroxyl generation would be able to enhance the development of FeOH hydrophilic coating on sphalerite resulting in its depression. This testing program was designed to evaluate the addition of $FeSO_4$ (600 g/t) during grinding. The tests were performed using stainless steel balls with and without the addition of $FeSO_4$. The surface analyses of sphalerite from the mill discharge samples reveal a higher relative proportion of FeO, FeOH, and FeOOH on the sphalerite surfaces in response to $FeSO_4$ addition (Figure 6). This is correlated with, a much lower surface copper content for the iron sulfate addition tests as compared to the tests with stainless steel balls alone (Figure 6). The implication here, that copper may be inhibited from attaching to the surface of the sphalerite grains as a result of $FeSO_4$ addition, can result in enhanced depression of sphalerite and lower recovery in a Cu concentrate.

Bench scale flotation tests were performed at Mine Matagami to evaluate the effect of iron sulfate on the flotation behavior of sphalerite. Metallurgists employed mild steel balls as grinding media and kept the solids content at 36%. After 45-minute grinding, P80 of this ore was 45 microns; 1300 g/t lime and 600 g/t $FeSO_4$ if required were added prior to grinding and 25 g/t 3418A as collector was used for ball mill discharge. Flotation data in Figure 7 show significant variation in

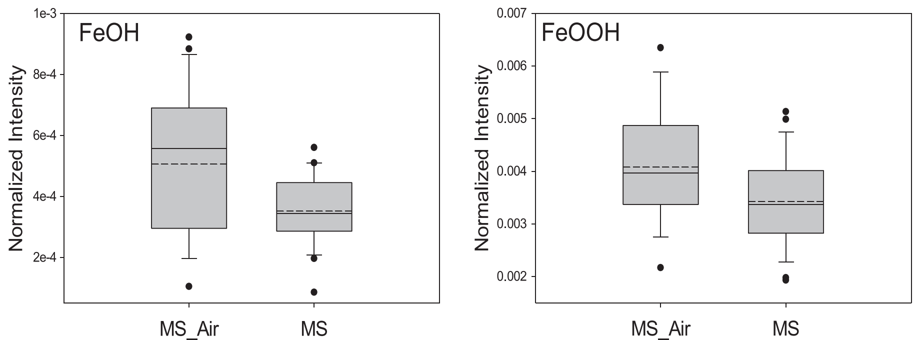

FIGURE 5 Normalized intensity of iron oxyhydroxyl species on sphalerite surfaces in grinding with air sparged ball mill (MS + Air) and without air sparging (MS)

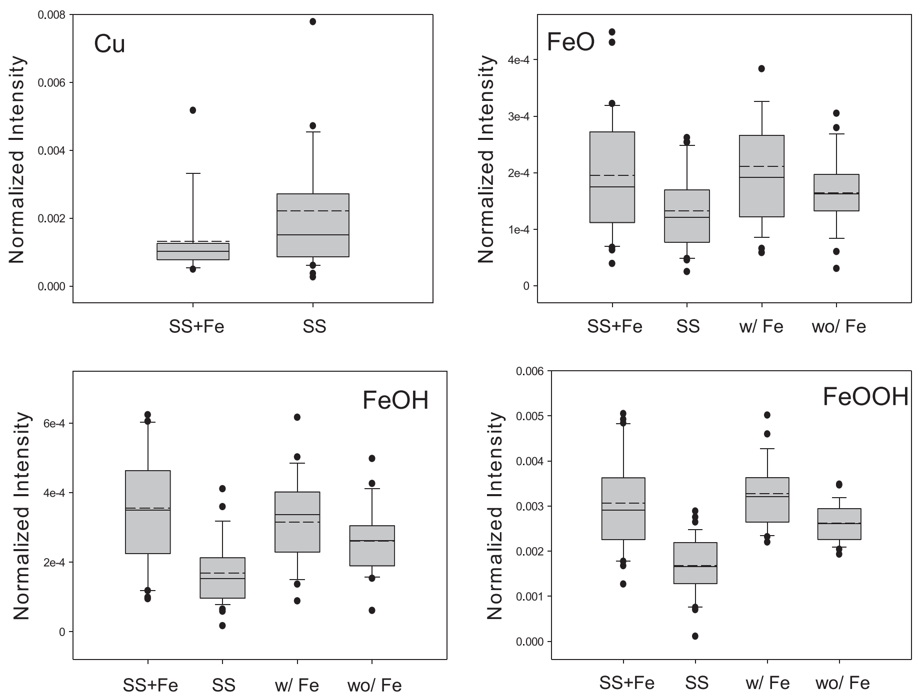

FIGURE 6 Normalized intensity of surface copper and iron oxyhydroxyl species on sphalerite surfaces during grinding with (shown in the picture as SS + Fe) and without (SS) the addition of $FeSO_4$. Also shown are normalized intensity of iron oxyhydroxyl species on sphalerite surfaces from bench scale testing at the plant; tests with $FeSO_4$ (W/Fe) and without $FeSO_4$ (WO/Fe). All tests were performed at addition rates of 600 g/t of $FeSO_4$

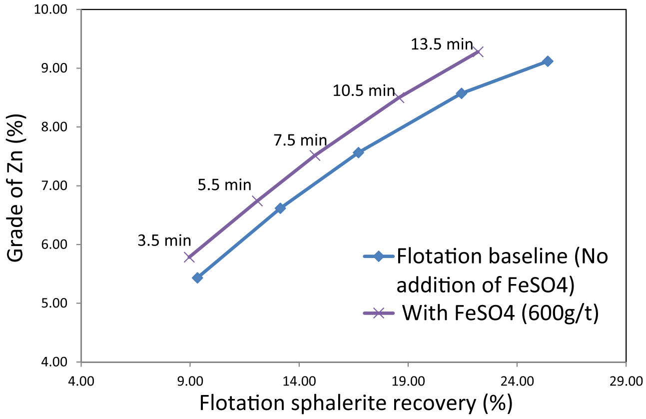

FIGURE 7 Bench scale flotation of sulphide ore with and without 600 g/t FeSO4

Zn recovery to the rougher Cu concentrates. After continuously floating for 10.5 minutes, Zn grade in the Cu concentrate is similar from the bench flotation either with or without $FeSO_4$, whereas the sphalerite recovery decrease from 21.45 (% w/w) to 8.58 (% w/w) owing to addition of $FeSO_4$. A pair of ball mill discharge samples (1 with no addition of $FeSO_4$ and a second with addition of iron sulfate) was sent to SSW to study sphalerite surface chemistry variation. The data show higher iron oxyhydroxyl species and lower surface copper on sphalerite in response to $FeSO_4$ addition. The tests results are in agreement to the lab tests performed at SSW.

3.4 | Increase pyrite content in feed ore

Owusu et al[14] reported that copper recovery was affected by the proportion and type of pyrite in the ore; both the chalcopyrite grade and recovery were reduced with higher pyrite content in the mixture. The authors[14] attributed the decrease in copper recovery to galvanic interactions occurring between chalcopyrite and pyrite during grinding prior to flotation which accelerates oxidation of chalcopyrite and also of pyrite. Pulp chemistry studies also reported that DO content and oxidation potential (ORP) decreased with increasing pyrite content in the mixture. The implication here is that the increased pyrite content in the feed ore results in an increase in oxygen consumption and the development of a less oxidizing environment. The flotation response however suggests that the depression due to the formation of hydrophilic surface coatings of iron hydroxide species on various mineral phases inhibiting sulphide flotation must then have been fairly rapid.

In the Cu/Zn separation case, the increased pyrite content benefits iron oxyhydroxyl species generation and may further depress the flotation of sphalerite. In this test program, a 200-g sample consisting of a mixture containing 180 g of Matagami ore and additional 20 g of pyrite was ground with stainless steel balls. The tests reveal that a higher portion of pyrite in the mixture results in lower dissolved oxygen in the slurry, generating a less oxidizing environment. Oxygen demand increases with increasing amount of pyrite in mineral systems. Similar results have been seen in the grinding of volcanogenic massive sulfide ores, which contain a large amount of pyrite,[15] where corrosion of the grinding media or the reaction of the dissolved oxygen with pyrite significantly reduces the dissolved oxygen concentration. Evidence from the ToF-SIMS analyses of sphalerite surfaces from the mill discharge samples shows that a much greater proportion of iron oxyhydroxyl species were produced as a result of increasing the pyrite in the feed ore (Figure 8). It can be seen from Figure 8 that by comparing the normalized intensity of FeO and FeOH on the sphalerite surface, the use of a high proportion of pyrite results in a significantly higher proportion of iron oxyhydroxyl species on sphalerite surfaces

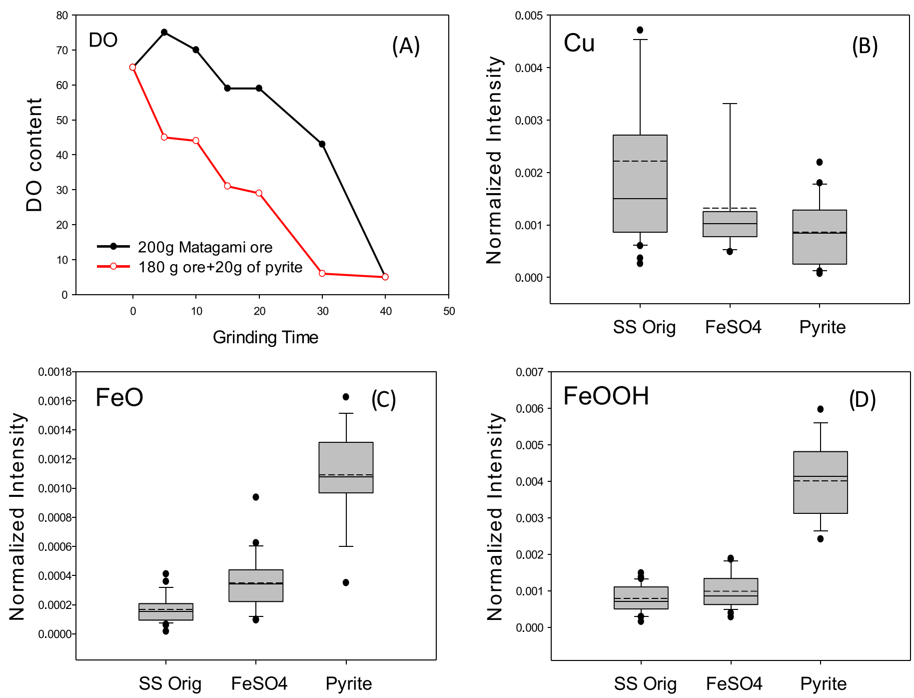

FIGURE 8 (A) Measured DO content in grinding tests with the Matagami ore and 20% pyrite increase in the same ore. (B), (C), and (D) Surface Cu, FeO, and FeOOH species on sphalerite surfaces as measured by TOF-SIMS. Abbreviations are as follow: no additions milling with stainless steel only (SS orig), milling with addition of $FeSO_4$ ($FeSO_4$), and with addition of 20% pyrite (pyrite)

relative to the tests with $FeSO_4$. The data indicate that pyrite relative to $FeSO_4$ may be more efficient in the depression of sphalerite. The results from the investigation show that the addition of extra pyrite during grinding facilitates an increase in oxygen consumption. Factors leading to the observed increase in the measured oxidative species on sphalerite could be related to an increase in the cathode to anode surface area ratio and, thereby, the possibility for greater galvanic interaction between the mineral phases in the pulp, along with increased rate of grinding media corrosion.

4 | CONCLUSION

Cathode mineral oxidation during grinding is an important factor in the selectivity of sphalerite and chalcopyrite flotation separation. Previous testing has established a link between surface species, especially the iron oxy-hydroxide oxyhydroxyl species, and poor sphalerite recovery. Here, 4 strategies were tested to evaluate the recovery of sphalerite by changes to the grinding environment: the use of 2 types of grinding media, milling with aeration, the addition of $FeSO_4$, and increasing the proportion of pyrite prior in the mill feed. Tests identified that grinding in a low oxidizing environment resulted in the development of a greater proportion metal oxide species on the surface of sphalerite. The sparging of air during grinding resulted in sufficient oxygen (seen increase in pulp DO) and a correlated increase in the development of oxy-hydroxide species on the surface of sphalerite grains. We believe that air supply in the mill accelerates the oxidation process of minerals and grinding media, generating more metal oxides/hydroxides (as observed on the surface of sphalerite) relative to the closed system test. Tests with the addition of 20% pyrite show lower DO and lower ORP relative to tests on the normal feed ores. The surface chemistry of sphalerite from the 20% additional pyrite tests shows significantly greater proportions of iron oxyhydroxyl species generated on the surface of sphalerite, resulting in sphalerite depression. The testing identifies that the amount of metal oxidation species on sphalerite surfaces varies with different grinding environments. Grinding testing and surface chemistry study have identified that a greater proportion of iron oxidation species produced on a sphalerite surface can potentially limit copper adsorption. In the case of using reagent of $FeSO_4$, bench scale flotation result shows a lower sphalerite grade in responding to more iron oxidation species on surface of sphalerite. The data suggest that there may be potential opportunities to control Zn recovery through changes in grinding regimes.

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