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Effect of grinding media on zinc depression in a lead cleaner circuit

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ABSTRACT

Laboratory testwork, investigating the effect of high chrome grinding media in a lead regrind application has on subsequent metallurgical performance, was conducted at a large silver–lead–zinc operation in Australia. The initial data showed that the use of a more inert grinding media could have increased zinc losses to the lead cleaner concentrate if careful attention was not paid to alloy selection.

Diagnostic tests showed that iron hydroxide surface coatings generated by grinding media corrosion reactions are an effective depressant for sphalerite in this ore body, even though it is known that an excess of these coatings could depress both galena and sphalerite flotation. These tests demonstrated that a 1% chrome alloy produced the desired pulp chemical conditions to yield an increase in lead concentrate grade through the rejection of sphalerite from the lead circuit.

A plant trial was conducted in one of the two parallel grinding/flotation trains, and data collected for statistical analysis. During the plant trial, pulp chemical surveys of the regrind circuit were also taken to compare the effect of grinding media on the cleaner one feed slurry pulp potentials, dissolved oxygen, pH, temperature and EDTA extractable iron.

The statistical analysis showed clearly that the change to 1% chrome grinding media had a significant positive impact on improving galena/sphalerite selectivity during lead cleaner flotation and improved the lead concentrate grade. The improved metallurgical performance is explained in terms of modified pulp chemistry.

1. Introduction

Sequential lead–zinc flotation processes generally use differential flotation to recover galena in the first stages of flotation, followed by sphalerite flotation. This is made possible through the application of appropriate regulators and depressants which allow the selective adsorption of collectors (Bulatovic and Wyslouzil, 1995). In the case of the galena–sphalerite system, thiol collectors can directly adsorb on galena via an electrochemical mechanism, whilst sphalerite first requires activation (Basilio et al., 1996).

However, as many authors have stated (Grano et al., 1988; Von Reeken et al., 1989; Houot and Raveneau, 1992; Basilio et al., 1996; Trahar et al., 1997; Rashchi et al., 2002), when a mixed sulphide mineral system oxidises, the sphalerite can be inadvertently activated by heavy metal ions such as Cu^{2+}, Pb^{2+}, Ag^{+}, and Au^{+}. When collector is added, a considerable amount of this activated sphalerite can be recovered into the lead circuit concentrate, thereby causing a loss in zinc recovery.

The mechanism by which lead activation of sphalerite occurs is not clear, but many authors speculate that Pb^{2+} ions replace the Zn^{2+} ions from the sphalerite surface:

$$ZnS + Pb^{2+} = PbS + Zn^{2+} \quad (1)$$

and the mineral surface is hydrophobised by the formation of stable lead xanthate and/or dixanthogens (Basilio et al., 1996). In the range pH 7–10, some researchers (Trahar et al., 1997; Houot and Raveneau, 1992; Rashchi et al., 2002) found that lead hydroxides were the activating species, following the reaction:

$$SZnOH + PbOH^{+} = SZn\text{-}O\text{-}Pb^{+} + H_2O \quad (2)$$

where S represents the sulphide surface of the mineral.

The activation mechanism of lead appears to be strongly dependent on pH (Trahar et al., 1997; Rashchi et al., 2002; Houot and Raveneau, 1992). Some work by Trahar et al. (1997) indicated that at pH values of between 4 and 9, the sphalerite recovery with an equivalent dosage of 200 ppm lead ions was above 95%. However, at pH 10 the recovery markedly decreased, and by pH 12 the sphalerite recovery was close to zero. One reason for this noticeable pH effect was postulated to be that the activation by lead hydroxide diminishes when the pH is raised to values above

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its point of zero charge, which is typically 7.5 (Carresquero-Durán and Flores, 2009). However, caution must be taken when adjusting the pH in the lead circuit to depress zinc, as above pH values of around 10, the galena surface tends to become passivated by a hydroxyl layer (Von Reeken et al., 1989), which depresses lead flotation.

It has been reported in depth elsewhere (Adam et al., 1984; Iwasaki et al., 1983; Natarajan and Iwasaki, 1984; Kocabag and Smith, 1985; Yelloji Rao and Natarajan, 1989a,b; Forssberg et al., 1993; Cheng et al., 1993; Yuan et al., 1996; Greet and Steinier, 2004; Greet et al., 2005; Wei and Sandenbergh, 2007) that galvanic interactions occur in every grinding media-sulphide mineral system, which affects the subsequent flotation properties of the sulphide minerals through unselective surface coatings by iron oxidation products.

The use of a more inert grinding medium would help in reducing corrosion in the milling process which may reduce the cost of grinding and improve flotation recovery, but unfortunately, this also may reduce the selectivity of the process. For example, Wei and Sandenbergh (2007) experimented using a complex Pb–Zn ore from Rosh Pinah in Namibia and found that milling in stainless steel resulted in the highest recovery of lead over the whole size range, but the concentrate grade was compromised by high recoveries of zinc, iron and other non-sulphide gangue minerals. When they milled the same ore in a steel mill, the lead recovery dropped, but the selectivity increased due to a lower zinc recovery. The authors therefore concluded that the sphalerite was either activated during milling or was not depressed by the precipitation of ferrous corrosion products as would be the case in the steel milling environment.

It is highly likely that the observations made by Wei and Sandenbergh, 2007 were related to the reagent regime for the stainless steel grinding environment not being optimised to counter the lower levels of iron hydroxide coatings on the particle surfaces reducing galena/sphalerite selectivity. A plant trial in a polymetallic concentrator (Kinal et al., 2006) demonstrated that with the use of high chrome media, the reagent dosage rates were able to be significantly reduced whilst maintaining concentrate grades and recoveries.

Similarly, laboratory experiments have been conducted using a sphalerite-quartz synthetic ore, in which it was found that the use of a stainless steel mill increased the flotability of sphalerite in the presence of lead ions (Trahar et al., 1997). The authors found that in the pH range of 2–9, grinding sphalerite in a steel mill resulted in negligible flotation response both with and without additions of lead nitrate equivalent to 30–200 ppm Pb^{2+}. However, after grinding in a stainless steel mill, the addition of 200 ppm lead ions produced a sphalerite recovery of 60% at pH 4.

Despite the above literary evidence which may suggest the contrary should have occurred, Mount Isa Mines achieved evident improvement in fine galena flotation response in the laboratory through converting the grinding media from high carbon steel to a high chromium alloy (Cullinan et al., 1999). It was found that the maximum recovery of fine galena increased, whilst maintaining the recovery of the intermediate size fractions. The selectivity of galena against sphalerite was improved, despite a significant increase in the amount of oxidised lead ions present, as measured by the EDTA extraction method. This indicates that the change in grinding pulp chemistry when using high chrome media was optimal for this ore type.

As previous work at Cannington demonstrated that changing the pulp chemistry in the secondary tower mill had positive flow on benefits to lead and zinc flotation, there was scope to continue this investigation in the lead regrind mills. The following describes the outcome of this work.

2. Experimental

2.1. Background

Laboratory work examining the impact of inert grinding media during primary grinding on lead and zinc metallurgy was completed in 2004. The results of this study culminated in a plant trial whereby the cylpebs in the secondary mill (a tower mill) were replaced with high chrome balls. The results of the plant trial suggested that converting the tower mill to high chrome grinding media had a positive effect on metallurgy. In fact, the change in pulp chemistry, brought about by the change to a more inert grinding media, saw an improvement in the selectivity for galena against sphalerite, which placed more zinc into the zinc rougher feed process stream. With more sphalerite reporting to the zinc rougher feed consistently higher zinc recoveries were reported in the plant.

The positive changes to pulp chemistry and metallurgical response observed with the change in grinding media in the primary tower mill work provided the impetus to commence examining the impact of high chrome grinding media added to the lead regrind circuit on lead metallurgy. The initial study conducted on lead rougher concentrate, comparing forged steel media with a variety of high chrome (above 15% chrome) alloys showed forged steel produced a superior lead grade/recovery curve, with low overall zinc losses when compared to the high chrome alloys. That is, as the chrome content of the grinding media increased, and the mill pulp chemical environment became more oxidising, the lead flotation rate, concentrate grade and recovery noticeably decreased. The deterioration in lead metallurgy appeared to be related to the increased flotability of sphalerite as the chrome content of the grinding media increased.

A series of laboratory tests was completed to develop an understanding of this behaviour, and determine which alloy was best to subsequently trial in the plant.

2.2. Laboratory testwork

A sample of fresh lead regrind feed from BHP Billiton's Cannington mine in North West Queensland was used for all of the experiments. The circuit configuration follows the conventional sequential lead–zinc flotation flowsheet with the lead and zinc rougher concentrates reground prior to cleaner flotation.

Feed analysis of the ore showed a grade of 37% Pb, 16.5% Zn, 1670 ppm Ag, 10.7% Fe and 16.6% S.

Grinding was conducted in the Magotteaux Mill®, as described elsewhere (Greet et al., 2004), and a variety of grinding media was used, including forged steel along with 1%, 15%, 21%, and 30% chrome alloys. The ore was ground to achieve approximately 80% passing 22 μm as measured by a Malvern Mastersizer. Flotation tests were conducted in a 5 L Runge bottom driven cell with four concentrates collected over a total flotation time of 6.5 min. Various reagents were added depending on the diagnostic tests being conducted.

Pulp chemistry readings of pH, Eh, dissolved oxygen and temperature were taken throughout grinding and flotation along with EDTA samples taken from the flotation cell just prior to commencing flotation. The design of the Magotteaux Mill® allows pulp chemistry to be continuously measured and recorded during the grinding process, and the grinding chamber was purged with the required amount of gas in order to achieve similar pulp chemistries to that previously measured in the plant.

A combined concentrate sample from each of the forged and high chrome standard tests was provided to the Ian Wark Research Institute for surface analysis by Time-of-Flight Secondary Ion Mass

Spectrometry (ToF-SIMS) using a PHI model TRIFT 2000 spectrometer equipped with Ga liquid metal ion gun. The following fragments were selected for the statistical evaluation of mass spectra:

- positive: Mg^+, Al^+, Si^+, Ca^+, Fe^+, Cu^+, Zn^+, $FeOH^+$, Ag^+, Pb^+ and $PbOH^+$
- negative: CH^-, O^-, OH^-, S^-, PO^-, SO_3^- and EX^-.

2.3. Plant trial

During a period in April 2007, daily pulp chemistry and EDTA surveys were conducted in the lead cleaner one circuit at the Cannington plant to compare the difference between the mill loaded with standard media (cylpebs) and the mill freshly loaded with low chrome balls. Samples from the lead rougher concentrate, regrind cyclone underflow, regrind mill discharge, regrind cyclone overflow, cleaner one feed and cleaner one tail were measured for Eh, pH, dissolved oxygen and temperature, as well as analysed for EDTA extractable lead, zinc and iron.

Cannington personnel conducted frequent surveys of the cleaner one block over the ensuing two months, resulting in 85 data points for each flotation train. As the plant trial was conducted on two parallel circuits, the shift data that was collected was suitable to be analysed using the paired t-test. This involves taking the difference of two performance values which powerfully reduces the standard deviation due to error (Napier-Munn, 2005).

Using this method, the data from both A and B sides before the trial were compared for feed, recovery and grade in order to determine the difference between the trains under standard conditions. Following this, the same procedure was used to determine the difference between the A side on cylpebs and B side on low chrome media. Thus, if the original differences between the two trains can be negated, an indication of the effect of changing grinding media can be achieved.

3. Laboratory results

A typical set of laboratory flotation tests were initially conducted, comparing forged steel media with a variety of high chrome alloys. The results showed forged steel produced a superior lead grade/recovery curve, with low overall losses of zinc. As the chrome content of the grinding media increased, and the mill pulp chemistry environment became more oxidising (Table 1), the lead flotation rate, grades and recoveries noticeably reduced. A main reason for the drop in lead flotation performance was found to be poor selectivity for galena against sphalerite and non-sulphide gangue minerals.

As the liberation characteristics were nominally the same for both media types, the conclusion was drawn that it must have been the change in pulp chemistry affecting selectivity. Surface analysis (ToF-SIMS) of the lead concentrate from a forged and high chrome test showed that the sphalerite reporting to the lead concentrate in the high chrome case had more surface coverage of chromium and zinc than the sphalerite particles prepared using forged steel, which had a higher surface coverage of iron, lead and silver (Fig. 1).

Table 1
Pulp chemistry data for laboratory lead cleaner tests comparing fresh lead regrind feed ground with forged steel and high chrome media.

Media	Mill discharge			Flotation feed		
	Eh, mV (SHE)	pH	DO, ppm	Eh, mV (SHE)	pH	DO, ppm
Forged	−62	7.9	0.0	129	7.6	0.0
High Cr	199	8.4	3.9	195	8.4	2.9

This is interesting as it shows that the zinc activating ions, silver and lead, were more prevalent in the forged test than the high chrome case, and conventional wisdom would suggest that sphalerite recoveries in this instance should be elevated. However, sphalerite recoveries to the lead concentrate after grinding with high chrome media were considerably greater than those observed for forged steel grinding. The presence of more iron species on the surfaces of sphalerite ground with the forged media compared with the high chrome case indicated that it was iron coatings (presumably iron hydroxides/oxy-hydroxides from grinding media corrosion) which were controlling its flotability. That is, when grinding with forged steel the iron corrosion products are an effective sphalerite depressant, and counter the presence of activating species (for example, silver and lead). However, when these corrosion products are significantly reduced, the activating properties of these ions may be restored, resulting in significantly higher zinc recoveries to lead concentrate. Further, as the surface contamination of sphalerite by media corrosion products is lower (i.e. "cleaner" particle surfaces), the threshold levels of activating ions needed to achieve sphalerite flotation may be significantly lower.

Given this surface analysis knowledge, a variety of diagnostic tests were conducted, in order to improve the lead–zinc selectivity when grinding with high chrome grinding media. These included:

- Examining the effect of high intensity conditioning to shear the activated lead from the sphalerite surfaces as demonstrated by Morey (1988),
- Adding nitrogen during the grind to decrease the grinding pulp potential,
- Adding high levels of SMBX and $ZnSO_4$ to the flotation conditioning stage to improve the lead selectivity against zinc, and
- Adding iron sulphate powder ($Fe_2(SO_4)_3$) to the grind to try and simulate the forged media test (Fig. 2).

Unfortunately, with the possible exception of the iron sulphate tests, none of these methods managed to improve galena/sphalerite selectivity when grinding with high chrome grinding media, and restore the lead grade/recovery curve to the same position as that observed for forged media (see Fig. 2). The iron sulphate tests indicated a more electrochemically active grinding media would yield improvements in galena/sphalerite selectivity, so a low chrome grinding ball was tested. The chemical composition of the low chrome media is quite similar to that of forged steel, however with better heat treatment this media is more than 10 Rockwell harder than cylpebs; therefore it is likely to be an economic proposition on a wear basis.

The difference in grinding pulp chemistry between the two media types was noticeably smaller when the low chrome media was used in place of a high chrome alloy (Table 2). However, there was still a slight increase in pulp potential which would have an impact on flotation performance. In fact, Fig. 3 shows that the low chrome media had a beneficial impact on lead cleaner flotation with a small increase in grade and recovery, compared to forged steel.

4. Plant trial

Given the positive indications from the laboratory tests using low chrome media, towards the end of March 2007 a plant trial was undertaken at Cannington where the grinding media from one of the lead regrind mills was dumped and replaced with low chrome. As the lead cleaner one circuit operates with two parallel grinding/flotation trains, a direct comparative trial was possible.

Fig. 4 shows the Eh profiles through the lead cleaner one circuit comparing the two regrind mills. It is quite clear that the pulp

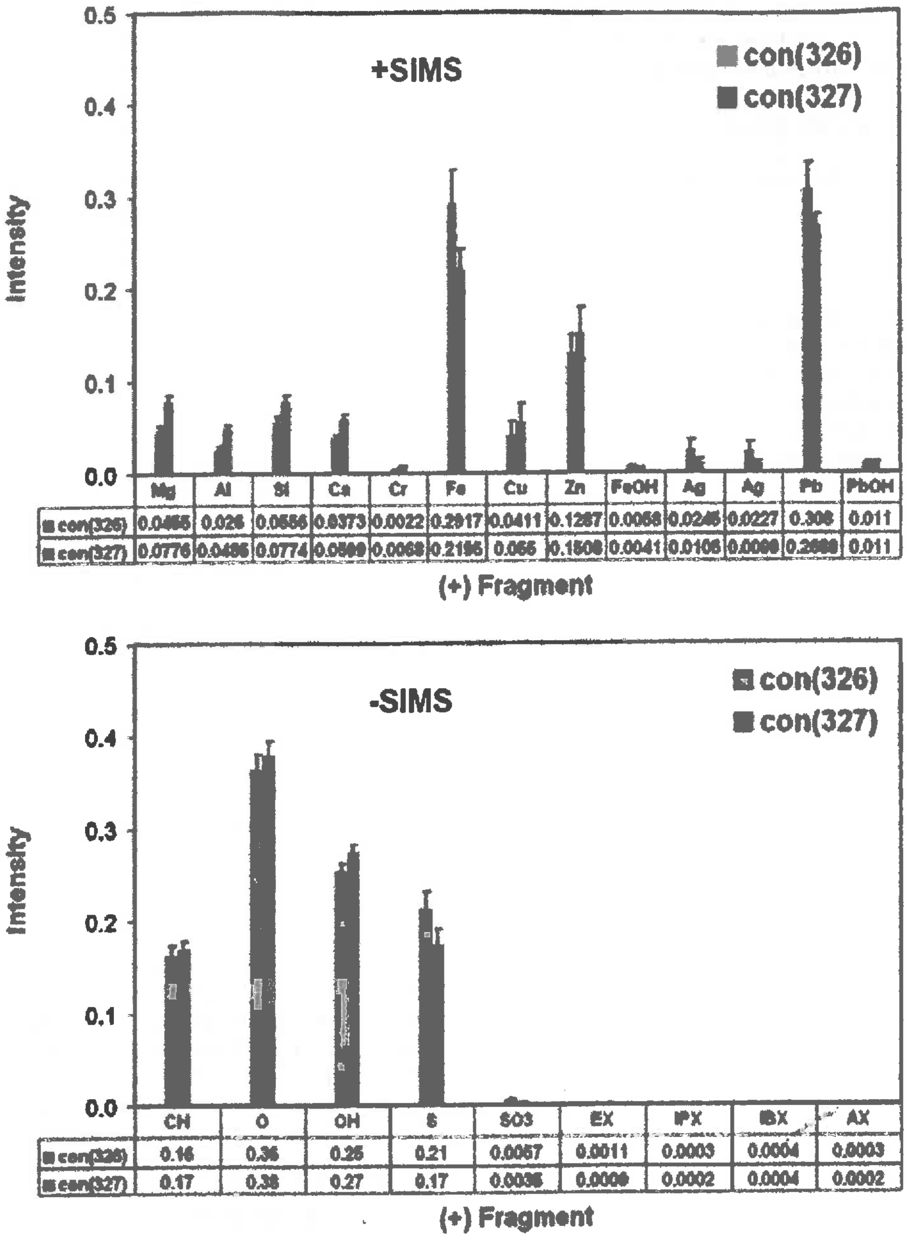

Fig. 1. Statistically evaluated (+) and (−) mass spectra for sphalerite in concentrates during tests 326 (Forged) and 327 (High Cr). Confidence intervals were calculated for $P = 95\%$.

potentials of the A side (cylpebs) regrind mill discharge are lower than that of the B side (low chrome) mill. This pulp chemistry change would likely be due to the lower quality of the cylpebs leading to increased wear rates during grinding. The pH and dissolved oxygen profiles showed negligible differences between the two mills. Interestingly, there was not much difference in the level of oxidised lead or iron as measured by the EDTA extractable method, throughout the grinding circuit regardless of grinding media type.

As there was insufficient data collected prior to the B side mill grinding media change, the confidence values are lower for the "before" period. The lowest acceptable confidence value for this type of plant trial is 90%, so any readings under that are assumed to indicate no difference between A and B sides.

A careful study of Table 3 indicates that under standard conditions with both regrind mills operating using cylpebs, there is no difference in lead feed grade between A and B trains but B side achieves about 0.5% lower lead recovery, with no statistical difference in grade. With the introduction of low chrome media to regrind mill B, the lead recovery decreased, resulting in an overall reduction of 0.09% lead recovery. However the difference in concentrate grade between the two trains increased by 1.54% lead, with greater than 99% confidence.

Shifting the focus to the zinc rejection in the lead cleaner one circuit, it appears that there was a significant difference in the zinc feed grades between A and B trains prior to the trial commencing with the B side receiving a zinc feed grade 0.73% higher than A side. For the period after the trial, the difference is reduced but still remains skewed towards the B side, with a zinc feed grade 0.58% higher. The difference in zinc recovery and grade to the lead cleaner one concentrate prior to the trial commencing is statistically insignificant, so it can be stated that the introduction of low chrome media to the regrind mill B resulted in a 2.94% decrease in zinc recovery with a corresponding 0.38% drop

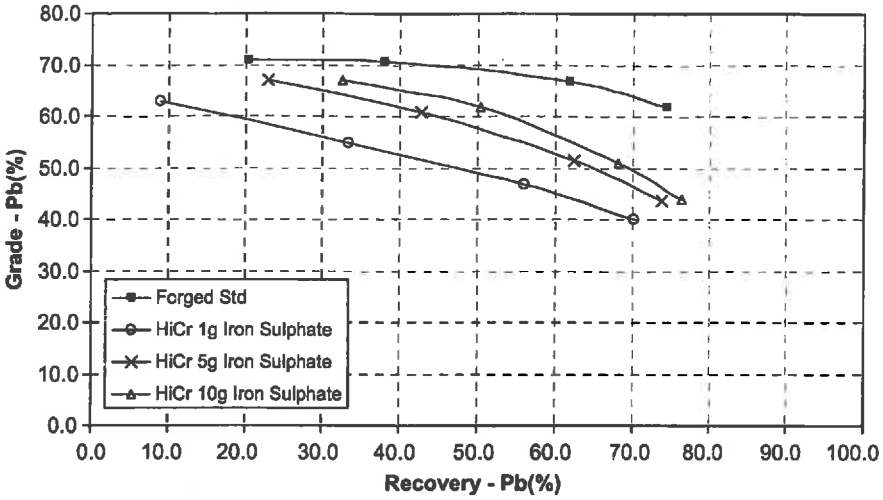

Fig. 2. Lead grade–recovery curves showing the effect of increasing the addition rate of iron sulphate powder to the high chrome grind.

Table 2
Pulp chemistry data for laboratory lead cleaner tests comparing fresh lead regrind feed ground with forged steel and low chrome media.

Media	Mill discharge			Flotation feed		
	Eh, mV (SHE)	pH	DO, ppm	Eh, mV (SHE)	pH	DO, ppm
Forged	−84	8.26	0.11	−110	8.34	0.00
Low Cr	−32	7.93	0.12	−69	8.13	0.00

in zinc grade. This change occurred with a 0.15% drop in zinc head grade.

Contrary to expectations, the silver performs in the opposite manner to the lead, in the lead cleaner one circuit. Table 1 shows that, with an increased head grade, there is negligible change in silver recovery but the B side silver grade noticeably improves over the A side, after the introduction of low chrome media.

5. Discussion

It is clear that given an oxidising environment in the Cannington lead regrind mill, the selectivity for galena against sphalerite is not maintained in the subsequent lead cleaner one flotation stage. The reasons for this were not through sphalerite activation (by lead or silver ions), as expected, but rather due to a lack of iron hydroxide coatings which were acting as a depressant.

Strong evidence exists in the literature regarding the activation effect of lead and silver ions on sphalerite, and as the ore in the Cannington lead regrind mill has an abundance of these minerals, this was thought to be a likely cause. As stated very early in the piece by Gaudin et al. (1960), a decrease in particle size may increase the degree of surface oxidation and the degree of interaction of the finer particles with other fine hydrophilic or slime particles. Supporting this, Basilio et al. (1996) mention that the lead activation of sphalerite in the presence of galena may become more serious when a lead–zinc ore is finely ground with a long retention time, followed by a long flotation time with a large circulating load, which is a similar case to that presented in this paper.

However, the surface analysis data taken from the flotation concentrates after grinding with forged versus high chrome media indicates that there is not an excess of the activating lead and silver ions present on the sphalerite in the high chrome case, but rather a lack of iron hydroxide coatings when compared with the samples ground with forged media. Therefore it appears that the strongest mechanism for sphalerite depression in this ore is deposition of iron hydroxide species from the grinding media corrosion reactions.

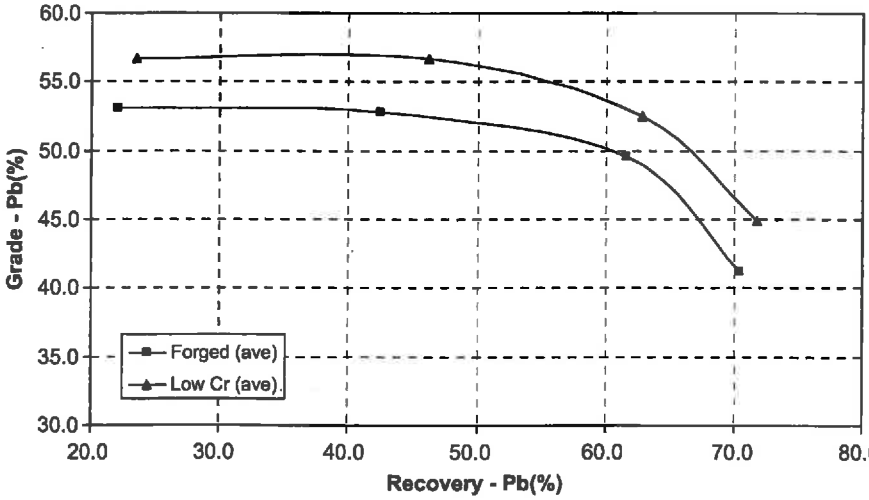

Fig. 3. Forged versus low chrome media on lead rougher concentrate samples collected from the plant in December 2005 (average from triplicate tests shown).

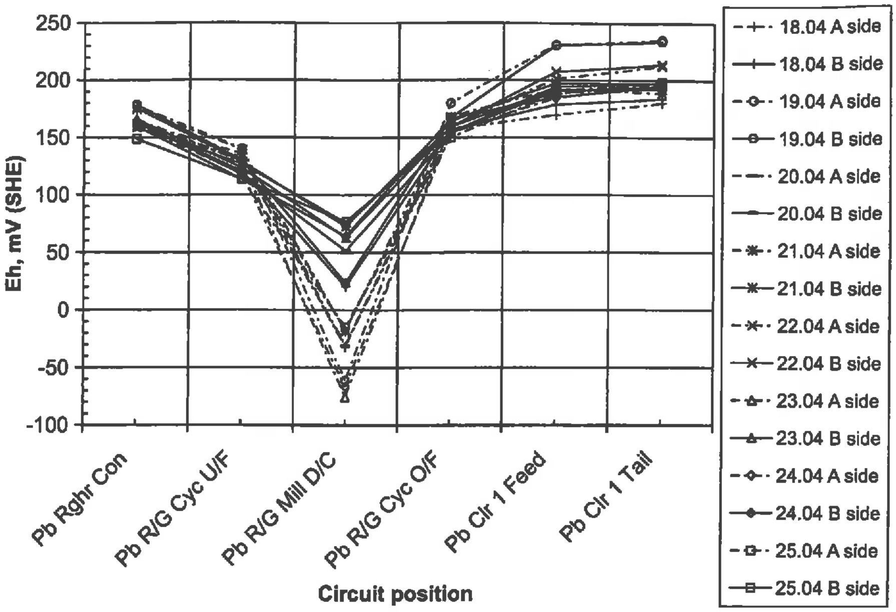

Fig. 4. Eh profile through the Cannington lead cleaner one circuit comparing the two regrind mills; one running on cylpebs (A side) and the other on low chrome (B side).

Table 3
Statistical difference and related confidence values between Line B and Line A (B–A) for lead cleaner one feed, recovery and grade before and after the B side regrind mill change to low chrome media, calculated using the paired *t*-test.

	Before		After	
	Difference	Confidence (%)	Difference	Confidence (%)
Lead				
Feed	0.48%	75.11	0.37%	88.28
Recovery	−0.52%	91.60	−0.61%	100.00
Grade	0.44%	87.19	1.54%	100.00
Zinc				
Feed	0.73%	100.00	0.58%	100.00
Recovery	−0.11%	52.25	−2.94%	99.96
Grade	−0.17%	85.85	−0.38%	99.89
Silver				
Feed	16.73 ppm	65.44	29.86 ppm	92.51
Recovery	−0.68%	93.21	−0.65%	99.90
Grade	11.44 ppm	63.13	91.34 ppm	99.98
Iron				
Feed	−0.06%	63.38	−0.02%	63.79
Recovery	3.04%	93.68	−2.52%	99.93
Grade	−0.02%	59.30	−0.79%	91.86

There were two issues to tackle with the Cannington trial. Firstly, the site was after an improvement in lead flotation performance, especially the fine lead, through the reduction in iron hydroxide coatings on the galena. Secondly, the improvement in lead flotation performance was to be achieved whilst maintaining, or improving the selectivity for galena against sphalerite. As adequate liberation was achieved at the target grind size, the problem had to be resolved by changing the grinding chemistry.

It took a number of experiments over a long time frame (approximately 2 years) before the solution, a low chrome alloy, was tested and eventually trialed. The use of this low chrome alloy appeared to cover both issues by slightly improving the grinding pulp chemistry through the use of a more wear resistant ball, whilst maintaining a level of iron hydroxide debris in the pulp to successfully depress the sphalerite.

This and observations made at other lead–zinc operations throughout the world suggests that it is possible to tailor the grinding chemistry of the system to achieve the best metallurgical performance by correct grinding media alloy selection. A one size fits all approach to the inclusion of high chrome grinding media within a circuit may end in disaster. The impact of the mineralogy and grinding media alloy selection on grinding chemistry and the subsequent metallurgical performance of a plant are not to be understated.

6. Conclusion

The following conclusions are drawn from this work:

- The use of high chrome grinding media during regrinding is potentially detrimental to lead cleaner flotation at Cannington, due to the cleaner system preventing iron hydroxides from coating the sphalerite particles and depressing its flotation.
- High intensity conditioning, nitrogen gas purging during grinding, and increased addition of SMBS and $ZnSO_4$ to the high chrome tests in the laboratory did not improve flotation to match that of forged media.
- The results of the addition of iron (as $Fe_2(SO_4)_3$) suggested that to improve galena/sphalerite selectivity a more electrochemically active alloy (i.e. a low chrome alloy ball) was required. Laboratory tests confirmed this result, and the trial of the low chrome alloy in the lead regrind mill improved the lead concentrate grade through the rejection of sphalerite at the same recovery, under the same reagent regime that is currently used.
- Changes in pulp chemistry in the Cannington lead regrinding circuit altered the lead to zinc selectivity relationship and therefore the lead grade/recovery curve.
- This and observations made at other lead/zinc operations throughout the world suggest that it is possible to tailor the grinding chemistry of the system to achieve the best metallurgical performance by correct grinding media alloy selection. A one size fits all approach to the inclusion of high chrome grinding media within a circuit is not appropriate. The impact of the mineralogy and grinding media alloy selection on grinding chemistry and the subsequent metallurgical performance of a plant are not to be understated.

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