

TECHNICAL PROGRAMME

MONDAY, OCTOBER 25, 2004, A.M.

9:00 — OPENING REMARKS: M.J. COLLINS AND V.G. PAPANGELAKIS

PLENARY SESSION

Session Chairs: V.G. PAPANGELAKIS and M.J. COLLINS
Room: Salon C

Plenary 1 — 9:15

Fifty Years of Pressure Hydrometallurgy at Fort Saskatchewan.
M.E. CHALKLEY, Sherritt International Corporation

Coffee Break 10:00 - 10:15

Plenary 2 — 10:15

Bayer Process Alumina Production – Alcan Bayer Experimental Centre.
G. FORTÉ, Alcan International

Plenary 3 — 11:00

Copper Pressure Leaching.
J. MARSDEN, Phelps Dodge Corporation

MONDAY, OCTOBER 25, 2004, P.M.

SESSION 1A - PRESSURE HYDROMETALLURGY OF COPPER I

Session Chair: R. BEREZOWSKY, Dynatec, Canada
Room: Salon B

Paper 1A.1 — 14:00

Testing and Modelling a Novel Iron Control Concept in a Two-stage Ferric Leach/Pressure Oxidation Process for the Sepon Copper Project.

K. BAXTER, T. PAVLIDES, Bateman Engineering, and
D. DIXON, University of British Columbia

The Sepon process plant utilises an autoclave to oxidize a flotation concentrate containing pyrite, residual copper minerals, and sulphur. The concentrate is produced from the residue of a preceding atmospheric copper leaching circuit. The function of the autoclave is to provide iron and acid for that atmospheric copper leaching circuit. The key issues in the operation of the autoclave are to maximise the production of acid and ferric ions and to avoid scaling issues related to sulphur balling in the autoclave. Scale control in the autoclave was examined using various dispersants and residue recycle without success. Operation of the autoclave at high temperature and high acidity addressed the scale issue through rapid oxidation of the sulphur and produced a ferric sulphate precipitate in the residue that could be re-leached to provide ferric iron. This paper presents the development steps followed, through batch and continuous piloting that resulted in an autoclave circuit design that addressed the above process requirements. Following definition of the process design basis, modelling of the autoclave operation was carried out to assist with engineering design. The model included thermodynamics and kinetics of oxidation of pyrite, elemental sulphur and covellite and also considered gas/liquid mixing, oxygen solubility as a function of temperature and solution composition, ferrous to ferric oxidation, gangue reactions with acid and precipitation/re-dissolution of basic ferric sulphate and alunite.

Paper 1A.2 — 14:25

Concentrate Leach Plant Start-up, Operation and Optimization at the Phelps Dodge Bagdad Mine in Arizona.

J.C. WILMOT, Phelps Dodge,
R.J. SMITH and R.E. BREWER, Phelps Dodge

In March 2003, Phelps Dodge started up an innovative pressure leach facility for processing copper concentrates at the Bagdad mine in Arizona. This paper describes the development of the concentrate leaching plant at Bagdad and the operational results from start up to present. Optimization efforts and an approach to mineralogical modeling of the concentrate to calculate oxygen utilization are also presented.

Paper 1A.3 — 14:50

Copper Concentrate Pressure Leaching Studies at Anglo
American Chile.

G. ZÁRATE and G. TAPIA, Anglo American

Anglo American Chile has been doing copper concentrate leaching studies at the laboratory and small pilot plant scale since 1992. Among the processes evaluated are bacterial leaching and pressure leaching using Mantos Blancos, Salobo Project and Quellaveco Project concentrates. Late in the nineties, it was decided to focus the research on pressure leaching, either medium or total pressure oxidation. Preliminary scouting tests were run with Mantos Blancos concentrate samples during 2000, which were repeated during 2003 together with samples of Los Bronces and El Soldado concentrates, in order to define the most suitable concentrate for running a pilot plant campaign at AARL, which was carried out in two campaigns, a first one from November 2003 through January 2004 and a second one during April 2004. Mantos Blancos was the selected concentrate and the laboratory and pilot plant results are discussed in this paper together with some preliminary economic estimates.

Coffee Break 15:15 - 15:30

SESSION 1B - LATERITES I

Session Chair: D. MUIR, CSIRO Minerals, Australia
Room: Salon C

Paper 1B.1 — 14:00

The Western Australian Ni Laterites Projects — What Have We Learned?
R.W. NICE, RW Nice & Associates Pty Limited

Presently about 60% of the world's nickel resources are tied up with lateritic ores while about 25% of the world's nickel is produced from sulphides. Based on this the industry has to focus on improving lateritic nickel economics in order to maintain the high use of the metal worldwide. In 1997 three junior mining companies in Australia organised finance against all odds and commenced development of three separate nickel and cobalt extraction plants treating lateritic nickel ores. All three projects utilise similar pressure acid leaching (PAL) technologies while each has unique metal recovery technologies. All projects have experienced numerous processing and maintenance related difficulties such that full ore throughput has been reached by only one operation after five years. This shortfall in performance has caused the owners considerable angst and financial pain. In 2000 Cawse went into receivership and was purchased by an international corporation. Recently, Bulong also went into receivership and has undergone an orderly shutdown, awaiting sale to a new buyer. Murrin Murrin has settled with its financiers and is in a position to operate as planned for many years forward. This paper will discuss a number of the technical difficulties that affect the ore preparation, PAL and solution treatment circuits of these plants. The objective is to identify the cause of some of the difficulties and propose possible solutions so that the next generation of lateritic operations can emerge as technically and, more importantly, economic successes. A number of these process improvements have been identified by the existing operators and in many instances have been or are being implemented into the existing operations.

Paper 1B.2 — 14:25

Effect of Process Water and Leach Temperature on the Leaching Behaviour of New Caledonian Nickel Laterite Ores.
M. BUARZAIGA and M. LAFRAMBOISE, Falconbridge

High-pressure acid leaching (HPAL) of nickel laterites has received considerable attention in recent years. High pressure extraction, good selectivity, and advances in pressure hydrometallurgical technology are some of the reasons for this interest. Falconbridge Limited (Sudbury, Ontario) investigated the HPAL process using both fresh water and seawater for ore processing. The use of seawater resulted in a chloride concentration of 7.8 g/L in the autoclave feed. A test program was developed to study the effect of process water and leach temperature on the leaching behaviour of different ore compositions from the Koniombo deposit in New Caledonia. The work showed that salinity has a major effect on autoclave chemistry. This paper explores the results.

Paper 1B.3 — 14:50

Effect of Ammonium, Sodium and Potassium Sulphates and Chlorides in the Pressure Acid Leaching of Western Australian Nickel Laterite Ore.

J.A. JOHNSON and B.I. WHITTINGTON, CSIRO Minerals

This paper examines the effect of cation type (i.e. NH_4^+ , Na^+ and K^+) and anion type (i.e. SO_4^{2-} and Cl^-) on the extraction of nickel during the pressure acid leaching of a Western Australian laterite ore. The results are compared with testwork conducted with other Western Australian nickel laterite ores and the possible process implications of adding an additive to a plant autoclave are considered. Addition of either $(\text{NH}_4)_2\text{SO}_4$, Na_2SO_4 or K_2SO_4 increased both the nickel extraction and the acid consumption, provided that the level of additive was not too great. For example, the nickel extraction of 93% increased to above 96% in the presence of additives, but decreased to 94% when, for example, NH_4^+ levels increased from 7 kg/t ore to 21 kg/t ore. The relative stability of the jarosite species formed when additives were present influenced the amount of jarosite in the residue and, hence, both the final free acidity and overall nickel extraction. The use of Cl^- versus SO_4^{2-} salts as additives had little effect on overall nickel extraction or overall free acid values, although the presence of SO_4^{2-} additives appeared to enhance nickel extraction kinetics over the first 60 minutes of the reaction. In contrast, manganese extraction was enhanced by the presence of Cl^- additives.

Coffee Break 15:15 - 15:30

SESSION 2A - PRESSURE HYDROMETALLURGY OF COPPER II

Session Chair: D. DIXON, University of BC, Canada
Room: Salon B

Paper 2A.1 — 15:30

The Effect of Surfactants on the Behaviour of Sulphur in the Oxidation of Chalcopyrite at Medium Temperature.

J.D.T. STEYL, Anglo American Research Labs (Pty) Limited

Anglo American Corporation and the University of British Columbia have developed a medium-temperature oxygen pressure leaching process for the extraction of copper from chalcopyrite concentrates in an acid-sulphate system (AAC/UBC process). Leaching is performed at 150 °C and surfactants are added to disperse molten elemental sulphur from the unreacted sulphide surface to prevent liquid sulphur occlusion. Copper extractions of over 95% and elemental sulphur yields of around 60% were observed over a 2-hour average residence time in a continuous pilot autoclave. Sporadic blockages occurring under continuous conditions were found to be caused by the accumulation of molten elemental sulphur mixed with unreacted sulphide mineral. A batch programme of experimentation was undertaken to establish the reason for this phenomenon. Batch experiments were conducted at low pulp densities on concentrate samples with narrow size distributions and at similar solution compositions to those experienced in the pilot autoclave. The overall rate of chalcopyrite oxidation was shown, under approximately constant solution composition, to be a strong function of the total available mineral surface area. Initial copper extraction rates followed the shrinking particle kinetic model under intrinsic surface control. Deviation from shrinking particle kinetics was however observed after a certain oxidation time. Secondary electron images identified small (typically less than 20 µm) sulphur conglomerates at the point of deviation, which 'collected' unreacted chalcopyrite mineral particles and lowered the total mineral surface area available for oxidation. These sulphur clusters are thought to be the precursor for blockages experienced in the continuous pilot autoclave. These results emphasised the importance of fine grinding (P_{80} 5-20 µm) and suggest that shorter residence times may minimise the formation of sulphur conglomerates in a continuous autoclave operating at medium temperature.

Paper 2A.2 — 15:55

Hydrogen Reduction of Copper (I) Oxide in an Autoclave.

E. JÄÄSKELÄINEN, O. HYVÄRINEN and M. HÄMÄLÄINEN, Outokumpu Research

HydroCopper™ is a new hydrometallurgical process for the treatment of copper sulphide concentrates, which has been developed by Outokumpu. The process includes chloride leaching of copper concentrate, solution purification and precipitation of copper(I) oxide, an intermediate product, which is reduced and cast into copper products. The reduction can be carried out by pyrometallurgical methods or hydrometallurgically in an autoclave. This paper discusses the thermodynamic and kinetic aspects of the hydrogen reduction of copper(I) oxide in an autoclave. The results of the laboratory tests are presented and discussed. Finally, the autoclave method is compared with other options.

Paper 2A.3 — 16:20

Hydrometallurgical Processing of Telfer Concentrates: Accepting and Winning the Challenge.

T. GIRAUDO, D. LUNT, J. CANTERFORD, GRD Minproc Limited,

R. DUNNE, D. MITCHELL, formerly Newcrest Mining Services Limited, and

D. DREISINGER, University of British Columbia

Telfer bulk concentrates typically contain 8% chalcopyrite, 8% chalcocite, 45% pyrite and 80-120 g/t gold. Although very high copper and gold recoveries can be obtained by subjecting the concentrate to a "conventional" high temperature pressure oxidation step followed by copper recovery by solvent extraction and electrowinning, and cyanidation of the leach residue for gold recovery, the cost of lime required for neutralisation and power for oxygen generation was such that the process was uneconomic in terms of operating costs. The challenge therefore was to develop an alternative flowsheet that maximised copper and gold recovery in which the reagent costs were commercially attractive. An additional objective was to reduce the capital cost so that the overall NPV and IRR values would enhance the economic viability of the total project. Following a series of batch and locked cycle testwork programs, supported by Metsim modelling, it was established that the same high levels of overall copper and gold recoveries can be obtained by use of a novel two-stage split leaching circuit in which the chalcocite component is leached in a low temperature (90-110 °C) circuit with the chalcopyrite component treated in a high temperature (220-250 °C) circuit. This split circuit has a number of advantages, including minimising the required capacity of the high temperature autoclave, as well as reducing the overall lime consumption and simplifying the total process water balance. The paper outlines the basis for the development of the flowsheet and discusses the integration of the leaching stages into the overall circuit.

Paper 2A.4 — 16:45

Hydrogen Reduction of Copper and Nickel in the Processing of Copper Bleed Solution of Indian Sea Nodules.

V. KUMAR, B.D. PANDEY, D. BAGCHI and PREMCHAND,

National Metallurgical Laboratory of India

During the separation and recovery of metals from the ammoniacal leach liquor of Indian Ocean nodules by solvent extraction-electrowinning (SX-EW) in close-loop operation, the copper electrolyte containing ~40 g/L Cu, 20 g/L Ni and 180 g/L sulphuric acid is bled-off to control the impurities. The recovery of copper and nickel as powders has been examined from the copper bleed solution following partial decopperisation, crystallization of mixed sulphate of the two metals and aqueous hydrogen reduction. From the dissolved sulphate solution containing 17.68 g/L Cu, 18.83 g/L Ni and 0.03 g/L Fe, the copper powder was precipitated at initial hydrogen pressure of 26 bar in an autoclave in 90 min at 140 °C. The residual copper from the spent solution was removed as sulphide and nickel powder was then recovered (99%) in two stages from a solution of pH 4.5 at 40 bar pressure and 190 °C. However, 98% nickel recovery in one stage was achieved when hydrogen reduction was carried out from an ammoniacal solution in the pH range 9-11. Copper and nickel powders were produced from the bleed solution with acceptable purity and specifications suitable for P/M applications.

SESSION 2B - LATERITES II

Session Chair: D. KERFOOT, INCO TSL, Canada
Room: Salon C

Paper 2B.1 — 15:30

Chromium Removal from Leach Liquors Produced during High-pressure Acid Leaching of Nickel Laterite Ores.
M. BUARZAIGA and D. MARSHALL, Falconbridge Limited

High-pressure acid leaching (HPAL) of nickel laterite ores typically extracts 95% of the nickel and 91% of the cobalt. Impurity elements in the feed, such as iron, aluminium and chromium, also dissolve and can be detrimental to downstream processes. Rejection of impurities within the autoclave is highly desirable. Aluminium and iron hydrolyze, and then precipitate in the autoclave leaving only small amounts that are subsequently removed by precipitation in a neutralization step. The concentration of dissolved chromium can range from 200 to 2000 ppm, depending on the feed composition. Chromium in the liquid phase, usually in the hexavalent state, introduces many problems and can limit refining options. For example, chromium poisons the organics in solvent extraction making SX an expensive option. Also, hexavalent chromium must be converted to the trivalent state before it can be precipitated along with iron and aluminium. Bench-scale work confirmed that mixing small amounts of carbon-base additives, such as coke, with the autoclave feed significantly reduced chromium concentration in the autoclave discharge without any detrimental effects on paymetals recoveries and selectivity.

Paper 2B.2 — 15:55

Murrin Murrin CCD1 Rake Mechanism Modification: The Decision and the Result.
J. BELKE, Outokumpu Technology Pty Ltd.
B. HEWITT, Minara Resources

Murrin Murrin Operations has not had a successful beginning. Numerous reliability and operability issues many of which are the result of design decisions have been addressed through mechanical and operational changes and the CCD circuit is no exception. The decision to install, the installation and the results of the CCD1 rake-lift mechanism and feedwell dilution system are discussed in this paper. The installation was justified on improved operational robustness and an increase in the in the underflow density of 1% (per cent w/w). At approximately the same time process modifications in the solution neutralisation circuit were implemented and these changes altered the rheology of the slurry reporting to CCD1. Since the modifications to CCD1 and the solution neutralisation circuit, the CCD circuit has averaged a 2% improvement in the circuit underflow density. The CCD1 modification was completed on time and mechanically has performed well. From a risk point of view, the central rake-lift and Turbodil® dilution system add a degree of freedom and robustness to CCD1 operation that has extended the range of operational confidence. In this respect, the modification has been a success and is complementary to solution neutralisation improvements.

Paper 2B.3 — 16:20

Kinetics of High Pressure Leaching of the Nickel Lateritic Ore.
S. STOPIC,
B.F. FRIEDRICH, IME Process Metallurgy & Metal Recycling, and
N. ANASTASIJEVIC, Outokumpu

The extraction of nickel from Serbian and Western Australian lateritic ore by sulphuric pressure acid leaching (PAL) has been studied. The following parameters were tested: stirring rate, acid to ore ratio, reaction time, pressure and temperature. These studies were performed with ore samples from the Rudjinci, Serbia and Silcrete, Australia, both belonging to the laterite-silicate group of deposits. An increase of the reaction temperature and the stirring speed at a fixed acid/ore ratio showed a positive influence on nickel extraction. The decrease of the oxidation potential with time correlated with the increase in Fe(II) in solution and nickel extraction as the reaction went to completion for both ores. The target values of 95 % Ni extraction and 60 % Co extraction were attained in 5 min at 250 °C for the Serbian silicate ore, at an agitation rate of 1500 rpm, 10 % solids, and acid/ore ratio of 0.40. Under similar conditions only 60 % Ni, but 69 % Co could be extracted from the Australian Silcrete ore due to different mineralogy. The nickel extraction was shown follow a diffusion-controlled reaction model.

Paper 2B.4 — 16:45

Process Development Studies Carried out by Dynatec as Part of the Ambatovy Nickel Project Evaluation.
M.J. COLLINS, L.A. BARTA, K.R. BUBAN, R. KALANCHEY, G. OWUSU, R. RAUDSEPP, J. STIKSMA and I.M. MASTERS, Dynatec Corporation

This paper describes the results of testwork for the treatment of Ambatovy laterite by an acid pressure leach process, with recovery of the soluble nickel and cobalt values as mixed sulphide intermediate by precipitation with hydrogen sulphide, and subsequent refining of the mixed sulphide to nickel and cobalt metals. These process development studies were carried out primarily in 2004, as part of a bankable feasibility study for the Ambatovy Nickel Project.

TUESDAY, OCTOBER 26, 2004, A.M.

SESSION 3A - PRESSURE HYDROMETALLURGY OF NICKEL SULPHIDES

Session Chair: D. DREISINGER, University of BC, Canada
Room: Salon B

Paper 3A.1 — 8:30

Flammability of Ammonia Leach Solution Vapours under an Atmosphere of Enriched Oxygen.

J. BUDAC, M. SJOGREN, D. BELTON and R. KOFLUK, Sherritt International

A series of tests were performed to determine the flammability limits of mixtures of oxygen, ammonia, steam and nitrogen, over a solution, in a sealed autoclave. The partial pressures of ammonia and oxygen in the vapour space were varied by adjusting the character of the solution and by adjusting the oxygen concentration in the oxidant gas respectively. The data shows that flammable conditions can be minimized by controlling the operating temperature, reducing the ammonia content in the solution, increasing the concentrations of Ni, Cu and Zn in solution and keeping the % oxygen in the gas phase less than 15.5. Observations were also made that suggest that the presence of an aerosol in the vapour space may hinder ignition.

Paper 3A.2 — 8:55

Investigations of Method for Addition of Surfactant to the Process of Pressure Leaching of Nickel Containing Pyrrhotite.

A.V. TARASOV and E.M. TIMOSHENKO, State Research Institute of Non-Ferrous Metals

The results of laboratory, bench-scale and semi-commercial investigations of a method for adding surfactant (technical-grade lignosulphonate) to the process of pressure leaching of nickel-containing pyrrhotite concentrates have been discussed. It has been demonstrated that feeding of technical-grade lignosulphonate distributed along the length of an autoclave ensures a reduction in its requirement by a factor of 1.5 to 2 as compared with single-point feeding; it also results in a decrease by 5% in the unit requirement of metallized iron-bearing precipitator in the process of non-ferrous metals precipitation and reduces by 0.05 to 0.1% (abs.) the nickel and copper contents in the tailings of the given process. Technical solutions have been proposed for commercial introduction of the process.

Paper 3A.3 — 9:20

Development of an Effective Process for Pressure Disintegration of Sulphur-Sulphide Concentrates in the Hydrometallurgical Treatment of Nickel Pyrrhotite Materials.

M.N. NAFTAL, Norilsk, and

S. NABOICHENKO, The Urals State

In the process of pressure disintegration of sulphur-sulphide concentrates (SSC), secondary chemical interactions proceed caused by the high basicity of calcium hydroxide used as a hydrophilizing agent. The sulphur-sulphide concentrates disintegration process has been developed according to which an active mineral additive (AMA) is used to fix $\text{Ca}(\text{OH})_2$ in a partially soluble complex under given conditions. Further, in the process of sulphur-sulphide concentrate disintegration, the "AMA \cdot Ca(OH) $_2$ " complex loses its stability and decomposes giving back into solution chemically free calcium hydroxide. The control of the kinetics of the complex disintegration allows control of $\text{Ca}(\text{OH})_2$ emission, which limits the depth of secondary chemical interactions. The developed disintegration process allows reduction of lime consumption by approximately 40% and two-fold reduction in elemental sulphur losses. The method of disintegration using the "AMA \cdot Ca(OH) $_2$ " complex has been adopted at Nadezhda Metallurgical Plant at Norilsk Nickel Polar Circle Division. The economic effect of this operation is about \$3,000,000 US per annum.

Coffee Break 9:45 - 10:30

SESSION 3B - AUTOCLAVE DESIGN I

Session Chair: K. FRASER, Hatch, Canada
Room: Salon C

Paper 3B.1 — 8:30

Oxidation Autoclave Agitation Review.

H. PIETERSE, Pieterse Consulting Inc.

Agitation requirements in oxidation autoclaves can vary widely depending on the mineralogy of the feed material and the specific process selected. Before selecting an agitation system it is important that the client specify the desired throughput, mineralogy, chemistry, kinetics, compartments, overall retention time, and the desired oxygen utilization. In selecting the agitation system, the factors mentioned above as well as oxygen solubility, oxygen mass transfer coefficients, and specific power are evaluated. Much research effort has been directed toward open tank systems for fermentation and oxidation processes. However, for high pressure autoclave oxidation processes little information is available. In this review the author's experience in the autoclave industry and some of the available literature are utilized to compare the installed power per unit volume with some of the methods proposed for open tank oxidation. Equations that appear to give results close to that experienced in the industry have been identified. Further work is required in this area to develop a higher degree of confidence in the ability to predict the power required for any given case.

Paper 3B.2 — 8:55

Measurement of Oxygen Transfer Rate with an EKATO Gassing Agitator in a
5 m³ Pressure Vessel.

J. JI, Placer Dome Research Centre, and

J. KING, Placer Dome Tech Services Limited

Oxygen requirement during acidic pressure oxidation of sulphide concentrates can be as high as 800 kg/(m³·h) in the first compartment of an autoclave when the total oxidation residence time is shortened to 30 minutes. To achieve such a high oxygen transfer rate while maintaining satisfactory oxygen utilization, a pilot plant campaign was launched at the Placer Dome Research Centre in 1998 to measure the oxygen mass transfer coefficient in a 5 m³ pressure vessel at a total pressure close to 500 psig with a 30 HP gassing agitator. Maximum oxygen flowrate to the pressure vessel was designed at 3600 kg/h. In this paper, a great deal of information is provided on the gassing agitator, pressure vessel, measurement procedure and results. Without oxygen sparging, the oxygen mass transfer coefficient is 2600 h⁻¹ with a mixing power input of 5.0 kW/m³ in solution. With oxygen sparging, the oxygen mass transfer coefficient increases to 3500 h⁻¹ with a mixing power input of 4.0 kW/m³ in solution.

Paper 3B.3 — 9:20

Design and Materials Selection of Autoclaves and Auxiliary Equipment.

R. CLARY, P. CHEUNG and J. GULYAS, SNC Lavalin Australia Pty Limited

Over the past 50 years, the use of autoclaves in the metallurgical industry has evolved from final metal recovery (hydrogen reduction) and purification (sulphide precipitate oxidation) to treating whole ores and concentrates. Recent process developments, such as the use of autoclaves for laterite leaching, have necessitated higher temperature and pressure operations, which in turn have seen marked advancements in technology and manufacturing capabilities to keep in step with process demands. This paper will discuss the advances in technology in terms of design, materials and construction of autoclaves and liners, typical dip tube design and geometric layout. This paper will also address specific auxiliary equipment, such as acid and steam injection systems, and components, such as agitator seal systems.

Paper 3B.4 — 9:45

Quality Assurance Programs for Fabrication of Specialized Vessels and Exotic Alloy Piping.

W. BRISTOWE, A. HANSON and M. PEARSON, Hatch

“There’s no such thing as quality assurance; only quality control ... by inspection.” A common misunderstanding in the procurement and construction of custom designed, fabricated pressure vessels and piping systems is that quality assurance can be achieved by simply referencing codes, standards, ISO 9002, QMS or other such programs in the contract documents. In the authors’ experience, nothing could be further from the truth, not detrimental to the performance of the equipment than the exclusion of inspection activities and reliance upon warranties and “fit-for-purpose” clauses. Likewise, the expectation that fabricators, suppliers, or third party inspectors can provide such assurances is also unrealistic where they have limited knowledge of the process, material selection criteria, or limited technical resources. The development and successful execution of a quality assurance program is a collaborative effort by engineers, designers, material suppliers, equipment fabricators, and field inspectors with the full understanding and support of the end user. This paper presents an outline of the quality control activities undertaken by the authors for several recently completed and on-going hydrometallurgical projects. The activities include: prequalification of fabricators and sub-vendors, shop audits, material validation testing, material source control and verification, positive material identification of weld metal overlay, specialised welding inspection, contamination control for titanium and titanium-clad fabrication, eddy current and mass spectrometer (helium) leak testing of shell-and-tube heat exchangers, hot cycle testing of titanium clad equipment, tolerance verification for refractory lined equipment, final inspection and cleaning.

Coffee Break 10:10 - 10:30

SESSION 4A - PRESSURE HYDROMETALLURGY OF PGMs

Session Chair: J. CANTERFORD, GRD Minproc Limited, Australia

Room: Salon B

Paper 4A.1 — 10:30

Development of a Process to Unlock Refractory Platinum Concentrates Prior to a Chloride-Assisted Pressure Leaching Process.

C.J. FERRON, C.A. FLEMING, SGS Lakefield,

D. DREISINGER, University of British Columbia, and

P.T. O’KANE, O’Kane Consultants

The PLATSOL[®] process, initially developed for the treatment of the NorthMet Cu/Ni/PGM concentrate, has seen over the recent years its applicability extended to include numerous other Cu/Ni concentrates of the same type, as well as other precious metals-containing feedstocks. However, several concentrates were found refractory to the typical PLATSOL[®] conditions, more particularly so the platinum. Attempts to extend those conditions, such as higher temperatures (250 °C), longer retention time, increased oxidation conditions, higher chloride additions, were not successful. Detailed mineralogical investigation of those PLATSOL[®] leach residues indicated the main refractory mineral to be cooperite (PtS). Further metallurgical investigations showed that a thermal pre-treatment of the concentrate effectively unlocked the refractory platinum minerals for the subsequent leach. Temperatures between 400 and 750 °C were tested, with the optimum being found around 650 °C. The kinetics of the pre-treatment process were fast, and one hour at temperature proved sufficient. Under those conditions, PLATSOL[®] extractions were re-established around 95% for both platinum and palladium. From a mineralogical examination of the calcine, it was shown that the effect of the pre-treatment was to transform the cooperite and other refractory sulphides into metallic platinum and alloys, amenable to the PLATSOL[®]. The implications of the pre-treatment step for the processing of PGM concentrates are briefly discussed.

Paper 4A.2 — 10:55

Modification of the Stillwater Base Metals Refinery Process.

L. NEWMAN, R. WYRICK, Stillwater Mining Company, and

R. BEREZOWSKY, Dynatec Corporation

The Stillwater Mining Company operates an integrated mines, mill, smelter and base metals refinery complex in Montana, USA for the production of a high-grade platinum group metals (PGM) bearing concentrate, nickel sulphate crystal and cathode copper from the PGM-containing nickel-copper converter matte. The complex was constructed in stages over several years, starting with the mine and the mill, followed by the smelter and finally, a base metals refinery, which was commissioned in 1996. Due to the relatively small quantity of matte production, a straightforward refinery process was initially selected to extract the base metals, which reported to a mixed nickel-copper solution for further treatment off-site, and yielded a high-grade PGM concentrate suitable for treatment in a PGM refinery elsewhere. Subsequently, as a result of increased matte production, the process was modified to separate the nickel and copper, and to produce a nickel sulphate salt and cathode copper. This paper describes the design and operation of the original and the modified processes utilized in the base metals refinery (BMR).

Paper 4A.3 — 11:20

A New Process for Treating Slimes after Copper and Nickel Electrorefining.

A.C. TER-OGANESYANTS, N. ANISIMOVA, G. KOTUKHOVA and G.N. DYDKO Norilsk Nickel

The process utilized at Norilsk Nickel Polar Circle Division for treating slimes from the electrorefining of copper and nickel was developed in the late nineteen fifties and has survived without any fundamental changes. This technology is a combination of pyro and hydrometallurgical operations to produce PGM concentrates that are sent away for refining. The characteristic feature of the current technology is multiple-stage, extended cycle treatment with significant incomplete production, low labour and automation efficiency and poor hygiene and sanitary conditions. To eliminate all negative aspects mentioned above, specialists of Norilsk Nickel Polar Circle Division have developed a new hydrometallurgical process for treating electrolytic slimes. The main steps of the flow sheet developed are autoclave leaching of the slimes and electrochemical opening-up of a cake from autoclave leaching. The implementation of the new process will allow Norilsk Nickel Polar Circle Division to shorten the process cycle 4-5-fold thus reducing the volume of incomplete production, considerably increase labour efficiency, simultaneously improving working conditions, upgrade the final concentrate, reducing costs and increasing PGM recovery during refining and, ensure higher security of precious metals by employing automated processes and vacuum sealed units.

SESSION 4B - AUTOCLAVE DESIGN II

Session Chair: J. GULYAS, SNC Lavalin, Australia

Room: Salon B

Paper 4B.1 — 10:30

Advantages of Bottom Entry Oxygen Introduction for Pressure Oxidation.

D.M. ADAMS, Lightnin, and

J.H. LAMMERS, Aker Kvaerner E&C

Most pressure oxidation operations introduce oxygen through a large multi-use nozzle in the vapour space of the autoclave. Sparge pipes extend inside the vessel to the point of discharge below the compartment agitator. However, two zinc pressure leach operations have successfully used a bottom entry oxygen sparge system for a number of years. Recently, the concept has been used in a whole ore gold pressure oxidation operation as well as a copper pressure leach plant. Both of the latter operations also introduce cooling water for temperature control through the same sparge pipe. This paper addresses the benefits of the bottom entry system with regards to autoclave performance and the positive effects on agitator performance.

Paper 4B.2 — 10:55

Slurry Dispersion Angle at Choke Outlet in Pressure Letdown with Multi-Phase Flows.

C.C. SMITH, Brigham Young University,

H. PIETERSE, Pieterse Consulting Inc.,

M. LUQUE, J. ROBISON and S. CHIPMAN, Caldera Engineering

Phase change during pressure let down releases significant amounts of energy that must be controlled to avoid undue wear and tear on equipment. Hydrometallurgical slurries require flow control equipment (valves and tanks) made of expensive materials to withstand the abrasive, and often corrosive nature of the slurry. Presented is a model of the nozzle of a let-down choke which is designed to control the flow pattern that exits the choke (valve) as it enters the receiving tank. This model can be used to design chokes which allow for the dissipation of the energy released during pressure let down by spreading the stream over a significant portion of the slurry pool in the receiving tank to minimize wear on equipment near the nozzle exit and the bottom of the slurry tank. An example uses the model to choose the nozzle expansion (area) ratio to select the outlet slurry velocity and slurry expansion angle.

Paper 4B.3 — 11:20

Increasing the Capacity of New and Existing Exothermic Autoclave Circuits.

G.M. DUNN, Hydromet Pty Ltd.

H.W. SCRIBA.

Pressure leach autoclave circuits are employed in the leaching of ores, concentrates, mattes, alloys and intermediates for the recovery of metals into solution. Once the metals are extracted into solution the value metals can be recovered by hydrometallurgical means such as by purification followed by electrowinning, hydrogen reduction, pyrohydrolysis, crystallisation and other unit operations. In many of these integrated flowsheets the pressure leach step is pivotal to the recovery of the value metals from the host material. In an expansion or a new project, a large component of the capital cost can be associated with the pressure leach step and process engineers frequently look for opportunities to mitigate costs. This paper examines a new route for increasing the capacity of an exothermic pressure oxidation leach for sulphide concentrates, sulphide and phosphide mattes and alloys.

TUESDAY, OCTOBER 26, 2004, P.M.

SESSION 5A - HIGH-TEMPERATURE MEASUREMENTS AND THERMODYNAMICS I

Session Chair: A. ALFANTAZI, University of BC, Canada
Room: Salon B

Paper 5A.1 — 14:00

Experimental Study of the Solubilities of Aluminum Oxy-hydroxo Phases to 300 °C.
D.A. PALMER, P. BÉNÉZETH and D.J. WESOLOWSKI, Oak Ridge National Laboratory
S. HILIC, AND-IT & Regulation Expertise for the Life Science Industry

The equilibrium solubilities of gibbsite, $\text{Al}(\text{OH})_3$, and boehmite, AlOOH , have been extensively studied in our laboratory from 30 to 90 °C, and 100 to 300 °C, respectively, to ionic strengths of $5 \text{ mol} \cdot \text{kg}^{-1}$ (NaCl) over a wide range of pH. The resulting solubility constants have been treated with consistent empirical models that incorporate appropriate experimental data taken from the literature. These studies were initiated in part to understand and quantify scale formation in geothermal fields as a result of fluid re-injection from electric power generating plants. Most of the experimental results were obtained through application of a unique potentiometric method that allows for rapid, precise measurement of the *in situ* pH and allows the approach to equilibrium to be monitored from under- and super-saturation. The experimental program was extended to deal with more basic solutions (e.g., five molal NaOH), in which the dissolution of boehmite was measured for application to the refining of bauxite by the Bayer process. More recent results in strong caustic solutions at temperatures below 90 °C were obtained to investigate the dissolution of aluminum-containing sludge from holding tanks at the Savannah River waste reprocessing site. In this case the solubility enhancing effects (kinetic and equilibrium) of triethanolamine were also established and quantified. Finally, potentiometric solubility experiments were conducted (50 °C in $0.1 \text{ mol} \cdot \text{kg}^{-1}$, NaCl) on aluminum alloy sheets to identify mainly the nature of the aluminum oxide/hydroxide surface layer formed under these conditions.

Paper 5A.2 — 14:25

Acid-Base Ionization and Metal Complexation under Hydrothermal Conditions by UV-Visible and Raman Spectroscopy.
L.N. TREVANI, P. TREMAINE and W.W. RUDOLPH, University of Guelph

UV-visible spectroscopy, Raman spectroscopy, and vibrating tube densimetry have been used in our laboratory to determine equilibrium constants of aqueous species at temperatures as high as 325 °C. The UV-visible and Raman spectra of solutions containing metal complexes and ionized acids or bases can be used to yield formation constants by applying curve fitting methods to resolve the contribution of each species. Colorimetric pH indicators identified by Johnston and coworkers at the University of Texas (Austin) provide an alternative means of determining ionization constants and the pH of complex mixtures. High precision density measurements over the same range yield standard partial molar volumes, which are key parameters for "equations of state" for aqueous species. Examples are given for the copper-ammonia system, simple carboxylic acids, and the iron-sulphate system.

Paper 5A.3 — 14:50

In Situ pH Monitoring of High Concentration Acidified Geothermal Brines and Acidic Sulphate Solutions at Elevated Temperatures.

S.N. LVOV, X.Y. ZHOU, S.M. ULYANOV and Z.F. Zhou, Pennsylvania State University,
Z. JANKOVIC and V.G. PAPANGELAKIS, University of Toronto

Recently developed flow-through electrochemical techniques (1,2) were employed for measuring pH in high concentration aqueous solutions and geothermal brines. The pH values of (1) $5.0 \text{ mol} \cdot \text{kg}^{-1} \text{ NaCl} + x \text{ mol} \cdot \text{kg}^{-1} \text{ HCl}$, (2) $3.0 \text{ mol} \cdot \text{kg}^{-1} \text{ NaCl} + 0.5 \text{ mol} \cdot \text{kg}^{-1} \text{ KCl} + 1.0 \text{ mol} \cdot \text{kg}^{-1} \text{ CaCl}_2 + x \text{ mol} \cdot \text{kg}^{-1} \text{ HCl}$ ($x < 0.1 \text{ mol} \cdot \text{kg}^{-1}$), and (3) mixtures of $\text{Al}_2(\text{SO}_4)_3\text{-H}_2\text{SO}_4\text{-H}_2\text{O}$, $\text{MgSO}_4\text{-H}_2\text{SO}_4\text{-H}_2\text{O}$, and $\text{NiSO}_4\text{-H}_2\text{SO}_4\text{-H}_2\text{O}$ solutions have been measured at temperatures from 100 to 280°C. The obtained experimental data and their thermodynamic analysis demonstrated that the electrochemical system can be used for on-line pH monitoring of high concentration aqueous solutions with an accuracy of $\pm 0.1\text{--}0.3$ pH units over a wide range of temperatures. Greater deviation exists in real laterite leach solution measurements due to in situ precipitation of Na-alunite. The system is excellent for research and process development work. Further work is needed to ensure robust operation under pressure leach conditions of laterites.

Coffee Break 15:15 - 15:30

SESSION 5B - MATERIALS OF CONSTRUCTION I

Session Chair: M. BUARZAIGA, Falconbridge TC, Canada
Room: Salon C

Paper 5B.1 — 14:00

Design Fundamentals for Hydrometallurgical Pressure Vessel Refractory Linings.

A. KONING and P. LAUZON, Hatch

Pressure hydrometallurgy operations require vessels to be lined with an impermeable membrane for corrosion protection and one or more courses of refractory or ceramic brick. Examples of unit operations that utilize composite lining systems include pressure oxidation autoclaves, sulphide precipitation autoclaves, chloride leach reactors, flash vessels, cyclone separators, and direct contact condensers (heater vessels and quench vessels). The refractory lining must satisfy multiple requirements: it must thermally insulate the membrane from process fluid, be structurally stable, provide erosion resistance, be chemically compatible with process fluid, and provide an economic service life. New hydrometallurgical processes are pushing the pressure, and temperature, with each new generation of plants. A fundamental understanding of all factors affecting the mechanical stability of the lining system is essential as lining designs move further away from the industry's experience base. The impact of irreversible chemical swell, operating factors, design factors, and installation factors are presented based on modifications to the traditional one-dimensional mechanical stress model. A new stability criterion – that the brick lining remain in contact with the steel shell membrane – is presented. This new stability criterion imposes new restrictions on the lining design. The effect of geometry and adding additional degrees of freedom to the analysis is explored using two-dimensional and three-dimensional Finite Element Analysis (FEA). The effect of these additional degrees of freedom on the results of the one-dimensional model are discussed.

Paper 5B.2 — 14:25

A Comparison of Refractory Lined and Metal-clad Process Vessels for Specific Operating Conditions.

L. ZUNTI and M. PEARSON, Hatch

Pressure vessels in hydrometallurgy and many chemical process industries throughout the world require the use of specific lining systems to protect the parent vessel materials from corrosion and/or abrasion. Typically, these lining systems are required for processes that are corrosive and operate at elevated temperatures. Two basic lining system types are: 1) refractory linings in conjunction with an impervious corrosion resistant membrane, and 2) metal-clad vessels consisting of titanium or other corrosion resistant metal bonded to a carbon steel shell. Operating conditions play a key role in selecting the type of lining system to be utilized for a specific application. Lining systems in turn have an impact on the process vessel size, which ultimately affects the capital cost of the vessel. The capital costs for each type of lining system must be considered for each application, as well as maintenance concerns, quality control, and delivery. It is clear that operating conditions play a key role in the final selection of lining systems utilized for specific projects based on initial capital cost, life-cycle maintenance costs, and overall service life. This paper will examine the fundamentals of each lining system, their inherent technical strengths and weaknesses, and present relative cost comparisons for each lining system as they apply to specific operating conditions and vessel sizes.

Paper 5B.3 — 14:50

Impact of Mortar Properties on the Integrity of an Autoclave Brick Lining System.

G. HUDON, Y. PÉPIN and J-F. TURGEON, Rio Tinto Iron & Titanium

The high temperature hydrochloric acid leaching of titanium slag in the QIT upgraded slag process (UGS) creates a harsh environment for materials of construction. Because of their excellent chemical and heat resistance, furan mortars are a key component in the lining system of the UGS pressure leach reactors. Similar products coming from different manufacturers have however shown unequal performances in service. In some cases, cracking of the brick lining has occurred during curing of the mortar. This study was initiated to investigate the impact of the curing conditions on the properties of carbon filled furan mortars. Laboratory tests were conducted to assess the effects of temperature and exposure to water and HCl solutions on mortar shrinkage and mechanical properties (tensile, compressive and bond strength). Three commercial furan mortars that have been used in the UGS leach reactors were thus characterized. Mortar properties were found to vary significantly among the products tested. Moreover, the curing conditions were shown to have a significant impact on mortar shrinkage and mechanical strength. The experimental findings provide explanations for the unequal performances of the mortars in service and for the problems of brick lining cracking that occurred in the UGS leach reactors. This study demonstrates that excessive mortar shrinkage compromises the integrity of a brick lining system.

Coffee Break 15:15 - 15:30

SESSION 6A - HIGH-TEMPERATURE MEASUREMENTS AND THERMODYNAMICS II

Session Chair: P. TREMAINE, University of Guelph, Canada
Room: Salon B

Paper 6A.1 — 15:30

Ammonia Pressure Hydrometallurgy: Estimation of High Temperature Stability Constants for Metal Ammine Complexes.
K. OSSEO-ASARE and S.W. ASIHENE, Penn State University

The thermodynamic data necessary for the quantitative treatment of Metal-NH₃-H₂O systems at elevated temperatures are lacking in the literature. As a result discussions of the chemical basis of high temperature processes, such as leaching and hydrogen reduction, have tended to rely on low temperature thermodynamic data. In this paper a critical review is presented of the available high temperature data for metal ammine formation in aqueous systems, and methods are suggested to enable extrapolation of elevated temperature stability constants from low temperature thermodynamic data. Predictions of the Van't Hoff Isobar, the simple cation Correspondence Principle (whereby metal ammine complexes are treated as simple cations) and Helgeson's Electrostatic-Nonelectrostatic Model are compared with the available high temperature stability constants for nickel amines. Up to 80 °C all three estimation methods yield results which are in agreement with experimental data. However, serious deviations occur at higher temperatures; the various models overestimate the stability constants in the order Van't Hoff Isobar > electrostatic-nonelectrostatic model > simple cation Correspondence Principle. Using the experimental nickel data as a basis, a method is presented which permits reasonable estimates to be made of the stability constants of metal ammine complexes for temperatures above 80 °C.

Paper 6A.2 — 15:55

The Thermodynamics of Titanium Corrosion in Acidic Systems.

J. VAUGHAN and A. ALFANTAZI, University of British Columbia

Recently, titanium and its alloys have been the materials of choice for the liner and internal components of autoclaves used to extract Ni and Co from laterite ores. There have been many corrosion incidents ranging from localized attacks due to scale formation or welding, to damage caused by pockets of concentrated acid. Most of the literature focuses on methods to avoid the corrosion altogether. Little has been published to increase awareness of the chemistry at these conditions. For this paper, a literature review of the thermodynamics of titanium was undertaken to decipher the slew of information available. E-pH diagrams were constructed from select data and compared with the results of electrochemical experiments. Since nickel laterite leaching takes place in sulphuric acid, and in many cases chloride concentrations are very high, the Ti-H₂O, Ti-Cl⁻-H₂O and Ti-SO₄⁻-H₂O systems were considered.

Paper 6A.3 — 16:20

Acidity Estimation at Temperature by Conductivity in Pressure Acid Leach Solutions.

M. HUANG and V.G. PAPANGELAKIS, University of Toronto

A conductivity cell, developed previously, was employed in an effort to investigate the inter-relation between electrical conductivity and acidity of sulphuric acid – metal sulphate solutions at high temperatures. The measured solutions include the H₂SO₄-H₂O, MgSO₄-H₂O, H₂SO₄-MgSO₄-H₂O, H₂SO₄-MgSO₄-Al₂(SO₄)₃-H₂O solutions. All solutions reported in this paper were measured from 15 to 250 °C at equilibrium vapour pressure. The maximum measured concentrations of H₂SO₄, MgSO₄ and Al₂(SO₄)₃ were 0.45 m, 0.30 m and 0.10 m, respectively. It was found that, from 15 to 250°C, there is a linear relationship between H₂SO₄ concentration and electrical conductivities for acid concentration up to 0.45 m. At 250 °C and constant H₂SO₄ concentration, the solution conductivity drops with the increase of MgSO₄ concentration. It is suggested that this drop is caused by a decrease in the concentration of H⁺. Finally, an empirical model was developed based on the available measurements. This model has been tested against real laterite leach solutions where sulphuric acid and metal sulphates are dominant electrolytes. The average difference between the estimate acid concentration from this model and the one from titration was less than 5%.

SESSION 6B - MATERIALS OF CONSTRUCTION II

Session Chair: R. KALANCHEY, Dynatec, Canada
Room: Salon C

Paper 6B.1 — 15:30

Ball Valves with Nanostructured Titanium Oxide Coatings for High-Pressure Acid-Leach Service: Development to Application.

J. WILLIAMS, Williams: Mogas Industries, Inc.,

G.E. KIM, Perpetual Technologies, Inc., and

J. WALKER, F.W. Gartner Thermal Spraying Co.

Since May of 2000, Mogas Industries, in partnership with F.W. Gartner Thermal Spraying Co. and using the services of Perpetual Technologies, has invested time, energy, and resources towards developing novel ball valves for high-pressure acid-leach (HPAL) service. In addition to developing new valve designs, an aggressive coatings development effort has been undertaken to improve the protective quality of the coatings so as to enhance the performance and extend the life of valves in severe HPAL conditions. In 2001, Mogas Industries qualified and applied a nanostructured titanium oxide (n-TiO₂) coating for gold and nickel/cobalt HPAL services. The n-TiO₂ coating provided dramatically superior protection against abrasive and erosive wear, fared very well against high-pressure autoclave corrosion testing, and has performed well in HPAL service.

Paper 6B.2 — 15:55

Severe Service Ball Valve Design Analysis Utilizing a Modified Kepner-Tregoe Method.

P. LAUZON, L. NIGHTINGALE-MERCER, M. PEARSON, Hatch, and
J. IRVINE, ABI Consultants

Severe service ball valve performance has a direct impact on the mechanical availability of high pressure oxidation and leach plants. The primary function of these valves is to isolate the autoclave for maintenance. Valve failure has historically contributed significantly to the maintenance downtime of these process plants. An understanding of the root causes of valve failures is the first step in increasing the reliability of the severe service ball valves. A rigorous failure analysis practice has been developed by Hatch for investigating refractory failures in furnaces and kilns based on Kepner-Tregoe methodology. The method has also been modified for valve failure analysis. From a root-cause failure tree analysis of each of the valve components, a list of all potential valve failure mechanisms was developed for ranking and evaluation, along with recommendations for further valve development. The purpose of this paper is to present the reader with the modified Kepner-Tregoe method and examples of this analysis.

Paper 6B.3 — 16:20

Brick Linings in High Pressure Acid Leaching (HPAL) Vessels.

H. FELDHEISER, DSB-Säurebau GmbH

Acid resistant brick linings are state of the art for anti-corrosion linings in tanks for high pressure acid leaching (HPAL) treatment. The design of these brick linings has to take into account the almost non-elastic behaviour of the brick lining inside a relatively highly elastic steel vessel. There must be tools and data available to determine for the steel vessels the thermal growth plus the pressure expansion and for the brick lining the linear thermal expansion and the chemical swell. Thermal shocks and pressure drops are not regular operating events but their negative influence on the lifetime of a brick lining has to be a part of the engineering study. Finite Element (FE) models as well as a lot of research and development on brick lining materials is the only way to find a path through the complex engineering work for pressure vessels.

Paper 6B.4 — 16:45

Reliable and Economic Use of Masonry Lining Technology for Hydrometallurgical Applications.

T.E. CRANDALL, R.J. WAKEFIELD, R.E. ALIASSO, R.J. STORMS and J. VAN DER MERWE, Stebbins Engineering & Manufacturing Company

New developments in ore processes have taken processes from mainly high temperature smelting to new pressure and atmospheric leaching. These developments require owners and consulting engineers to evaluate different types of reliable materials of construction, such as masonry and membrane systems. Masonry and membrane systems have been reliably used for over 100 years in many commercial process industries to resist corrosion and abrasion in process vessels. The focus of this technical paper is to review a reliable material of construction option for corrosion resistance in atmospheric or pressurized process vessels. The masonry and membrane system is a composite of three components – structural envelope, corrosion barrier to protect the structural envelope and the masonry layer to protect the corrosion barrier. Many applications exist for reliable and economic use of membrane and masonry systems. Several new commercial process applications have already selected masonry and membrane systems for their corrosion protection. The selection in these new processes is a result of owner and consultant engineer evaluations based on comfort, proven reliability and sound economics. Several of these new commercial applications will be discussed in detail. Owners and consultant engineers not yet aware of the background, history and acceptance of masonry and membrane systems will gain insight on a new range of materials of construction for corrosion and abrasion resistance in the mining processing industry.

WEDNESDAY, OCTOBER 27, 2004, A.M.

SESSION 7A - PRESSURE HYDROMETALLURGY OF REFRACTORY GOLD I

Session Chair: J. MCMULLEN, Barrick Gold, Canada

Room: Salon B

Paper 7A.1 — 8:30

Reducing the Energy Consumption of the Barrick Goldstrike Autoclave.

S. CASHIN, D. KASHUBA and W. ESPLIN, Barrick Gold

Barrick has had 6 autoclaves in operation at the Goldstrike Mine, North of Carlin Nevada, since 1993. The 6 autoclaves operate in parallel, processing slurry of gold ore. The gold in the ore is predominantly associated with, and encapsulated by, sulphide minerals. It is necessary to oxidize at least 90% of the sulphide mineral before the gold can be concentrated using a cyanide/carbon-in-leach (CIL) circuit. Sulphide oxidation chemical reactions occurring in the autoclaves are exothermic; however, the sulphide content in the ore is not high enough to operate the autoclaves in an auto-thermal mode. Heat from steam is added to the first 2 of 5 autoclave compartments to initiate the sulphide oxidation reactions. The steam used to heat the autoclaves is supplied by three boilers, which are fuelled with propane gas. The completion of a few operations and maintenance projects in the last 2 years has resulted in a 50% reduction in propane energy requirements. The autoclave operating temperatures were minimized. Excess oxygen addition was held as low as possible. The material of construction for the compartment walls inside the autoclaves was changed from brick to Titanium metal. And, the speed of the autoclave slurry agitators was increased. All of these activities had an impact on the propane energy requirements of the autoclaves. Presented below is a description of the autoclave systems, and a discussion of how the energy needs were reduced, resulting in significant operating cost savings.

Paper 7A.2 — 8:55

High Temperature POX of Precious/Base Metal Concentrates from Newmont's Phoenix Project, Using Controlled Precipitation of Sulphate Species to Enhance Silver Recovery.

G.L. SIMMONS and J.C. GATHJE, Newmont Mining Corporation

Newmont acquired the Phoenix Project through the acquisition of Battle Mountain Gold in 2000. Shortly after the acquisition, Newmont Metallurgical Services (NMS) and Nevada Operations began development work on a process to Pressure Oxidize (POX) precious metal rich copper concentrates from Phoenix using the Lone Tree POX facilities at Valmy, Nevada. The high precious metals content of the Phoenix concentrate – Au up to ~300 g/t and especially Ag, up to ~1700 g/t – led Newmont to the development of a High Temperature POX process that achieves high cyanide leach recovery of Au and Ag, from the POX solids, without the requirement for a messy and expensive “lime boil” or sulphate destruction process. Base metals content in Phoenix concentrates range from: 15-25 % for Cu, 1-10 % for Zn and 0-2 % for Pb. Excellent solution extraction of Cu and Zn is achieved while Pb is precipitated in the POX residue. In addition to achieving high Au and Ag recovery this process produces very clean POX discharge liquors that require minimum effort to remove contaminant metals and POX solids that have superior settling and filtration characteristics. All of these factors contribute to lower capital and operating costs for the overall process. This paper describes the laboratory and POX pilot plant developments for this process.

Paper 7A.3 — 9:20

Safety Protocols for Campbell Mine Autoclave Shutdowns.

M. TIMMINS, Placer Dome Campbell Mine

Campbell Mine operates a 100 tpd gold autoclave that treats sulphide concentrates at 305 psi and 200 °C. Autoclave feed consists of fresh pretreated concentrate, an acidic oxide slurry recycle as well as other high arsenic sources. The brickwork that is used to construct the five compartments of the vessel must be inspected and repaired periodically. Shutdowns are scheduled to inspect this brickwork and at times replace entire sections of the brick walls or lining. It also provides an opportunity to repair or replace other internal features such as agitator blades or oxygen spargers. Mechanics and contractors are scheduled around the clock to perform the maintenance requirements. There are many safety considerations and precautions that must be taken in order to protect the well-being of the workers and minimize damage to sensitive pieces of equipment. Heat, arsenic and lead contamination and confined space issues are just a few of the hazards involved. The following paper will describe how these safety concerns are addressed and will serve as a guide for future autoclave shutdowns.

Paper 7A.4 — 9:45

Pilot Plant Evaluation of a Hybrid Biological Leaching-Pressure Oxidation Process for Auriferous Arsenopyrite/Pyrite Feedstocks.

I. DYMOV, C.J. FERRON, SGS Lakefield, and

W. PHILLIPS, Kinross Corporation

A process has been evaluated, on a continuous pilot plant scale, to treat refractory gold arsenopyrite/pyrite concentrates. The process consisted of biological leaching to partially oxidise sulphides, followed by pressure leaching for complete sulphide oxidation and arsenic precipitation, followed by liquor neutralisation and cyanidation of the autoclave residue for gold recovery. This paper provides a brief description of the treatment flowsheet and discusses some of the main parameters and results of the process. During integrated pilot plant campaigns, it was confirmed that acceptable sulphide oxidation and gold extraction of 96-98% could be achieved by a two-stage oxidation with BIOX[®] (4 days total retention time) followed by POX (40 minutes retention time at 220 °C). Only partial arsenic precipitation was achieved in the autoclave, followed by complete precipitation by neutralization under atmospheric conditions. The overall process could be significantly simplified, and capital and operating costs reduced, with a flowsheet modification allowing all of the BIOX[®] liquor to bypass the autoclave directly to the neutralization circuit.

Coffee Break 10:10 - 10:30

SESSION 7B - PROCESS DEVELOPMENT STUDIES I

Session Chair: M. DRY, Consultant, Canada
Room: Salon C

Paper 7B.1 — 8:30

Hydrothermal Synthesis of Functional Inorganic Materials from Aluminum Recycling Wastes.

J. SHIBATA, N. MURAYAMA and H. YAMAMOTO, Kansai University

As one of effective usages of wastes discharged in the non-ferrous metal industry, the syntheses of $\text{AlPO}_4\text{-5}$ and hydrotalcite were carried out using aluminium dross as a raw material. $\text{AlPO}_4\text{-5}$ is an aluminium phosphate condensate with properties of zeolitic materials and large pore size. Hydrotalcite is an inorganic anion exchanger and has a layered structure of complex hydroxide. They were synthesized in an autoclave at high temperature and pressure. Various physical properties such as crystal structure, surface texture, and specific surface area were measured. $\text{AlPO}_4\text{-5}$ can be synthesized from aluminium dross under hydrothermal conditions at 180°C-200 °C, and 3 h. At the same time, $\text{AlPO}_4\text{-34}$ and non-porous AlPO_4 were also formed as by-products. The crystals of $\text{AlPO}_4\text{-5}$ have a hexagonal structure. It is desirable to heat the reaction product at around 550 °C to remove tri-ethyl amine. The specific surface areas of the reaction product before and after heat treatment at 550 °C were 17.9 m²/g and 358 m²/g, respectively. Hydrotalcite can be obtained by a co-precipitation method from the aluminium dross and waste MgCl_2 solution discharged in the aluminium regeneration process. Small amounts of SiO_2 , Al_2O_3 , and Fe_2O_3 remain in the reaction product as an impurity component. The interlayer distance of the obtained reaction product is about 0.3 nm. Hydrotalcite changes to Mg-Al oxide during calcination at 500 °C for 3h, and then hydrotalcite is formed again by a rehydration operation. The physical properties of the reaction products obtained from the wastes were very similar to those from fresh reagents.

Paper 7B.2 — 8:55

Production of Thorium Concentrate from Egyptian Monazite.

A.M. ABDEL-REHIM, Alexandria University

The present work reports a study of production of thorium concentrate from Egyptian monazite by decomposition of ammonium carbonate solutions containing ammonium thorium and uranyl carbonate complexes. These solutions were obtained from the processing of monazite by alkaline leaching in ball-mill autoclaves, followed by selective separation of thorium and uranium from lanthanides by autoclave leaching of the hydroxide cake obtained with ammonium carbonate – bicarbonate solutions. The separation of thorium and uranium in the form of thorium concentrate from their carbonate complexes was achieved by different methods, including neutralization of solutions at pH = 6.7 by acid, addition of alkali at pH = 11.5, heating of solution at 100 °C and processing of carbonate solution with steam under pressure in steel autoclaves. Processing of solutions of thorium and uranyl carbonate complexes in an autoclave with steam causes their decomposition. Complete recovery of thorium 99.8% and 98.4% uranium recovery in thorium concentrate was attained at 120 °C and steam pressure 2 atm, within 10 min. Meanwhile, the recovery of lanthanides was low and did not exceed 1.1%. The thorium concentrate produced contains 67.82% Th, 4.6% U and 3.14% REE. All methods achieve complete separation of thorium and uranium from their carbonate solutions, but the first two methods are unfavourable for the industry due to the high cost of reagents and their inability to regenerate the ammonium carbonate. The decomposition of ammonium thorium and uranyl carbonate complexes with steam under pressure is recommended for industrial application for its simplicity and the ability to regenerate the ammonium carbonate.

Paper 7B.3 — 9:20

Pressure Acid Leaching of Non-ferrous Smelter Slags for the Recovery of their Base Metal Values.

W. CURLOOK, University of Toronto,

V.G. PAPANGELAKIS and M. BAGHALHA, University of Toronto

A pressure acid leaching process has been developed for the extraction of metal values, nickel, copper, cobalt and zinc, from base metal smelter slags. The basic concept contemplates cooling the smelter slags through solidification sufficiently slowly to induce a good measure of crystallization; and subsequently, after fine grinding, to pressure leach such slags at around 250 °C while applying oxygen overpressure, utilizing smelter byproduct sulphuric acid. High levels of extraction of the slags' metal values are thereby achieved. It has been shown that if the same slags are quenched thereby producing an amorphous structure, the resulting metal extractions are very low. The process is applicable to smelting furnace throwaway/discard/dump slags, as well as to in-process converter slags. By extracting the bulk of the residual base metal values in the throwaway/discard/dump slags, the resulting leached residues are rendered virtually benign vis-à-vis the external environment.

Paper 7B.4 — 9:45

Features of High-temperature Oxidation of Non-ferrous Metal Sulphides and Pyrite in Acidic Sulphate Solutions under Oxygen Pressure Conditions.

S.S. NABOICHENKO and K.N. BOLATBAEV, The Ural State Technical University

The initial rates of dissolution of pyrite, chalcocite, sphalerite and galena in acidic sulphate solutions under oxygen pressure were measured in the range of $P_{O_2} = 0.2-1$ MPa, $130-180$ °C, $[H_2SO_4]_0 = 0.2-0.7$ M, and in the presence of surface-active agents and agitation producing $Re = 4-16 \times 10^3$. Reaction orders, activation energies, and values of reaction constants for these oxidation reactions were established. The reactions also were shown to be diffusion-controlled; and the rates demonstrate a linear dependence on the surface area of the sulphides. A surface reaction model, in which oxygen is transported through a solid layer of surface products, has been advanced. The initial stage of high-temperature interaction at $pH < 7$ is suggested to involve a chemical activation of the sulphide surface and is described by the following general reaction:



The subsequent oxidation of surface complex HS^- to S^0 or SO_4^{2-} occurs with the assistance of Fe(III), Cu(II) ions depending on temperature and the redox potential of the medium, which are defined by oxygen pressure and composition of the reaction mixture, especially concentrations of H_2SO_4 and metals with variable valence. General process kinetics are complicated due to the formation of by-products on the basis of Fe^{3+} , Pb^{2+} , Cu^+ ions, which displace the kinetics into the diffusion region.

Coffee Break 10:10 - 10:30

SESSION 8A - PRESSURE HYDROMETALLURGY OF REFRACTORY GOLD II AND ZINC I

Session Chair: G. DEMOPOULOS, McGill University, Canada

Room: Salon B

Paper 8A.1 — 10:30

Process Modifications to the São Bento Concentrator of Eldorado Gold.

L. SILVA, R. GUIMARÃES, São Bento Mineração S.A., and

J. MILBOURNE, AMEC Mining & Metals Consulting

The São Bento concentrator was built by Gencor in 1985 to treat refractory gold-bearing flotation concentrate. The plant was the first commercial application of pressure oxidation for refractory gold ore following about a year after the first use of pressure oxidation on a whole ore at Homestake's McLaughlin operation in California. Ore from the São Bento mine is remarkably consistent in gold grade and arsenic and sulphide concentration as is typical in banded iron formations (BIF) (1). Gold content is approximately 9 g/t with approximately 30 – 40% free milling. The balance is associated with arsenopyrite and pyrrhotite. An expansion of both process facility and mine was undertaken between 1996 and 1998, raising the treatment capacity from 32,000 t/mo to 45,000 t/mo.

Paper 8A.2 — 10:55

Experiences with Zinc Pressure Leaching of 100% Red Dog Concentrate at Teck Cominco.

C. D'ODORICO, Teck Cominco Limited

The zinc pressure leaching (ZPL) plant at Teck Cominco's Trail Operations was started up in 1981 and represented the world's first commercialization of the technology. From the outset, the process was designed to operate using iron-rich Sullivan Mine zinc concentrate. When the Sullivan Mine reached the end of its life in 2002, the ZPL plant underwent a significant process change with the treatment of 100% Red Dog zinc concentrate. Through careful improvements to existing plant processes, controlled changes to plant operating parameters and a rapid adjustment to the modified process by operations personnel, the plant has been successfully converted to processing a feed of 100% Red Dog zinc concentrate. This paper discusses the challenges faced during the conversion and describes the modifications to plant operating procedures, as well as changes to plant processes.

Paper 8A.3 — 11:20

Commercialization of the Dynatec Zinc Pressure Leach Process at Kazakhmys.

S. SADYKOV, Kazakhmys Corporation,

E. McCONAGHY, J. STIKSMA, K. BUBAN, J. OFSTIE and R. KALANCHEY, Dynatec Corporation

The Kazakhmys zinc refinery in Balkhash, Kazakhstan, which employs the Dynatec Zinc Pressure Leach Process, officially launched autoclave operations on December 27, 2003. The Kazakhmys plant is the world's first greenfields zinc refinery based on Dynatec's two-stage pressure leach technology. Production of zinc was realized on February 9, 2004, when the first cathode was stripped. Currently the plant start-up is ongoing, with progression to two-stage zinc pressure leach operation and ramp-up to full design capacity continuing through 2004.

SESSION 8B - AUTOCLAVE DESIGN III

Session Chair: J. BUDAC, Sherritt International, Canada
Room: Salon C

Paper 8B.1 — 10:30

Special Design Considerations for Pressure Hydrometallurgy Pilot Plants.
P. MARTIN, Zeton Inc.

Pilot plants for pressure hydrometallurgy represent special design and project execution challenges unique to the industry and to the pilot- and demonstration-scale. Pilot plant projects have different objectives and shorter timescales than commercial plant projects. Design considerations and strategies, and project execution methods that meet the special needs of pilot plant projects, are discussed. Specific guidance is given for the selection of appropriate pressure and temperature design points for pressure hydrometallurgical process equipment, and for the selection of materials of construction for pilot-scale equipment. The balance between the need for process similarity with future commercial operations and the realities of pilot-scale equipment is also discussed.

Paper 8B.2 — 10:55

Self-cleaning Fluidized Bed Slurry Heat Exchangers: Processing of Laterite Ore Slurries for the Extraction of Nickel and Cobalt in High-pressure Acid Leach (HPAL) Plants.

D. G. KLAREN, E.F. de BOER, Klaren BV, and
B. CROSSLEY, Met Chem Cons.

Self-cleaning fluidized bed heat exchangers have demonstrated unparalleled performance internationally for the implementation of indirect heat transfer in severely fouling fluid environments. The ingenious configuration of recirculated cleaning particles through the tubes of vertical shell and tube heat exchangers provides the ability to solve any problems that arise as a consequence of tube-side fouling during indirect heat transfer to process fluids or slurries. In most cases, the cleaning particles consist of chopped metal wire, however other process-specific materials have been used. In addition to the mitigation of fouling effects, a number of other significant benefits for slurry heating are afforded by the mode of operation of the self-cleaning fluidized bed heat exchangers. Increased turbulence at the slurry/tube interface is imparted by the action of the fluidised particles, resulting in very high heat transfers at relatively low liquid velocities. Furthermore, given the thixotropic nature of most process slurries, the action of the fluidised particles imparts additional shear to the process slurry, resulting in shear thinning effects with a consequent reduction in pressure drop and enhancement of heat transfer across the heat exchanger. The benefits provided by the self-cleaning fluidized bed heat exchanger make it the ideal option for the indirect heating of laterite nickel slurry for High-Pressure-Acid-Leach (HPAL) plants. This article explains the principle and application of the self-cleaning fluidized bed heat exchanger technology for the processing of laterite nickel and cobalt slurries in HPAL plants. It also presents an elegant method to revamp existing direct heated laterite nickel and cobalt processing plants into an indirect heated configuration employing self-cleaning fluidized bed heat exchangers.

WEDNESDAY, OCTOBER 27, 2004, P.M.

SESSION 9A - PRESSURE HYDROMETALLURGY OF ZINC II

Session Chair: J. DUTRIZAC, CANMET, Canada
Room: Salon B

Paper 9A.1 — 14:00

Autoclave Application for Zinc Leach Residue Treatment by Akita Zinc Co. Ltd.
Y. KUDO and H. ARIMA, Akita Zinc Company

In general, the iron content of zinc leach residue produced by sulphuric acid leaching from calcine is disposed as jarosite or goethite into a tailing dam. More recently, these iron residues have been treated pyrometallurgically to avoid waste disposal. The Iijima refinery selected a different zinc leach residue treatment process for iron control when it was established in 1971. The Iijima refinery is the only plant in the world that has processed a zinc leach residue by the combination of SO₂ pressure leaching and O₂ pressure hematite precipitation. The process enhances zinc recovery and recovers gold, silver, copper, lead and rare metals in saleable by-products. This paper focuses on the two stages of autoclave application in the zinc leach residue treatment process. The detailed flowsheet and recent advancements to improve the quality of by-products are discussed.

Paper 9A.2 — 14:25

The Hematite Process — New Concepts for Increased Throughput and Clean Hematite Production.
T.C. CHENG and G. DEMOPOULOS, McGill University

It is the object of this paper to discuss various options for improving the hematite process in terms of increased throughput hence lower capital cost and production of saleable quality hematite. With reference to increasing the throughput of the process, it has now been determined via the analysis of Akita Zinc Co's continuous industrial autoclave performance data that the process kinetics is limited by the rate of the hydrolytic precipitation reaction. By correlating the hydrolytic precipitation rate to supersaturation ratio, it is shown that accelerated kinetics can be achieved via the following measures: (1) *in-situ* neutralization with addition of zinc calcine; (2) elevation of temperature; and (3) dilution of feed; of which, the first one was retained for further evaluation. According to presented estimates, addition of calcine, amounting to only 2.6% of the total zinc calcine processed, can allow for shortening the retention time by at least 50%. Such drastic shortening of retention time may be either used to increase the throughput of a given autoclave circuit or alternatively to release autoclave capacity for clean hematite production. Hydrothermal treatment of the crude hematite product proved effective in producing clean hematite as according to laboratory tests, hence allowing the sulphur content of hematite to be reduced from around 4% to less than 1%. A conceptual flowsheet incorporating *in-situ* neutralization for accelerated kinetics with hydrothermal upgrading of crude hematite without sacrificing production capacity and with minimum upset of the plant's water balance is described.

Paper 9A.3 — 14:50

Mathematical Modelling of Pressure Leaching of Sulphide Zinc Concentrate.

E.M. VIGDORCHIK, E.E. ZHMARIN, Y.M. SHNEERSON, A.Y. LAPIN and V.M. SHPAER, Gipronickel Institute

This paper describes studies of zinc concentrate oxidizing pressure leaching using the method of mathematical modeling. The concept of a kinetic function was taken as a basis for mathematical modeling of the leaching process. The kinetic function describes dependence of the unreacted component share ω on the relative time x (the relative time is the ratio of the test duration and the time of complete dissolution). This kinetic function is determined in accordance with the results of batch laboratory tests. The laboratory tests have been carried out on zinc concentrate produced from Kazakhstan zinc-copper ores. Zinc concentrate consisted mainly of three types of minerals: sphalerite (70%), pyrite (13%) and chalcopyrite (10%). Oxidation reactions of sphalerite, pyrite and chalcopyrite have been considered as principal ones. The kinetic characteristics of the process (kinetic function, reaction order, activation energy, duration of complete dissolution of the concentrate components) have been calculated in the course of laboratory tests. The mathematical model was used for estimation of the main technological leaching process parameters (such as process rate, degree of metals extraction, autoclave capacity, heat balance, etc.). Mathematical modeling based on the kinetic function concept allows safe designing of metallurgical equipment and scaling the technology to industrial capacity.

Coffee Break 15:15 - 15:30

SESSION 9B - PROCESS DEVELOPMENT STUDIES II

Session Chair: M. COLLINS, Dynatec, Canada

Room: Salon C

Paper 9B.1 — 14:00

Applications of NSC Pressure Leaching.

C. ANDERSON, CAMP-Montana Tech

Industrially proven and applied nitrogen species catalyzed (i.e. NSC) acidic pressure leaching has been utilized to treat a number of ores, concentrates and recycled materials. This versatile technology offers many advantages including low temperatures, low pressures, low capital costs, low operating costs, faster throughput and less costly materials of construction than conventional pressure oxidation. This paper will elucidate the application of this technology for production of gold, silver, palladium, rhodium, zinc, germanium, gallium, cobalt, copper and nickel. Recycled material applications will also be elucidated. In addition, details of an application for treatment of a refractory chalcopyrite copper and gold bearing ore will be presented and pertinent economics discussed.

Paper 9B.2 — 14:25

Interactions of Ferriferous Copper-Nickel Mattes with Sulphuric Acid: Study of Chemistry and Kinetics.

M.I. KALASHNIKOVA, Y.M. SHNEERSON and M.V. KESKINOVA, Gipronickel Institute

Interaction between mattes having various compositions and solutions with a wide range of free acid and non-ferrous metals concentration have been studied in terms of chemistry, mechanism and kinetics. The solution acidity and oxidizers to reducers ratio in the matte-solution-gas phase system have been demonstrated to be the major factors determining the process behaviour. It was found that the types of copper and nickel secondary sulphides resulted from nonferrous metals precipitation from solution, depend on of the solution and initial matte composition. Based on these studies, technologies of matte leaching and nonferrous metal precipitation in the form of rich sulphide concentrates are developed.

Paper 9B.3 — 14:50

Recovery of Europium and Yttrium from Spent Fluorescent Lamps.

M.A. RABAH, Central Metallurgical R&D Institute (CMRDI)

This study aims to recover europium, yttrium metals, some valuable salts or oxides from the white powder (apatite) coating the inner surface of spent fluorescent lamps (SFLs). Pressure leaching using sulphuric/nitric acid successfully dissolved the metals of interest. Europium or yttrium metals were obtained by hydrogen reduction of their oxides. Addition of potassium thiocyanate converted the dissolved sulphates to thiocyanates. Selective separation of Eu and Y was then carried out by solvent extraction from the thiocyanate solution. The used solvent was trimethyl-benzylammonium chloride. N-tributylphosphate in 1 M nitric acid was used to strip the metal loaded in the organic phase. Results showed that the quality of the cleaned glass tubes of the SFL met the standard requirement for the lamp manufacture. The white powder (WP) is composed of calcium sulphate 35.2%, phosphate 61.5% and oxides of europium and yttrium 1.65%wt. respectively. Autoclave digestion of the WP for 4 h at 120 °C and 500 kPa in sulphuric acid/nitric acid mixture dissolved 96.4% of yttrium and 92.8% of europium. The conversion rate of sulphate to thiocyanate was favoured at low temperature. Stripping of Eu and Y from the loaded organic phase behaved in an opposite way. A metal separation factor of 9.4 was obtained. The recovered europium and yttrium salts were selectively separated using ethyl alcohol or dilute nitric acid. Yttrium was very soluble while europium was not. Europium and yttrium metals were obtained by hydrogen gas reduction at 850 °C and 1575 °C respectively.