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Method for the Preferential Leach of Value Metals From Sulphide Concentrates

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Abstract

A hydrometallurgical method for treating a sulphide concentrate 101 in a continuously operating oxidative leach reactor, the sulphide concentrate 101 comprising a blend of electronegative and electropositive elements, the method comprising:

submitting a slurry comprising the concentrate 101 in an aqueous stream containing at least water to an oxidative leach targeting the recovery of one or more electronegative elements over the more electropositive elements within the concentrate; and

adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit to preferentially leach one or more of the electronegative elements over the more electropositive elements within the concentrate; and

wherein the electronegative elements comprise at least one of copper, nickel, cobalt or zinc.

“METHOD FOR THE PREFERENTIAL LEACH OF VALUE METALS FROM SULPHIDE CONCENTRATES”

Field of the Invention

The present invention relates to a method for the preferential leach of value
5 metals from sulphide and alloy containing concentrates and relates
particularly, though not exclusively, to such a method for the processing of
polymetallic sulphide concentrates. The value metals typically include at
least one of copper, zinc, cobalt, nickel, selenium, tellurium, including the
precious metals (such as gold and silver) and platinum group metals (such
10 as platinum, palladium, ruthenium, rhodium, osmium and iridium).

Background to the Invention

Processing of polymetallic sulphide concentrates is conventionally
undertaken in a roaster which removes the sulphur and produces a calcine
15 product which is amenable to leaching. The roasting process generates
large volumes of SO_2/SO_3 off-gas which needs to be captured, usually in the
form of sulphuric acid. The cost of disposal of the sulphuric acid is
expensive and in addition, the roasting process can release toxic metals to
the environment such as mercury and thallium.

20 A competing process to roasting may be the total oxidative pressure leach
of the sulphide concentrates or the biological oxidation process using tank
or heap leaching. The total pressure oxidative leach, particularly for a high
pyrite containing concentrate is an expensive process and may not be
economic due to the high oxygen consumption and large volumes of acid
25 generated which need to be neutralised. The biological leaching processes,
both tank and heap leach, are known to be slow and often result in poor
value metals recovery.

Some sulphide minerals contain copper in the form of chalcopyrite and
cobalt in the form of carrolite, from which copper and cobalt recovery has

conventionally taken place in a roasting step. Again, competing processes to roasting may be a total oxidative pressure leach of the copper-cobalt sulphide concentrate or possibly a biological oxidation process. Another cobalt sulphide mineral could be cobalt linnaeite or cattierite intergrown with iron sulphide from which cobalt recovery conventionally would take place either by biological leaching or by pressure oxidative leaching.

For these sulphide minerals, presented as examples, total oxidative pressure leaching and biological leaching generally have unfavourable economics. For pressure oxidation, the unfavourable economics may relate to the large quantities of oxygen required to oxidise the gangue sulphides and after that to neutralise the sulphuric acid generated from the leach. For biological leaching, it is known that chalcopyrite can be passivated in a low-intensity leach process, and for that reason yields of the value metals can remain relatively low.

The Albion process is disclosed in US patent 5,993,635. One essential characteristic of the process is a sulphide concentrate milling step to a P80 of 20 microns or less, after which leaching of the sulphide mineral is performed under atmospheric conditions, i.e., at ambient pressure and a temperature between 60°C and the boiling point of the solution. It is characteristic of the Albion leaching process that it takes place using a solution containing sulphuric acid and trivalent iron and by feeding oxygen into the leaching stage. The sulphuric acid concentration of the solution, when leaching chalcopyrite, is in the region of 30 to 40 g/l. The iron used in leaching is derived largely from the pyrite contained in the sulphide mineral or other copper-iron-sulphide minerals that may be present. The value metals to be leached contain at least one of copper, nickel, cobalt or zinc. The acidic sulphate solution containing the dissolved value metal is next admitted to acid neutralisation and iron precipitation. Neutralisation and iron precipitation are carried out with limestone. The solution purification of the value metal-containing sulphate solution is performed by solvent extraction and some of the value metals may be recovered by electrowinning.

Another sulphide concentrate leaching process is disclosed by Dominion Mining in US patent 5,917,116 and is based on a fine grind of the copper sulphide concentrate to P80 of between 2 and 20 micron, with a low-temperature (below 100°C) pressure leach employing oxygen pressure at about 10 bar in the presence of chloride ions in an amount of from 2 to 10 g/l. The sulphuric acid concentration in the leach step is approximately 100 to 120 g/l.

A further sulphide concentrate leach method is disclosed by Outotec Oy in US patent application 20120103827 which relates to leaching bulk concentrates containing chalcopyrite. This process operates at atmospheric pressure conditions and at a temperature between 75°C and the boiling point of the solution. The concentrate leach process typically operates with a coarser grind with a P80 below 60 to 100 micron and the concentrate is leached in an aqueous solution with a sulphuric acid concentration between 20 to 90 g/l. The preferred embodiment of this invention is to regulate the acid concentration to between 40 to 70 g/l, and to subject the concentrate to a short purification milling before leaching in order to clean the mineral surfaces. This process relates typically to a chalcopyrite concentrate, which is a bulk concentrate including zinc sulphide.

A further concentrate leach method is disclosed in US patent application 2005/269208, which operates in atmospheric conditions and at a temperature of 50 to 120°C and where the concentrate is fed into leaching at a coarser degree of grinding (e.g. P80 below 106 microns). An essential feature of the process is the feed of pyrite into leaching. The ratio of chalcopyrite to pyrite is specified as being between 4:1 and 1:20. The method operates in conditions where the pyrite does not dissolve, and the redox is in the region of 350-520 mV vs. Ag/AgCl.

Yet a further electrochemically controlled leach method is disclosed by Orway Mineral Consultants in US patent US 9587290-B2 employing a metathesis mechanism. In this process the removal of radionuclides and/or iron from certain copper concentrates is taught.

- Most leach processes are conducted in a leaching system comprising multiple stirred reactors either as discrete tanks or compartments as in a multi-compartment autoclave. In these systems a terminal electrochemical potential is typically sought in the final reactor or compartment and once this potential is reached the objective is considered having been achieved. For example, in the extraction of copper from a copper sulphide mineral employing total oxidative pressure leaching, a terminal electrochemical potential of approximately 550 mV (Ag/AgCl) in the final reactor may be considered sufficient for adequate copper extraction from the concentrate.
- However, in this same example and at this high terminal electrochemical potential, excessive pyrite and other iron sulphides can oxidise, possibly resulting in excess quantities of iron and sulphuric acid in solution. In addition, the high redox potential can result in precious metals being co-extracted with copper. The outcome, whilst acceptable for copper, may be undesirable as valuable precious metals in the concentrate may report with the leached copper and report to anode slimes in the copper electrowinning process. Similarly, total or excessive oxidation of the iron and sulphur may be unnecessary in the quest to have an economic process for recovering copper.
- The present invention was developed to provide a process in which value metals, which are more electronegative, can be extracted preferentially from sulphide concentrates and alloys in a first leach step, whilst precious metals and lower value metals remain in the residue.

References to prior art in this specification are provided for illustrative purposes only and are not to be taken as an admission that such prior art is part of the common general knowledge in Australia or elsewhere.

Summary of the Invention

According to one aspect of the present invention there is provided a hydrometallurgical method for treating a sulphide concentrate in a continuously operating oxidative leach reactor, the sulphide concentrate

comprising a blend of electronegative and electropositive elements, the method comprising:

submitting a slurry comprising the concentrate in an aqueous stream containing at least water to an oxidative leach targeting the recovery of one or more electronegative elements over the more electropositive elements within the concentrate; and,

adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit to preferentially leach one or more of the electronegative elements over the more electropositive elements within the concentrate; and

wherein the electronegative elements comprise at least one of copper, nickel, cobalt or zinc.

Preferably the method also comprises adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit to preferentially leach one or more value metal sulphides over the oxidation or extraction of lower value metals or elements within the concentrate.

Typically, one or more of the electronegative and electropositive elements may be capable of being passivated in an oxidative leaching process. Advantageously the method also comprises adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit to minimise the passivation of one or more value metals in the concentrate to achieve economically high extractions of the one or more value metals.

Preferably a lixiviant is added to the concentrate to establish a lixiviant-concentrate blend either prior to, or during, the oxidative leach. Preferably the lixiviant-concentrate is continuously fed into the reactor vessel. The lixiviant may be one or more of a blend of mineral acids such as hydrochloric acid, sulphuric acid or nitric acid, and may include one or more of their salts.

Preferably the oxidative leach is conducted in a reactor vessel comprising one or more compartments. More preferably the oxidative leach is conducted in a reactor vessel comprising a single compartment.

Preferably the reactor vessel is a single compartment autoclave.

- 5 Optionally the reactor vessel may be a two-compartment vessel where the flows are configured to enhance the selectivity of one or more value metals over other value metals. Additionally, this flow scheme can be employed to build value metal concentrations.

- 10 It is to be noted that the word compartment is to be understood to have a definition of a mechanically agitated cell within the autoclave complete with dividing walls, such that each compartment is separated from another by its dividing walls and where the slurry transfers from one compartment to another with minimal back mixing.

- 15 Preferably the partial pressure of oxygen within the leach circuit is in the range of 20 to 500 kPa. Preferably the electrochemical potential is controlled within the range of 280 to 600mV (Ag/AgCl).

Preferably the electrochemical potential is adjusted by altering the partial pressure of oxygen within the leach circuit.

- 20 Preferably in the oxidative leach process the extraction of precious metals (such as gold and silver) and platinum group metals (such as platinum, palladium, ruthenium, rhodium, osmium and iridium) is minimised, such that they remain in the leach residues for recovery by other means.

- 25 Preferably the electrochemical potential is adjusted so that the extraction of gangue sulphides is kinetically slower than that of the more electronegative value metals, thus permitting a significant separation to the leachate between, for example, the value metals and gangue components, and/or the precious metals.

Optionally low concentrations of chlorides are added to the pressure leach autoclave feed slurry to preferentially leach one or more of the

electronegative elements over the more electropositive elements within the concentrate. Typically, the concentration of chlorides added is in the range of about 5-10 g/l. Suitably, chlorides may be used where copper is the dominant mineral in the sulphide concentrate.

- 5 Advantageously other additives to the oxidative leach may be incorporated which reduce or remove the chalcopyrite or millerite mineral surface passivation layer and thus render these minerals amenable to near quantitative leaching.

- 10 Optionally any excess exothermic heat generated in the autoclave is removed with a flash cooling process employing the removal of flash steam and the return to the autoclave of the flash cooled slurry. Preferably any excess exothermic heat generated in the autoclave is attenuated by the addition of quench solutions, and/or the use of internal and or external cooling coils and the like.

- 15 Preferably surfactants are added to the oxidative leach to disperse the elemental sulphur formed during the leach.

Optionally the electrochemical potential on conclusion of the oxidative leach is anaerobically lowered to enhance the leach extent without incurring further gangue metal sulphide oxidation.

- 20 For the purposes of this invention a concentrate refers to any one of the group of concentrates comprising high-pyrite-containing flotation concentrates, sulphide concentrates, partially oxidised sulphide concentrates, oxide ores, sulphide mattes, an alloy or a low sulphide matte containing alloy (malloy), and any combination of these concentrates. The
25 concentrate may, for example, comprise one or more of the following minerals: pentlandite, heazlewoodite, pyrrhotite, millerite, pyrite, sphalerite, carrolite, chalcopyrite and the like.

- Throughout the specification, unless the context requires otherwise, the word "comprise" or variations such as "comprises" or "comprising", will be
30 understood to imply the inclusion of a stated integer or group of integers but

not the exclusion of any other integer or group of integers. Likewise, the word “preferably” or variations such as “preferred”, will be understood to imply that a stated integer or group of integers is desirable but not essential to the working of the invention.

5

Brief Description of the Drawings

The nature of the invention will be better understood from the following detailed description of a specific embodiment of the hydrometallurgical method, given by way of example only, with reference to the accompanying
10 drawings, in which:

Figure 1 is a block flow diagram of a preferred embodiment of the hydrometallurgical method according to the present invention.

Detailed Description of Preferred Embodiments

15 An embodiment of the hydrometallurgical method in accordance with the invention, as illustrated in Figure 1, comprises submitting a concentrate 101 to an oxidative leach 110. The concentrate 101 is typically fed to the leach stage 110 in a slurry form comprising the concentrate in an aqueous stream containing at least water. The concentrate 101 consists of a blend of
20 electronegative and electropositive elements with the elements having differing electronegativity in the classical electrochemical series. Some of the electronegative and electropositive elements may be capable of being passivated in an oxidative leaching process. Preferably the concentrate 101 comprises any one of high pyrite containing flotation concentrates, sulphide
25 concentrates, partially oxidised sulphide concentrates, oxide ores, sulphide mattes, an alloy or a low sulphide matte containing alloy (malloy) or any combination of these concentrates. The concentrate may, for example, comprise one or more of the following minerals: pentlandite, heazlewoodite, pyrrhotite, millerite, pyrite, sphalerite, carrolite, chalcopyrite and the like.

The concentrate 101 comprising electronegative, electropositive and gangue metals or elements is preferably delivered to a repulp step 100. The concentrate 101 can be repulped in a recycle liquor comprising electrowinning spent electrolyte, solvent extraction raffinate or similar barren liquor 102, or a recycle leachate 103 as in a leachate from a secondary leach step, or water 104; or an acid stream 105 or a blend of these streams that are fed to the repulp step 100. The acid stream 105 can be one or more of a blend of mineral acids such as hydrochloric acid, sulphuric acid or nitric acid, and may include one or more of their salts.

- 10 Preferably a lixiviant is added to the concentrate 101 to establish a lixiviant-concentrate blend either prior to, or during, the oxidative leach. This may have the benefits of removing gangue minerals such as carbonates, and also partially leaching the value metals.

The repulped concentrate slurry 106 is then fed to an oxidative leach step 110 targeting the recovery of one or more electronegative elements over the electropositive elements within the concentrate blend. The leach process 110 can be conducted at ambient or elevated temperature at either atmospheric or elevated pressure. Preferably the oxidative leach 110 is supported by the introduction of an oxidant 111. Preferably an inert gas 112 is introduced to dilute the oxidant 111 concentration in the leach step 110. Alternately the inert gas may be a natural diluent within the oxidant as low levels of nitrogen are present within commercial oxygen. Alternately the diluent gas can be internally generated, for example, as carbon dioxide.

As noted above, the leach step 110 can be further supported by the introduction of a lixiviant 113 to establish a lixiviant-concentrate blend. The lixiviant may be a sulphate, chloride, nitrate or a blend of these lixiviants. Typically, the oxidant is drawn from one or more of oxygen, air, chlorine, peroxide or any similar liquid or gaseous oxidants.

Additionally, the leach step 110 can be further supported by the introduction, as may be required, of a quench fluid 114 that is intended to control any exotherm in the leach 110. Additionally, the leach step 110 can be further

supported by the introduction, as may be required, of a dispersant as in a lignosulfonate or a defoamer.

In the leach step 110 conditions are chosen to ensure that one or more of the value elements are extracted into the aqueous phase or oxidised quasi-selectively or kinetically preferentially over others. In leach step 110 the more electronegative metals (hereafter also referred to as value metals) are quasi-selectively or preferentially extracted and/or oxidised over the more electropositive elements. Similarly, in leach step 110 the more electronegative metals may be extracted kinetically more efficiently than the more electropositive elements. Advantageously the method exploits the difference in the oxidation kinetics of value minerals over those of gangue minerals specifically pyrite, tellurides, selenides, arsenopyrite, etc.

The hydrometallurgical method according to the invention preferably comprises adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit to preferentially leach one or more of the electronegative elements over the more electropositive elements within the concentrate. Advantageously the method also comprises adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit to preferentially leach one or more of the more electronegative metals in the concentrate over the oxidation or extraction of the more electropositive elements in the same concentrate. Advantageously adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit minimises the passivation of one or more value metals in the concentrate to achieve economically high extractions of the one or more value metals.

Typically, the oxidative leach 110 is conducted in a leach reactor vessel being a continuously operating oxidative pressure leach autoclave, comprising a single compartment. The lixiviant-concentrate blend is continuously fed into the autoclave. The electrochemical potential of the bulk concentrate slurry 106 in the oxidative leach may be readily controlled

by adjusting the oxygen partial pressure and employing an autoclave with a single compartment for the oxidative leach 110.

By operating an autoclave continuously, a steady and stable operation of the leach process is achieved by making minor adjustments to the feed
5 solids density, slurry flowrate, retention time, free acid and oxygen partial pressure, in responses to changes in the feed chemistry and mineralogy. The oxidative leach conditions are controlled by frequently sampling the pregnant leach solution and the leached solids. In this way it is possible to achieve far more uniform leaching performance than would be possible in a
10 batch operation. The use of a continuous operation allows for effective production throughput.

In a batch operation, a steady and stable operation of the oxidative leach is not possible.

The use of a single compartment oxidative pressure leach autoclave
15 effectively allows for the adjustment of the electrochemical potential throughout the entire leach circuit, by control of the partial oxygen pressure.

In contrast, in a multi-compartment autoclave, it is difficult to control the partial oxygen pressure within a compartment as the headspace is shared by all compartments. Thus, it is not possible to influence the
20 electrochemical potential in more than one compartment of a multi-compartment autoclave.

By removing the compartment walls and creating one compartment, it becomes possible, by controlling the partial oxygen pressure in the headspace, to control the slurry electrochemical potential and hence
25 influence the reaction extent everywhere in the slurry within the autoclave, targeting the value metals over the gangue, and minimising the passivation of sulphide minerals.

Furthermore, by use of a single compartment, it becomes possible by controlling the partial oxygen pressure in the headspace, to control the
30 slurry electrochemical potential and hence limit sulphide sulphur oxidation

extent in the slurry, thereby reducing wasteful oxidation of gangue sulphides.

In this preferred single compartment leach reactor, the feed slurry 106 may be introduced into the final compartment of an equivalent multi-compartment
5 reactor or autoclave, and the influent alloy and or sulphide minerals in the solids influences the electrochemical potential of the slurry emanating from the autoclave.

In the preferred embodiment, the electrochemical potential is controlled by adjusting the bulk slurry phase partial oxygen pressure in the leach vessel.
10 Typically, the quasi-selective or kinetically preferential leach is undertaken under atmospheric or pressurised conditions in a horizontal or vertical leach vessel. The leach stage 110 may incorporate one or more of the above combinations of reactor vessel conditions. The leach stage 110 may involve a semi-continuous or a continuous process.

15 Preferably the concentrate is continuously fed into the reactor vessel. In one embodiment the reactor vessel is an autoclave.

Preferably in the oxidative leach the leaching of precious and platinum group metals is minimised, such that they remain in the leach residues for recovery by other means.

20 Advantageously, additives to the oxidative leach are incorporated which reduce or remove the chalcopyrite mineral surface passivation layer and thus render the minerals amenable to leaching.

Additionally, any excess exothermic heat generated in the autoclave is removed with a flash cooling process employing the removal of flash steam
25 and the return to the autoclave, in part or the whole, of the flash cooled slurry or solution. Preferably the excess exothermic heat generated in the autoclave is attenuated by the addition of quench solutions, and or the use of internal and or external cooling coils and the like.

Preferably surfactants are added to the leach to disperse the elemental sulphur formed during the leach. Additionally, the electrochemical potential on conclusion of the oxidative leach may be anaerobically lowered to enhance the leach extent without incurring further gangue metal sulphide oxidation.

Once the leach stage is complete, the products 115 of the leach are then subjected to recovery by means of solid-liquid separation 120. Solid-liquid separation may comprise one or more of thickening stages, washing, filtration, centrifuging and the like, or alternatively by direct extraction from the leach discharge slurry 115 by means of ion exchange. The solids fraction in the separation step 120 can be washed partially or totally free of solute by employing a wash fluid 121.

The filtrate stream 122 and the washate stream 123 from the separation step 120 may be combined as filtrate 122 or processed separately. The solids fraction from separation step 120 can then, but not necessarily, proceed as stream 124 to a discharge product. The filtrate stream 122 can be treated to recover the value metal or metals extracted in the leach 110 in a combination of purification and a metal recovery step or steps 160. The barren liquor from metal recovery 160 can comprise a raffinate or spent electrolyte or any other barren liquor stream 102, and this can be recycled for repulping the leach concentrate 101 in repulp step 100.

The hydrometallurgical method according to the invention will now be further illustrated by several examples:

25 **Example 1**

A polymetallic sulphide concentrate with high pyrite content, containing copper, zinc, lead, cobalt, nickel and precious metals was subjected to the leach step in Figure 1.

The concentrate assayed approximately:

		%
	Cu	2.8
	Pb	0.9
	Zn	0.4
5	Co	0.4
	Ni	0.2
	Fe	31
	Sulphur (total)	40
	Sulphide	35
10	Ag	50ppm

The primary sulphide minerals comprised pyrite, some marcasite, chalcopyrite and sphalerite.

The more electronegative value metals in this example were considered to be the copper, cobalt, nickel and zinc.

The concentrate was ground to 50 microns and was continuously leached in a single compartment autoclave under steady state conditions at a partial oxygen pressure within the range of 180 to 240 kPa and a temperature of 150°C employing sulphuric acid and 5 g/l of chloride.

Under these conditions the electrochemical potential was controlled within the range of 400 to 480mV (Ag/AgCl).

The extraction of the value metals was:

		%
	Cu	95
25	Co	82
	Ni	78
	Zn	95

While the oxidation of the more electropositive sulphide sulphur, including that associated with copper and zinc, was 25% of the contained sulphide sulphur, under those conditions the oxidation kinetics of the pyrite was

kinetically impaired sufficient to maintain the pyrite oxidation below 25%. This permitted an economic separation between the value elements of copper, cobalt and zinc, over that of the gangue iron and associated sulphide.

5 Example 2

A polymetallic sulphide concentrate with high pyrite content, containing copper, zinc, lead, cobalt, nickel and precious metals was subjected to the leach step in Figure 1.

The concentrate assayed approximately:

	%
Cu	2.88
Zn	0.39
Co	0.37
Fe	31.9
Sulphur (total)	40.1
Sulphide	33.6

10

The primary sulphide minerals comprised pyrite, some marcasite, chalcopyrite and sphalerite.

The more electronegative value metals in this example were considered to be the copper, cobalt and zinc.

- 15 The concentrate was ground to 20 microns and continuously leached in a single compartment autoclave under steady state conditions at a partial oxygen pressure within the range of 170 to 240 kPa and a temperature of 130°C employing sulphuric acid and 5 g/l of chloride.

- 20 Under these conditions the electrochemical potential was controlled within the range of 440 to 480mV (Ag/AgCl).

The extraction of the value metals was:

	%
Cu	93
Co	86
Zn	96

- 5 While the oxidation of the more electropositive sulphide sulphur, including that associated with copper and zinc, was 15% of the contained sulphide sulphur, under those conditions the oxidation kinetics of the pyrite was kinetically impaired sufficient to maintain the gangue pyrite oxidation below
- 10 15%, this permitted an economic separation between the value elements of copper, cobalt and zinc over that of the iron and associated sulphide.

Example 3

- A polymetallic sulphide concentrate with high pyrite content, containing copper, zinc, lead, cobalt, nickel and precious metals was subjected to the
- 15 leach step in Figure 1.

The concentrate assayed approximately:

	%
Cu	1.8
Co	0.24
Pb	2.1
Zn	1.3
Fe	31.5
Sulphur (total)	37

- The primary sulphide minerals comprised pyrite, some marcasite, chalcopyrite, galena and sphalerite.
- 20 The more electronegative value metals in this example were considered to be the copper, cobalt and zinc.

The concentrate was ground to 50 microns and continuously leached in a single compartment autoclave under steady state conditions at a partial oxygen pressure within the range of 180 to 240 kPa and a temperature of 150°C employing sulphuric acid and 10 g/l of chloride.

- 5 Under these conditions the electrochemical potential was controlled within the range of 400 to 450mV (Ag/AgCl).

The extraction of the value metals was:

	%
Cu	95
10 Co	80
Zn	97
Pb	0

- While the oxidation of the more electropositive sulphide sulphur, including that associated with copper and zinc, was 15% of the contained sulphide sulphur, under those conditions the oxidation kinetics of the pyrite was kinetically impaired to maintain the gangue pyrite oxidation below 15%, in order to allow an economic separation between the more electronegative value elements of copper, cobalt and zinc over that of the gangue iron and associated sulphide.
- 15
- 20

Example 4

A polymetallic sulphide concentrate with high pyrite content, containing copper, zinc, lead, cobalt, nickel and precious metals was subjected to the leach step in Figure 1.

- 25 The concentrate assayed approximately:

	%
Cu	3.0
Co	0.37
Zn	0.42
Fe	31.7

Sulphur (total)	36.5
Sulphide	36.3

The primary sulphide minerals comprised pyrite, some marcasite, chalcopyrite and sphalerite.

5 The more electronegative value metals in this example were considered to be the copper, cobalt and zinc.

The concentrate had an “as-received” sizing of 95 microns and was not ground before being continuously leached in a single compartment autoclave under steady state conditions at a partial oxygen pressure within the range of 80 to 140 kPa and a temperature of 150°C employing sulphuric
10 acid and 10 g/l of chloride.

Under these conditions the electrochemical potential was controlled within the range of 400 to 440mV (Ag/AgCl).

The extraction of the value metals was:

		%
15	Cu	95
	Co	80
	Zn	96

While the oxidation of the more electropositive sulphide sulphur, including
20 that associated with copper and zinc, was 40% of the contained sulphide sulphur, under those conditions the oxidation kinetics of the pyrite was kinetically impaired sufficiently to maintain the pyrite oxidation below 40%. For this example, the targeted pyrite oxidation was specifically increased to allow for an increase in cobalt and nickel extraction, these minerals being
25 closely associated with the gangue pyrite.

Example 5

A polymetallic sulphide concentrate with high pyrite content, containing nickel, copper, zinc, cobalt was subjected to the leach step in Figure 1.

The concentrate assayed approximately:

	%
Ni	10.0
Cu	0.6
Zn	2.1
Fe	27.1
Sulphur (total)	31

5

The primary sulphide minerals comprised pentlandite, pyrite, millerite, chalcopyrite, and sphalerite.

The more electronegative value metals in this example were considered to be the nickel, copper and zinc.

- 10 The concentrate was continuously leached in a single compartment autoclave under steady state conditions at a partial oxygen pressure of approximately 110 to 130 kPa and a temperature of 165°C employing sulphuric acid. The total retention time was 3 hours.

- 15 Under these conditions the electrochemical potential was controlled in the range 460 to 470mV (Ag/AgCl).

The extraction of the value metals was:

	%
Ni	>95
Cu	60
20 Zn	91 to 92

The extraction of the iron gangue was:

	%
Fe	6 to 10

While the oxidation of the more electropositive sulphide sulphur, including that associated with nickel, copper and zinc, was 67 to 71% of the contained sulphide sulphur, under those conditions the oxidation kinetics of the gangue pyrite was kinetically impaired. This permitted an economic separation between the more electronegative value elements of nickel, and zinc over that of the gangue iron and associated sulphide.

When the same test was repeated employing a partial oxygen pressure of 600 kPa with all other conditions identical the electrochemical potential was approximately 515 to 520 mV (Ag/AgCl)

The extraction of the value metals was:

	%
Ni	87 to 88
15 Cu	58 to 62
Zn	93 to 94

The extraction of the iron gangue was:

	%
20 Fe	11 to 12

The oxidation of the more electropositive sulphide sulphur, including that associated with nickel, copper and zinc, was 97-99% of the contained sulphide sulphur.

However, of significance in Example 5 is the higher partial pressure of oxygen, rather than enhancing the extraction of nickel, has resulted in a much lower extraction of nickel as a consequence of passivation of the millerite.

Example 6

A polymetallic sulphide concentrate with high pyrite content, containing nickel, copper, zinc, cobalt was subjected to the leach step in Figure 1 in

which the autoclave had two compartments with a dividing wall between the two compartments and a shared vapour head space.

The concentrate assayed approximately:

	%
Ni	10.0
Cu	0.6
Zn	2.9
Co	0.3
Fe	27.1
Sulphur (total)	31

- 5 The primary sulphide minerals comprised pentlandite, pyrite, millerite, chalcopyrite, and sphalerite.

The more electronegative value metals in this example were considered to be the nickel, copper, zinc and cobalt and amongst these, the nickel, zinc and cobalt were selectively separated from copper.

- 10 The concentrate was repulped in a copper SX raffinate containing sulfuric acid derived from the leachate of the second compartment and continuously leached in the first of a two-compartment autoclave. The slurry from this first compartment was split with some slurry passing over the dividing wall to the second compartment. The balance of the first compartment slurry was
- 15 continuously discharged, thickened with the underflow returned to the first compartment feed tank. This is a classical Flash Thicken Recycle step. The thickener overflow was advanced to recover nickel. The slurry in the second compartment was further leached and the exotherm quenched with direct water injection. The contents of the second compartment were discharged
- 20 to a partial neutralisation step. The mother liquor from this neutralisation was treated to recover copper. Steady state conditions were maintained at a partial oxygen pressure of approximately 110 to 130 kPa and a temperature of 165°C. The total retention time was 3 hours.

Under these conditions the electrochemical potential was controlled in the range of 290 to 320 mV (Ag/AgCl) in the first compartment and in the range 400 to 440mV (Ag/AgCl) in the second compartment.

- 5 The extraction of the value metals in the first compartment was:

		%
	Ni	70
	Cu	Negative
	Zn	73
10	Co	40
	Fe	8

- The thickener overflow liquor from the first compartment discharge had negligible copper thus identifying an opportunity to improve the leach selectively for nickel-over-copper in a two-compartment autoclave. The nickel concentration in this thickener overflow was 50 to 55 g/l.

The second compartment discharge residue composition was:

		%
	Ni	0.52 (majority of which was non-sulphide nickel)
20	Cu	0.02
	Zn	0.17
	Co	0.11
	Fe	29.0
	S	26.2

- 25 The overall extraction of the value metals from this two-compartment autoclave was:

		%
	Ni	96-97 (includes non-recoverable non-sulphide nickel)
	Cu	97
30	Zn	96

Co	75
Fe	10

- While the oxidation of the more electropositive sulphide sulphur, including that associated with nickel, copper and zinc, was 45-50% of the contained sulphide sulphur, under those conditions the oxidation kinetics of the gangue pyrite was kinetically impaired. This permitted an economic separation between the more electronegative value elements of nickel, copper and zinc over that of the gangue iron and associated sulphide. Additionally, by splitting the autoclave into two compartments enhanced the leach selectivity of nickel, zinc and cobalt over that of copper.

Example 7

A copper nickel sulphide concentrate containing precious metal was subjected to the leach step in Figure 1.

- The concentrate assayed approximately:

	%
Cu	49.8
Ni	3.1
S	24.0
Pd + Rh + Ag	1150 g/t

- The electronegative elements in this example are the base metals of copper and nickel. The more electropositive elements are the precious metals of palladium, rhodium and silver and while sulphide sulphur was considered to be electropositive; the objective in this test was to near-quantitatively separate the base metals from the precious metals by controlling the electrode potential employing the oxygen partial pressure.

The concentrate was continuously leached at 10% solids in a spent electrolyte in a single compartment autoclave with a lixiviant that comprised:

	g/L
Cu	39
Ni	24
H ₂ SO ₄	7

The primary leach was conducted at 200°C with an ORP of approximately 400mV (Ag/AgCl) employing an oxygen partial pressure of 35 kPa employing the flowsheet in Figure 1.

- 5 In the leach, the extraction of the more electronegative metals of copper and nickel was:

	%
Cu	93 to 96
Ni	97

- 10 The more electropositive metals comprising palladium, rhodium and silver (PGE) were poorly extracted in leach to the extent that the ratio of copper to the sum of all the PGE ratio in the leachate was in excess of 20,000.

Example 8

An alloy comprising nickel and copper of the type produced in a DC Arc furnace was subjected to the leach step in Figure 1.

The alloy concentrate assayed approximately:

	%
Cu + Ni + Fe	89
S	0.2
Precious Metals	4.9

15

The electronegative elements in this example are the base metals of copper, nickel and iron. The more electropositive elements are the precious metals comprising largely palladium, rhodium and silver and while sulphide sulphur was considered to be electropositive; the objective in this test was

to near-quantitatively separate the base metals from the precious metals by controlling the electrode potential employing the oxygen partial pressure.

The alloy was continuously leached at 130°C in a single compartment autoclave and at a partial oxygen pressure of below 50 kPa and with an
5 oxygen utilisation in the leach in excess of 97%.

Under these conditions the electrochemical potential was controlled below 150 mV (Ag/AgCl).

The alloy was repulped with a blend of spent electrolyte and secondary leach filtrate comprising sulphuric acid, nickel and copper sulphate, and
10 some soluble precious metals that derived from a secondary leachate. This slurry was fed to a continuously operated autoclave leach vessel comprising a single compartment.

The mean retention in the autoclave was just under one hour and the free acidity in the leach was 20-30 g/l.

15 The extraction of the electronegative elements (nickel, iron and copper) was in excess of 94% whereas the concentration of the electropositive elements in the leachate as measured by silver was typically less than 500 parts per billion (total).

Now that a preferred embodiment of the hydrometallurgical method has
20 been described in detail, it will be apparent that the described embodiment provides a number of advantages over the prior art, including the following:

(i) The process provides conditions in which the oxidation and or extraction of more electronegative elements can be achieved without significant co-extraction or oxidation of more electropositive
25 elements. This separation in the extraction of the more electronegative elements over the electropositive elements is achieved by controlling the pressure oxidation autoclave at lower partial oxygen pressures.

- 5 (ii) The process also provides conditions, as a consequence of employing an autoclave with a single compartment, in which the continuous introduction of feed concentrate favourably influences the electrochemical conditions within the autoclave discharge, thereby permitting a lower electrochemical potential in the discharge slurry.
- 10 (iii) The process of allowing very small quantities of influent feed slurry to blend with bulk leached slurry reduces the electrochemical potential very significantly without incurring significant loss of extraction of the more electronegative value metals therein permitting the extractive separation of the more electronegative elements over the more electropositive elements.
- 15 (iv) The process also provides conditions that can control the passivation of minerals such as chalcopyrite and millerite which passivation is considered to be as a consequence of the activity of polyvalent oxidants in the aqueous phase of the type ferric, cobaltic, manganic and the like.
- 20 (v) The process also provides for reduced sulphur oxidation in high sulphide concentrates where residual sulphides are instrumental in attenuating the electrochemical potential in the autoclave.
- 25 It will be readily apparent to persons skilled in the relevant arts that various modifications and improvements may be made to the foregoing embodiments, in addition to those already described, without departing from the basic inventive concepts of the present invention. Therefore, it will be appreciated that the scope of the invention is not limited to the specific embodiments described.

5

Claims

10 1. A hydrometallurgical method for treating a sulphide concentrate in a continuously operating oxidative leach reactor, the sulphide concentrate comprising a blend of electronegative and electropositive elements, the method comprising:

submitting a slurry comprising the concentrate in an aqueous stream
15 containing at least water to an oxidative leach targeting the recovery of one or more electronegative elements over the more electropositive elements within the concentrate; and

adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit to preferentially leach one or more of the
20 electronegative elements over the more electropositive elements within the concentrate; and

wherein the electronegative elements comprise at least one of copper, nickel, cobalt or zinc.

2. The hydrometallurgical method as claimed in claim 1, wherein the
25 method also comprises adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit to preferentially leach one or more value or more electronegative metal sulphides over the oxidation or extraction of gangue or more electropositive metal sulphides within the concentrate.

30 3. The hydrometallurgical method as claimed in claim 1 or claim 2, wherein the partial pressure of oxygen within the leach circuit is in the range of 20 to 500 kPa.

4. The hydrometallurgical method as claimed in any one of claims 1 to 3, wherein the electrochemical potential is controlled within the range of 280 to
35 600mV (Ag/AgCl).

- 5 5. The hydrometallurgical method as claimed in any one of claims 1 to 4, wherein the process also provides conditions in which influent feed concentrate favourably influences the electrochemical conditions within the autoclave discharge thereby permitting a lower electrochemical potential in the discharge slurry.
- 10 6. The hydrometallurgical method as claimed in claim 5 wherein small quantities of the influent feed concentrate are required to lower the electrochemical potential in the discharge slurry.
7. The hydrometallurgical method as claimed in any one of claims 1 to 7, wherein one or more of the electronegative and electropositive metals in the
- 15 concentrate may be capable of being passivated in the concentrate in an oxidative leaching process, and wherein the method also comprises adjusting the electrochemical potential by controlling the partial pressure of oxygen within the leach circuit to minimise the passivation of one or more value metals in the concentrate.
- 20 8. The hydrometallurgical method as claimed in claim 1, in which the electrochemical potential is adjusted by controlling the partial pressure of oxygen within the leach circuit such that the extraction of more electronegative or value elements can be achieved without significant co-extraction or oxidation of more electropositive elements or compounds as in
- 25 iron sulphides or elemental sulphur.
9. The hydrometallurgical method as claimed in any one of claims 1 to 8, wherein the process also provides for reduced sulphur oxidation in high sulphide concentrates.
10. The hydrometallurgical method as claimed in any one of claims 1 to 9,
- 30 wherein a lixiviant is added to the concentrate to establish a lixiviant-concentrate blend either prior to, or during, the oxidative leach.
11. The hydrometallurgical method as claimed in claim 10, wherein the lixiviant is an acid or a blend of mineral acids.

- 5 12. The hydrometallurgical method as claimed in claim 11, wherein the acid is hydrochloric acid, sulphuric acid or nitric acid, and/or one or more of the salts of hydrochloric acid, sulphuric acid or nitric acid.
13. The hydrometallurgical method as claimed in claim 1, wherein the sulphide concentrate may comprise an alloy of some of the electronegative
10 elements.
14. The hydrometallurgical method as claimed in claim 1, wherein the oxidative leach is conducted in a reactor vessel comprising one or more compartments.
15. The hydrometallurgical method as claimed in claim 14, wherein the
15 reactor vessel comprises one compartment.
16. The hydrometallurgical method as claimed in claim 14, wherein the reactor vessel comprises two or more compartments to enhance the leach selectivity of one or more of the electronegative elements over others.
17. The hydrometallurgical method as claimed in claim 14, wherein the
20 reactor vessel is a pressure leach autoclave.
18. The hydrometallurgical method as claimed in claim 17, wherein low concentrations of chlorides are added to the pressure leach autoclave feed slurry to preferentially leach one or more of the electronegative elements over the more electropositive elements within the concentrate.
- 25 19. The hydrometallurgical method as claimed in claim 18, wherein the concentration of chlorides added is in the range of about 5 to 10 g/l.

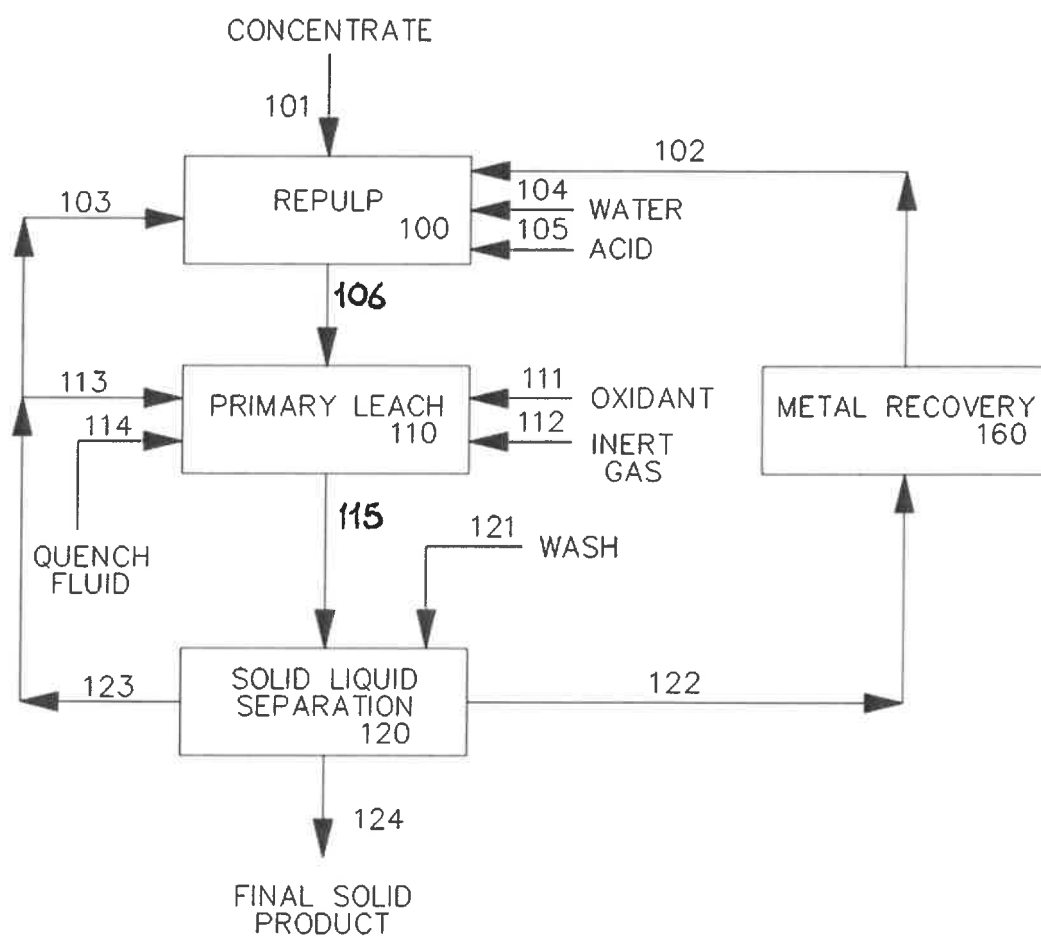


Figure 1