

## NEW FRONTIERS IN EXTRACTIVE METALLURGY

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### Abstrakt

Kým pyrometalurgia je najvhodnejšia metóda spracovania vysoko bohatých oxidických rúd, hydrometalurgia je vhodná na spracovanie chudobných oxidických rúd a taktiež je vhodná pre sulfidické koncentráty. Elektrometalurgia v súčasnosti dominuje pri výrobe hliníka, elektrolytickej rafinácii medi, a elektrolytickom získavaní zinku. Táto metóda bola taktiež úspešná pri elektrolytickom získavaní medi z roztoku získaného lúhovaním – kvapalinovou extrakciou. Avšak, ak sa priemysel medi posúva smerom od tavenia ku tlakovému lúhovaniu, potom elektrolytické získavanie kovov bude dominantné v oblasti hydro - elektrometalurgie. V súčasnosti všetky fakty poukazujú na tlakové lúhovanie ako významnú cestu spracovania medených sulfidických koncentrátov v dvadsiatom prvom storočí. Táto technológia sa teraz využíva na spracovanie zinkových sulfidických koncentrátov a odolných koncentrátov zlata a zmierňuje problém znečistenia SO<sub>2</sub>.

### Abstract

While pyrometallurgy is most suitable for treating high grade oxide ores, hydrometallurgy is suitable for low grade oxide ores and for sulfide concentrates. Electrometallurgy is presently dominated by the production of aluminum, the electrorefining of copper, and the electrowinning of zinc. It has been also successful in the electrowinning of copper from solution obtained by leaching-solvent extraction. However, if the copper industry shifts from smelting to pressure leaching then electrowinning will dominate the area of aqueous electrometallurgy. At present all data point out to pressure leaching as the eminent route for treating copper sulfide concentrates in the twenty first century. This technology now used for treating zinc sulfide and refractory gold concentrates, alleviates pollution problems by SO<sub>2</sub>.

**Key words:** extractive metallurgy

### Introduction

Extractive metallurgy is the science and engineering of extracting metals from ores. Closely related to this technology is mineral beneficiation whereby ores are treated by mechanical, physical, and physico-chemical means to prepare concentrates either for metal production or to be used for the chemical and other industries as industrial minerals Fig.1. Extractive metallurgy can be divided into three large sectors Fig.2.

- *Pyrometallurgy.* The use of high temperature for recovering metals

- *Hydrometallurgy*. The use of aqueous solutions
- *Electrometallurgy*. The use of electric current to effect a chemical reaction

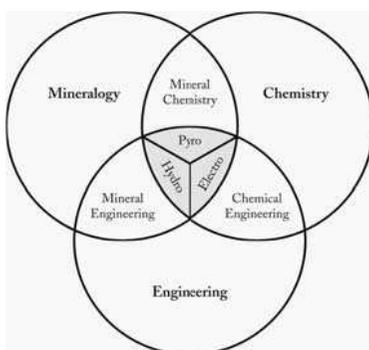
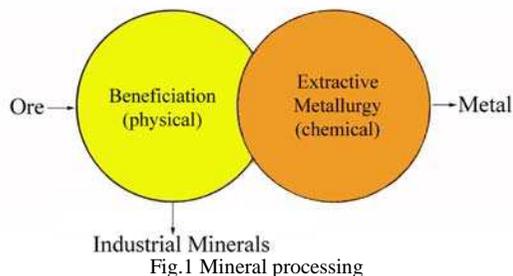


Fig.2 Extractive metallurgy is divided into pyro-, hydro-, and electrometallurgy

### Pyrometallurgy

Pyrometallurgy is the oldest of these technologies. The ancient Egyptians melted native gold Fig. 3 and produced copper and iron from their oxide ores by thermal methods. Furnaces increased in size Fig. 4, 5. Today it is the only route to produce iron and it is certain that it will remain so for many decades to come because pyrometallurgy is most suitable for the treatment of high grade oxide ores. Even when high grade iron ores are exhausted, it is possible to beneficiate the low grade ores and produce pellets suitable for reduction in the blast furnace which is a very efficient large reactor, being at the same time a heat exchanger.



Fig.3 Melting of ores was practiced by the ancient Egyptians

### *Copper*

Copper is another ancient metal. Like gold it also occurs in the native state but to a minor extent. Its main occurrences are as oxides or sulfides. In ancient civilizations, the oxides were extensively exploited by reduction with carbonaceous material (timber) in small furnaces. When man started to exploit the sulfides, new problems arose:

- Emission of large amounts of  $\text{SO}_2$  because of the partial oxidation of the sulfides.
- The material melted during heating because of the formation of matte.



Fig.4 A Roman furnace to produce iron. To the left, a kiln for producing charcoal from timber

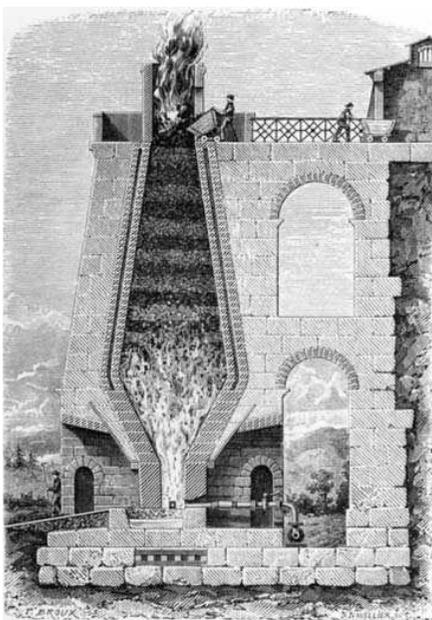
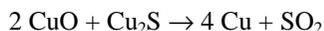


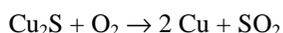
Fig.5 A nineteenth century furnace

Eventually, a process was developed, known as the roast-reaction process, whereby the solidified matte was crushed and finely ground; half of it was oxidized then mixed thoroughly with the other half and the mixture was melted in a furnace with fluxes so that the roast-reaction between copper oxide and copper sulfide would take place to produce metallic copper, and at the same time iron was eliminated as a slag:



The Welsh metallurgists were skilled in conducting this process, which became known as the Welsh Process. Matte from as far away as Butte, Montana and Chuquicamata, Chile was shipped to Swansea, Wales for transformation to copper. Sulfur dioxide was emitted in this step and no attempt was made to capture it.

Not far from Swansea, Henry Bessemer invented his revolutionary process in 1856 to produce steel from pig iron by blowing air through the molten material. The process was conducted in a special reactor known as converter. In this process, the time to produce a batch of steel was reduced from few days to few minutes and at the same time the need to use fuel for making the transformation was eliminated. The copper industry adapted the same principle few years later and as a result, the roast reaction was displaced by the conversion reaction.



### *The horizontal furnace*

It is natural that metallurgists will treat rich ores first before they consider low-grade material because it is more economical. In the copper industry high-grade massive sulfide ores were broken down into small lumps and charged to a vertical furnace whereby the unwanted rock was removed as a slag and the copper-containing minerals were collected as a matte. When rich massive copper sulfide deposits became scarce, mining engineers turned to low-grade ores. This move coincided with the invention of the flotation process at the beginning of the twentieth century. Hence it became possible to obtain rich pulverised concentrates from low-grade ores. These concentrates, however, were not suitable for charging to the vertical furnace, since the charge would be blown out of the furnace when air was introduced at the lower part of the shaft. As a result, copper metallurgists turned their attention to adapting the same horizontal furnace that was used for the roast-reaction to melt the concentrates. This was the beginning of the era of the reverberatory furnace which has dominated the copper industry worldwide during the twentieth century.

While the vertical furnace is an excellent reactor: it is a heat exchanger as well as counter-current mass transfer reactor, the horizontal furnace suffers from the following disadvantages:

- Inefficient heat transfer since heat is mainly transferred by radiation from the roof, thus more fuel is burned unnecessarily.
- Excessive dust formation since the powdered concentrate is charged at right angles to the gas flow thus necessitating the installation of a large dust recovery system.
- Gases leaving the furnace are at high temperature necessitating the use of a bulky and expensive heat recovery system.
- The gases contain small amounts of  $\text{SO}_2$  which cannot be economically recovered for making  $\text{H}_2\text{SO}_4$ , hence they are emitted in the atmosphere.

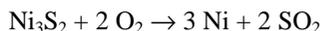
New smelting routes were developed, directed mainly towards energy economy. The most successful were the flash smelting process (Outokumpu Process), the bath smelting process (Noranda and INCO) and the continuous Mitsubishi Process. All these processes make use of oxygen or oxygen-enriched air instead of air and have several advantages:

- All the steps leading to the production of the raw metal are exothermic.
- Sulfur dioxide is produced in high concentrations and can be economically captured for sulfuric acid manufacture. However a nearby market for this acid must be available.

### Nickel

Nickel sulfide or nickel-copper sulfide concentrates are roasted to decrease their sulfur content, then melted in a flash smelting furnace or in an electric furnace to separate the gangue minerals as slag and obtain a matte. The matte is then purified in a converter to remove the iron and obtain a relatively pure  $\text{Ni}_3\text{S}_2$  or a binary sulfides  $\text{Ni}_3\text{S}_2\text{-Cu}_2\text{S}$ . During this process, also large amounts of  $\text{SO}_2$  are formed.

- At INCO refinery in Sudbury, the molten binary sulfides  $\text{Ni}_3\text{S}_2\text{-Cu}_2\text{S}$  are cast in molds and allowed to cool slowly, then crushed, ground, and separated by flotation. The  $\text{Ni}_3\text{S}_2$  concentrate is then melted in a flash smelting furnace while the  $\text{Cu}_2\text{S}$  concentrate is converted to blister copper in a flash converting furnace. During these operations large amounts of  $\text{SO}_2$  are also formed.
- In some plants,  $\text{Ni}_3\text{S}_2$  is oxidized to  $\text{NiO}$  which can either be: reduced by carbon to metallic nickel which is then refined electrolytically or by the carbonyl process, or dissolved in  $\text{H}_2\text{SO}_4$  to form nickel sulfate solution from which pure nickel is obtained by electrowinning. Sulfur dioxide is formed in the oxidation step and must be captured.
- Nickel sulfide undergoes conversion with oxygen in a top blown rotary converter:



This route is more efficient than the oxidation-reduction route described above, because the process is not only exothermic but also does not require a reducing agent. However, sulfur dioxide must be captured and transformed into acid.

For each ton nickel produced about 10 tons of  $\text{SO}_2$  are generated. Part of this  $\text{SO}_2$  is liquified to supply the pulp and paper industry requirement and another part is converted to  $\text{H}_2\text{SO}_4$  for local use. But the bulk of  $\text{SO}_2$  is emitted to the atmosphere. To minimize the impact of this  $\text{SO}_2$  on the local environment, INCO constructed in 1972 the tallest chimney in the world -- 381 meter high, as high as the Empire State building Fig. 6.



Fig.6 INCO chimney at Sudbury, 381 m high

### Coke

The transition from timber to coal and the transformation of coal into coke in the eighteenth century marked the beginning of the Industrial Revolution. Coke is the backbone of

pyrometallurgy; it is used in large quantities in blast furnaces. It is produced by heating coal in retorts in absence of air then quenching the product by water. It is a highly polluting operation Fig. 7. Recent technology uses dry quenching by nitrogen gas Fig. 8. In this process cooling the coke is done in a closed circuit thus no pollution and in addition there is heat recovery. The process was introduced in Japan and now adopted in Korea, India, China, and some European countries but not yet in North America.



Fig.7 Coke production is a highly polluting process

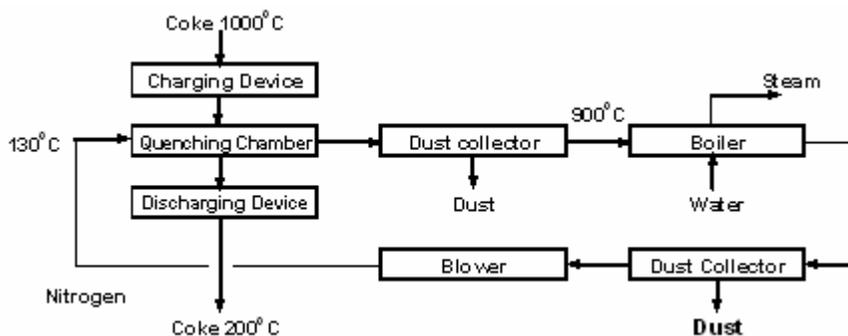


Fig.8 Dry quenching of coke is pollution-free and heat is recovered

## Hydrometallurgy

The pyrometallurgical route for treating sulfide concentrates was the logical way because, historically this was the only way known. When, however, hydrometallurgical knowledge became well established, it became evident that other routes are possible based on the formation of elemental sulfur, thus avoiding the  $\text{SO}_2$  problem. The zinc industry was the first to adopt such technology. The application of pressure leaching technology to the treatment of refractory gold ores and its expansion in the treatment of low-grade nickel oxide ores are a convincing evidence that such technology can be applied effectively to sulfide concentrates of copper, nickel, and lead.

## Gold

Gold was the first metal used by man. It is found in nature in the native state in alluvial deposits or in quartz veins, can be easily melted and cast. When gold particles are extremely fine in the quartz veins, extensive crushing and grinding is necessary - - both are expensive operations, and the simple methods based on gravity concentration are no longer efficient. That is why new technology was sought at the end of the nineteenth century to solve this problem. This resulted in the invention of the cyanidation process.

The application of cyanidation process on industrial scale marked the beginning of modern hydrometallurgy (The Bayer Process for recovering  $\text{Al}_2\text{O}_3$  from bauxite was invented at the same time. It is today the largest pressure leaching operation in the world.). The process has been used world wide for over a century without any significant change. In the past two decades, however, some gold ores did not respond to cyanidation because finely divided gold particles are locked up in a pyrite and arsenopyrite matrix. This type of ore became known as "refractory". Two routes for liberating the gold from the sulfide matrix prior to cyanidation were adopted:

- *Thermal oxidation.* This route takes place in fluidized bed reactors to oxidize the sulfides to oxides. However, it suffered from  $\text{SO}_2$  and arsenic oxide emissions.
- *Aqueous oxidation.* This route takes place in pressure reactors (autoclaves) to oxidize the sulfide to sulfate proved to be pollution-free.

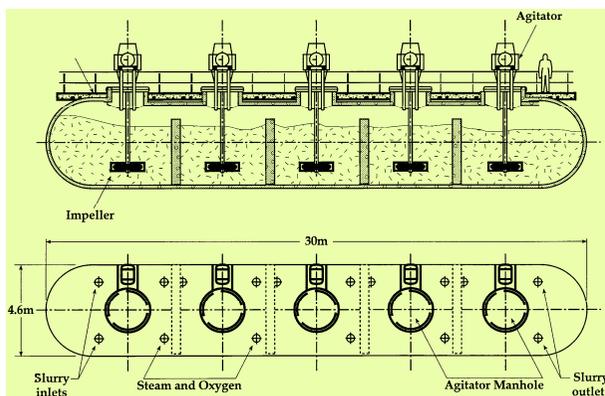


Fig.9 An autoclave 30 m long and 4.6 m diameter

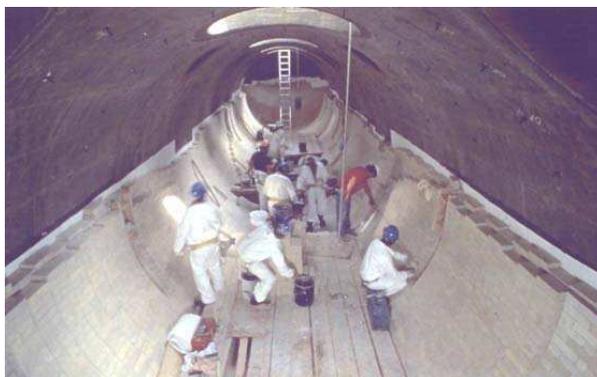
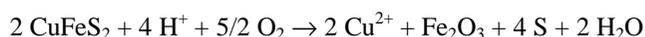


Fig.10 Lining of the interior of the autoclave with acid resisting brick

The aqueous oxidation of sulfidic and arsenical gold ores is another milestone in the history of hydrometallurgy because it opened a new door for the treatment of sulfide concentrates. Huge reactors are used, about 5 m diameters and 30 m long, brick-lined, can withstand a temperature of 200° C and a pressure of 500 kPa Fig. 9 and Fig.10. The reactor has a capacity for treating about 3000 tons per day continuously. Numerous plants using this technology were constructed in USA, Brazil, and other countries (Table 1).

### Copper

The hydrometallurgical route offers a unique opportunity for producing copper without going through SO<sub>2</sub>. The following reaction:



takes place at 150°C and 4000 kPa oxygen partial pressure and therefore must be conducted in an autoclave. The residue from the leaching operation, after flotation of sulfur, should be agglomerated with Portland cement and stock piled on impervious prepared terrain, in the form of dumps to be treated by cyanidation for precious metals recovery. The advantage of this route is the following:

- Cu<sup>2+</sup> is already separated from Fe<sup>2+</sup>
- All the sulfur in the concentrate can be obtained in the elemental form
- When copper is recovered from solution by electrowinning, the acid generated at the anode is equal to that required for leaching.

Table 1 Status of Treatment of Refractory Gold Ores by Pressure Hydrometallurgy

Start up	Plant Location	Owner	Feed	Medium	Capacity t/d	Numer of autoclaves
1985	McLaughlin USA	Homestake USA	ore	acid	2700	3
1986	San Bento Brazil	Genmin S. Africa	concentrate	acid	240	2
1988	Mercur, Utah USA	American Barrick Canada	ore	alkaline	680	1
1989	Getchell USA	First Miss Gold	ore	acid	2730	3
1990	Goldstrike Nevada, USA	American Barrick Canada	ore	acid	1360	1
1991	Goldstrike Nevada, USA	American Barrick Canada	ore	acid	5450	3
1991	Porgera, Papua New Guinea	Placer Dome Canada	concentrate	acid	1350	3
1991	Campbell Canada	Placer Dome Canada	concentrate	acid	70	1
1992	Con Lihir	Nerco Minerals	concentrate	---	90	1
1993	Goldstrike USA	American Barrick Canada	ore	acid	11580	6
1994	Porgera, Papua New Guinea	Placer Dome Canada	concentrate	acid	2700	6
1997	Lihir, Papua New Guinea	Rio Tinto	---	---	---	3

## Zinc

Zinc is relatively a new metal that became known in Europe only 400 years ago. Before World War I it was produced exclusively by the retort processes which were highly polluting and energy intensive, until the roasting-leaching-electrowinning route was developed, and finally the pressure leaching technology was introduced in the 1970's whereby elemental sulfur was produced instead of SO<sub>2</sub>. In the retort processes zinc sulfide concentrate was roasted to oxide and SO<sub>2</sub>, the oxide was then reduced to metal vapors in reactors which were condensed rapidly to yield a low grade zinc. The crude zinc is then refined by distillation at high temperature under vacuum. Horizontal retorts were used for over four centuries. These were then replaced by vertical retorts, but the process still suffered from high fuel costs, low efficiency, as well as severe environmental problems.

The roasting-leaching-electrowinning process was introduced during World War I to exploit low-grade zinc concentrates which were not economical to treat by the retort process. The ZnO produced by roasting was leached by recycle acid obtained from the subsequent electrowinning step. The process required extensive solution purification and was generally successful, because it yielded directly high-purity zinc but suffered from the following:

- Sulfuric acid must be produced from the SO<sub>2</sub> generated in the roasting step.
- A large portion of zinc was in the residue in form of ferrite, ZnFe<sub>2</sub>O<sub>4</sub>, which was insoluble in the recycle acid and had to be treated in a separate circuit. Processes were developed to treat this material but these created other problems, e.g., the final residue was more voluminous than the original, thus creating a stock piling problem.

Pressure leaching process was developed in Canada in the 1970's and at present there are four operating plants using this technology. It is based on the aqueous oxidation of zinc sulfide concentrates in dilute H<sub>2</sub>SO<sub>4</sub> at 150°C and under oxygen pressure of 700 kPa Fig. 11:



Autoclaves lined with acid-resisting bricks are used. The reaction is exothermic and yields elemental sulfur. No ferrites are formed, and therefore the residue can be disposed of directly and the solution can be purified in the usual way and electrolyzed to yield pure metallic zinc and acid for recycle to the leaching step.

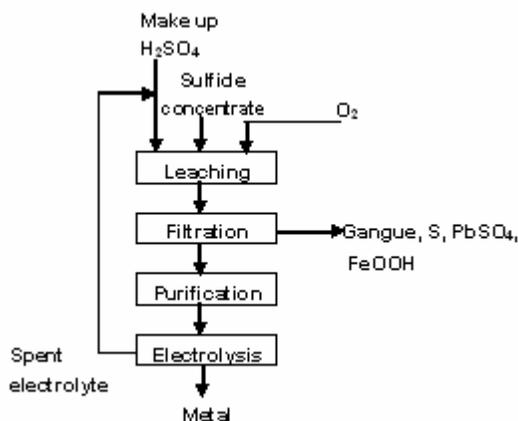


Fig.11 Aqueous oxidation of sulphide concentrates

### Lead

Researchers at Engitec Tehnologies in Milan, Italy found out that lead in galena concentrates was solubilized in fluoroboric acid containing ferric fluoroborate at 80°C liberating elemental sulfur:



Any silver in the concentrate remains in the residue. The solution is then electrolysed in a diaphragm cell where pure compact lead is deposited at the cathode and the ferrous fluoroborate is oxidized at the anode to ferric fluoroborate for recycle Fig. 12:

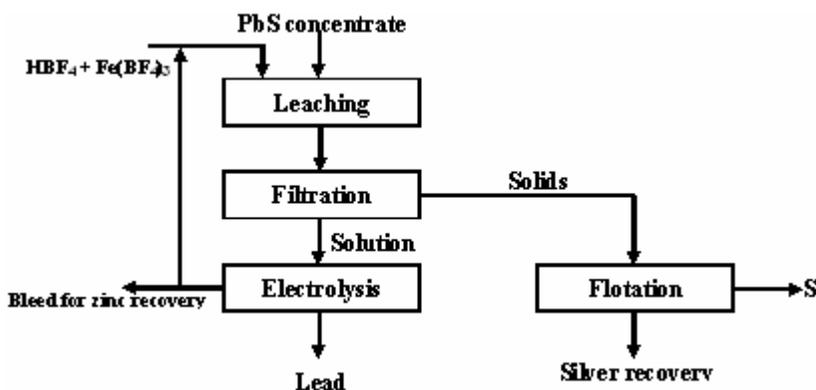
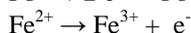
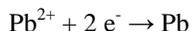


Fig.12 Processing of lead sulfide concentrate by the fluoroboric acid process

Fluoroboric acid,  $\text{HBF}_4$ , is prepared industrially by reacting hydrofluoric acid with boric acid; the reaction is exothermic. It is available only as a 48% solution. Ferric fluoroborate is prepared by reacting the acid with  $\text{Fe}_2\text{O}_3$ . It seems that this is the best method to treat galena concentrates. Zinc had a similar history like lead up to 1980s. It was produced exclusively by roasting the sulfide concentrate to form zinc oxide, thermal reduction of the oxide, then refining the crude metal by vacuum distillation. In 1980, the total hydrometallurgical route, i.e., aqueous oxidation of the sulfide concentrate to get zinc sulfate solution and elemental sulfur was introduced on industrial scale. Copper had also a similar history: at the beginning of the twenty first century the aqueous oxidation of copper sulfide concentrates was also introduced on industrial scale. Will lead follow a similar situation? The present writer is convinced that the answer is yes.

### Nickel

Nickel deposits are two types:

- Sulfide mineral pentlandite,  $(\text{Ni,Fe})_9\text{S}_8$  which may occur alone as in Manitoba or associated with chalcopyrite as in Sudbury. In both cases, the nickel is associated with a large amount of pyrrhotite.
- Low-grade oxide associated with iron oxide, known as laterite.

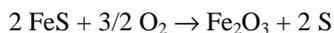
### *Treatment of laterites*

There are a variety of methods used industrially for recovering nickel and cobalt from laterites. One of these, however, has received recently great attention as a result of significant technological development in the design and operation of high pressure autoclaves. The acid pressure leaching process has been in operation in Cuba since 1959. The plant was originally composed of 4 units each unit has 4 vertical autoclaves in which the ore containing 1.3% Ni and 0.12% Co was leached with sulfuric acid at 250°C and 4000 kPa. In 1996, a new unit was added attesting to the validity of this technology.

In Western Australia, three nickel laterite projects are being developed all are using acid pressure leaching. One of these is using four horizontal autoclaves 5 meters diameter and 35 meters long. The autoclaves are titanium lined and divided into six compartments.

### *Treatment of sulfide concentrates*

In the 1960's, INCO operated a small scale plant treating low-grade nickel-containing pyrrhotite, analysing: 0.8% Ni, 57% Fe, 37% S, and the balance gangue. The concentrate was roasted, and the hot calcine was then reduced in a rotary kiln so the nickel was transformed in the metallic state that can then be leached by ammonia. The process generated large amounts of SO<sub>2</sub> and was shut down after few years of operation. However, when pyrrhotite concentrate is suspended in dilute acid and subjected to aqueous oxidation in an autoclave at 110°C and 4000 kPa oxygen partial pressure, the following overall reaction takes place:



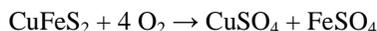
Any nickel (or copper) will go in solution from which it can be recovered by electrolysis while the platinum metals, usually associated with nickel, will be found in the residue from which they can be recovered. This process which was developed at the Mines Branch in Ottawa is much superior to the previous process :

- Elemental sulfur is formed instead of SO<sub>2</sub>
- Nickel is solubilized in a single step (pressure leaching) instead of roasting, reduction, and atmospheric leaching
- Acid consumed in the reaction is equivalent to the nickel solubilized, and this can be recovered in the electrowining step
- There is no need to use an expensive leaching agent such as ammonia

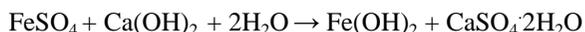
Recently, this technology which was rejected by INCO in the 1960s has been applied recently to treat the newly-discovered sulfide deposits at Voisey Bay in the Canadian North. Using this technology solves the SO<sub>2</sub> problem associated with the treatment of nickel sulfide concentrates, and it is believed that it will have an important future impact not only for the low-grade material but also for the high-grade concentrate.

### **Biohydrometalurgy**

The discovery in 1960s that certain autotrophic microorganisms catalyze the oxidation of sulfides, prompted researchers to apply this knowledge in large scale heap leaching operations. This was also applied to decompose pyrite concentrates containing gold to render gold accessible to cyanidation. This technology, however, cannot be used economically for leaching chalcopyrite concentrates:



since a large amount of oxygen will be consumed, a large amount of lime will be needed to precipitate ferrous sulfate, and there will be an excessive disposal problem of ferrous hydroxide–gypsum mixture:



### Electrometallurgy

Electrometallurgy is the newest field of extractive metallurgy. It is the basic technology for the production of aluminum. As a result of extensive research the energy consumption per kg of aluminium produced has decreased over the years Fig. 13 and the productivity of the cell increased. Aluminum is now the second largest metal produced after iron, taking over the position of copper since 1960. Its price also became less than that of copper since 1940 thus favouring its use in many applications that were usually reserved for copper.

With increased tendency towards pressure leaching to replace smelting of copper concentrates there will be a corresponding increase in electrowinning operations. At present electrowinning has been well established in the major metals industry and has been successful when applied to leaching-solvent extraction process for low-grade copper oxide ores. However, electrowinning from aqueous solutions did not change much through the years and suffers from the following problems:

- Extensive surface area is needed for the electrolytic cells.
- Low productivity because of current density limitations.
- The formation of acid mist due to the liberation of oxygen at the anode.

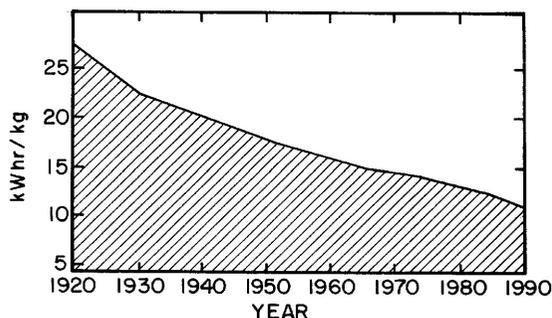
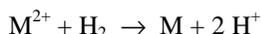


Fig.13 Energy consumption per kg of aluminium produced has decreased over the years

Precipitation by hydrogen under pressure in autoclaves is used industrially for the recovery of pure nickel and cobalt:



The process is competitive with electrowinning, being conducted in closed reactors with high productivity in a small surface area. However, it suffers from the necessity to neutralize the acid by ammonia as soon as it is formed to shift the equilibrium to the right, thus

resulting in the formation of ammonium sulfate which can be marketed as a fertilizer. Copper, however, can be precipitated by hydrogen under pressure at high acidity Fig.14 and this distinct behaviour should be exploited as a substitute for electrowinning.

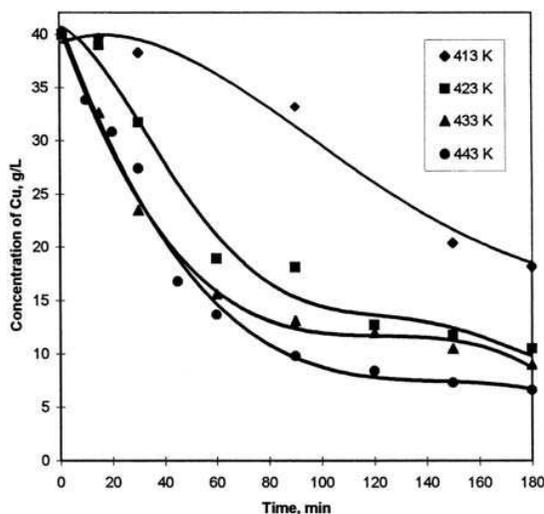


Fig.14 Precipitation of copper from 140 g/L sulfuric acid by hydrogen under pressure

## Conclusions

In the past four decades intensive effort has been conducted to solve the energy problem in the major metal industries which resulted in the flash smelting technology for copper and nickel. However, no serious work was done to solve the sulfur problem associated with these metals. For example, the copper industry seems to be content with producing 3 tons  $H_2SO_4$  per ton of copper and the nickel industry is capturing only a fraction of its  $SO_2$  emissions. The zinc industry on the other hand has already solved this problem by using pressure leaching technology. Gold industry at present is the largest user of pressure reactors for treating refractory gold ores prior to cyanidation and it is expected that this technology will have great impact in the future on the processing of the primary metals copper, nickel, and lead. It is also expected that electrowinning will play an important role in the future as a result of increased use of pressure leaching of sulfide concentrates. However, radical change in this technology is needed to decrease space requirement and increase productivity and probably replacing this technology by hydrogen reduction in autoclaves for copper.

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