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## Nickel-Cobalt-Copper Keynote

## HPAL IN PAST, PRESENT AND FUTURE

By

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## ABSTRACT

Sumitomo Metal Mining Co., Ltd. (SMM) has commissioned two HPAL projects in the Philippines. Both of the plants successfully produce Mixed Sulfide (MS) of nickel and cobalt from low grade lateritic ore and the annual production of nickel is 54,000 tons in the Philippines. Three Australian laterite projects were commissioned in late 1990's. Those projects suffered from low availability of the plant and low recovery of nickel in the ramp up stage. The first challenge of SMM was the Coral Bay Nickel Project which commenced in 2002. The project achieved the first track record to reach the name plate capacity. SMM proved operational reliability of HPAL by the CBNC project. However, we have never terminated our challenge to establish the most competitive HPAL plant in the world. The second challenge of SMM was the Taganito HPAL project, located on Mindanao Island, Philippines. As compared with the CBNC project, the project possesses disadvantages of "Wet Weather" and lower grade ore containing nickel at less than 1.1 %. The plant is designed to produce 30,000 tons of nickel and 2,600 tons of cobalt as MS. Construction of the THPAL plant commenced in 2010 and the plant was mechanically completed in June 2013. The plant has attained name plate capacity since May 2014 with excellent performance in terms of nickel recovery and plant availability. Following the success of the Taganito project, pilot studies have been conducted at CBNC and THPAL aiming at further advancement in the future. Those studies include recovery of chromite, hematite and scandium from the laterite ore. SMM believes those technologies are contributing to the development of lower grade lateritic nickel resources.

## INTRODUCTION

Over the last decade a number of laterite projects have been developed worldwide and are now operating. HPAL (High Pressure Acid Leach) was originally applied to the Moa Bay plant in Cuba. However, the Moa Bay plant was built in the late of 1950's and was not designed for maximum energy conservation. Nickel reserves are largely in the form of laterite ore bodies. The experience gained with large horizontal autoclaves and heat recovery in the gold industry appeared to afford significant operating cost saving over the Moa design. The second generation of the HPAL plant was constructed in Western Australia in the late of 1990's. The three nickel projects located in Western Australia - Bulong, Cawse and Murrin Murrin, had a combined production capacity of 63 k ton per year. That was equivalent to about 6 % of world capacity of nickel production at that time. It was indicative that HPAL is a promising process for laterite deposits. The concept of heat recovery was multi-stage heating and flashing of feed slurry and leached slurry respectively and was similar for each of the Western Australia projects, but the equipment design differed and this proved to have a considerable influence on reliability. Also, for energy conservation, different reactors operating at lower temperature than at Moa were selected for the sulfide precipitation circuit at the Murrin Murrin plant.

The third generation was Ravensthorpe, Goro, Ambatovy, Ramu and SMM (Sumitomo Metal Mining Co. Ltd.) project. Some of these projects developed their own new technology. The Ravensthorpe project installed atmospheric tank leach. The Goro project applied completely new technologies comprised of solvent extraction and pyrohydrolysis of nickel chloride to produce nickel oxide. Many of these projects have undergone very severe commissioning "difficulties", while the two Sumitomo projects have been successful commissioned as shown in Table 1. The first line of CBNC (Coral Bay Nickel Co.) has been in operation since 2005. The production rate was doubled by installation of the second line at CBNC in 2009. THPAL has been in operation since 2013 and is positioned to achieve the name plate capacity of 30,000 ton-Ni in 2015.

Both CBNC and THPAL (Taganito HPAL Co.) produce Mixed Sulfide of nickel and cobalt from limonite ore at an annual production rate of 54 k ton-Ni. The MS is exported to Japan. SMM's Niihama Nickel Refinery in Japan refines this intermediate product to 99.99% nickel metal. Some of the MS is also refined to nickel sulfate crystal at the Harima Division of SMM in Japan.

	Capacity (kT/A)	Start Up	Achieving Design Capacity (Year)	2014 Production (kT)	Cost Index (PJ Cost/A-Nilb)
Murrin Murrin	45	1999	2013	44.1	N.A
Ravensthorpe	45	2008	-	36.4	28.2
Goro	60	2011	-	19.8	>52.9
Ramu	31	2012	-	19.9	30.6
Ambatovy	60	2012	-	37.0	52.9
CBNC	20	2009	2011	24.0	12.6
THPAL	30	2013	2015	26.3	27.1

## Table 1: HPAL Projects Worldwide

## CORAL BAY NICKEL PROJECT

In the year 2000, SMM conducted a bankable feasibility study for the HPAL plant at Rio Tuba, Palawan Island, Philippines, using the stockpiled ore of Rio Tuba Nickel Co. RTN exports raw nickel ore to Japan, and ores below cut-off grade were segregated into stockpiles and conveniently mapped. The CBNC plant was inaugurated in April, 2005<sup>(1)</sup>. The plant was designed to produce 10 k ton of nickel and 750 ton of cobalt per year. It achieved nameplate capacity in the year 2007, merely 2 years after its mechanical completion, when 10,078 ton of nickel was produced.

The expansion project of the CBNC plant started in 2006. In incorporating key improvements, such as tie-in lines of vital solutions and slurries, the availability of the plant was dramatically improved. The mechanical completion was achieved in February, 2009. The plant produced 24,019 ton of nickel in 2014. Figure 2 shows the process flow sheet of the CBNC plant. The flow sheet on the left shows Line-1 and the flow sheet on the right is Line-2. Line-2 is basically designed the same as Line-1. The size of key equipment such as autoclave, thickeners, and reactors are fundamentally the same.

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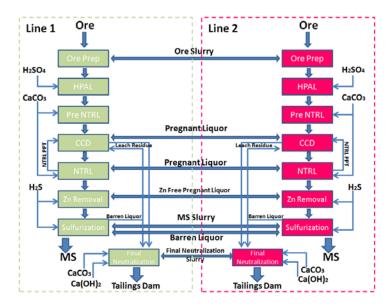


Figure 1: Flow Sheet at CBNC

The process dissolves nickel and cobalt from ore by high pressure acid leach. This is followed by neutralization of excess acid using ground limestone. Nickel and cobalt are recovered as sulfides by precipitation with hydrogen sulfide gas after eliminating zinc from the pregnant solution. Tie-in lines between Line 1 and Line 2 allowed more flexible operation and attained high availability of the plant as described latter.

Based on plant experience<sup>(2)(3)</sup>, nickel recovery of the HPAL process was rather low. Therefore, high nickel recovery was set as one of the goals in the process development. To minimize the nickel loss from the process, intensive test work was carried out at the Niihama Research Laboratory of SMM. Furthermore, the product purity was another concern. The MCLE (Matte Chlorine Leach Electrowinning) process has been in practice at Niihama since 1990, and SMM produces high purity electrolytic nickel from nickel matte by this technology. In the early stages of the process development, several possible flow sheets were considered. The CBNC process, including HPAL and MS precipitation, was selected as the best match to the MCLE process. As a consequence, electrolytic nickel of high purity has been commercially produced from low grade lateritic nickel ore.

## **Development of De-Zinc Process**

Zinc is a critical element that contaminates electrolytic nickel. Zinc normally electro-deposits preferentially to nickel. Several methods to eliminate zinc were examined in the initial stages of the process development. Sulfide precipitation of zinc has been selected, due to the sharp separation from nickel and the availability of hydrogen sulfide produced at the plant site. However, it has been found that local reaction between the metal ions and hydrogen sulfide occurs, which leads to the co-precipitation of some nickel with zinc. The precipitation of nickel accelerates the dissolution of zinc sulfide with acid formed by the co-precipitation. Thus, homogeneous reaction is very important for the complete removal of zinc from the solution<sup>(4)</sup> To achieve a homogeneous reaction, it was found that good dispersion of  $H_2S$  gas in the solution is essential. The concentration of dissolved hydrogen sulfide is properly controlled for the purpose and adapting low temperature at less than  $60^{\circ}C$  prevents co-precipitation of nickel resulting in better selectivity. The nickel loss with zinc precipitate is less than 1 % in the plant practice at CBNC.

## **MS Precipitation Circuit**

MS precipitation was a conventional technology for separation of nickel and cobalt from other metals. Solvent extraction and precipitation of a mixed hydroxide have been practiced in several laterite projects. Organic loss and raffinate treatment seemed to be disadvantages when solvent extraction treats a large volume of process liquor. Mixed sulfide precipitation is more selective than mixed hydroxide precipitation for some key impurities especially like manganese. Therefore, CBNC selected mixed sulfide precipitation to concentrate the solubilized nickel and cobalt. In the early of 2000's, two of the HPAL plants in operation used sulfide precipitation for primary concentration of nickel. These are Moa Bay<sup>5)</sup> and Murrin Murrin<sup>6)</sup>. Both plants prefer relatively high temperatures in the sulfide precipitation reaction. The purpose of high temperature operation is the high recovery of

nickel and the fast kinetics. However, a low reaction temperature of 80°C has been selected for the CBNC process.

It appears that slow kinetics is preferred for preventing scale formation in the reactor, while a relatively long retention time is required for sufficient nickel recovery under the low temperature conditions Therefore, test work was carried out in bench and pilot scale equipment to prevent scale formation while achieving high nickel recovery. Those studies led to application of MS sulfurization under relatively high  $H_2S$  pressure as described later.

## THPAL PROJECT

The objective of the Taganito HPAL project is to produce 30 k ton of nickel and 2.6 k ton of cobalt for 30-years utilizing lateritic nickel ore at Taganito Nickel Mine Co., Mindanao Island, Philippines. The MS product is further refined to electrolytic nickel or nickel sulfate at the SMM plants in Japan. The major challenge at SMM was to achieve feasible operation utilizing the lowest grade nickel ore used in laterite projects. The process and engineering design of the plant was carefully optimized based on lessons learned in the previous HPAL project at CBNC. Those developments enable high recovery of nickel, high plant availability and reduction in operating cost.

## **Nickel Production**

Figure 2 shows the flow sheet of the THPAL plant. The ore preparation and HPAL circuits consist of two lines to reduce the risk of total plant shutdown. While the downstream facilities following HPAL were constructed as only a single train, since reliable operation has been established in the practice of CBNC. This concept contributes to reduction in capital cost of the THPAL project.

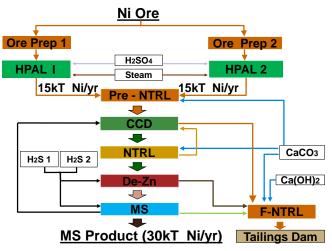


Figure 2: Process Flow Sheet of the THPAL Plant

The plant has been commissioned since mid of 2013. Capital cost of the Taganito project is 1590 MUSD, which is more expensive than 510 MUSD for the 20 k ton plant at CBNC. This is due to the larger production capacity, extensive civil work, comprehensive infrastructure and delay in the implementation. Wet weather and a local security issue were the major causes of the delay. However, the plant was successfully commissioned and the annual production in 2014 was 26,320 ton of nickel as shown in Figure 3. It is notable that the production rate in August 2014 was 3,022 ton of nickel, which exceeded the design capacity.

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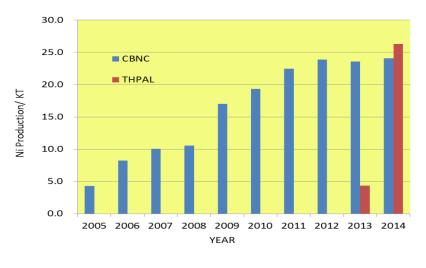


Figure 3: Production Rate of Nickel at CBNC and THPAL

## **Recovery of Nickel**

Table 2 shows the nickel and magnesium contents of ores treated at both the HPAL plants in the Philippines. Although magnesium is low at THPAL, the content of nickel is very low. The THPAL ore required better extraction of nickel in acid leach and better recovery of nickel in MS precipitation compared with CBNC.

Operation CBNC THPAL				
Operation	CBNC	THPAL		
	(2005-2014)	(2014)		
Ni (%)	1.25	1.00		
Mg (%)	1.69	1.10		

## Table 2: Ni and Mg Content of Ore at CBNC and THPAL

To maximize extraction of nickel from ore, computer simulation utilizing data obtained in the existing practice was widely applied to autoclave design, including agitators and position of the feed pipe. The retention time in acid leach is prolonged and the structure of the compartments is modified to effectively prevent short circuiting of unreacted ores. These modifications achieve an excellent extraction rate of nickel and prevent corrosion inside the autoclave by rapid diffusion of sulfuric acid.

The partial pressure of H<sub>2</sub>S gas in the MS reactor greatly affects the recovery of nickel. Temperature and purity of H<sub>2</sub>S gas are major factors dominating the partial pressure of H<sub>2</sub>S gas. The purity of H<sub>2</sub>S gas used is maintained above 98 %. An increase of the partial pressure of H<sub>2</sub>S gas increases solubility of H<sub>2</sub>S in solution and enhances the recovery of nickel and cobalt from solution. Decreasing the temperature also increases the solubility of H<sub>2</sub>S gas. It appears that low temperature is desirable for recovery and energy conservation.

Table 3 shows the condition in the MS circuits in the existing operations. It is noticeable that THPAL adopts high pressure and low temperature for the sulfurization reaction as compared with others. The circuit utilizes four vessels in series and the operating pressure is maintained at a maximum of 350 kPaG. The temperature is relatively low compared with other operations resulting in better recovery of nickel and cobalt and effective prevention of scale formation.

	•	
Operation	Temperature (°C)	Pressure (kPaG)
Moa Bay	120	1000
Murrin Murrin	95	100
CBNC	80	270
THPAL	70	350

#### **Table 3: Comparison of MS Precipitation Conditions**

These applications achieve total recovery of nickel of more than 90 % in the plant practice. The total recovery of nickel is reported generally at around 85-90 % with a feed assay of nickel of more than  $1.5\%^{(2)(3)}$ . This shows that THPAL is achieving feasible practice utilizing the lowest grade of nickel laterite in the world.

## **Plant Availability**

The HPAL plant generally suffers from low availability due to the heavy duty of the pumps and valves and corrosion. Meanwhile, scale control methods in the autoclave and in the MS reactors have been established in the CBNC operation. Furthermore, maintenance of valves has also improved and it is not a constraint on plant availability. Ultimately the remaining concern was erosion in the heat recovery system of acid leach. The erosion of steam recovery pipes between flash tanks and heater vessels is one of critical issues for continuous operation of HPAL. The steam generated in the flash tanks is supplied to the corresponding heater vessels for heating the slurry. A thorough investigation of the heat recovery system was undertaken based on the operational data at CBNC. This investigation indicates that decreasing the pipe velocity of steam prevents erosion of pipes and accelerating the condensation of acid mist in the flash tank decreases carry-over of the acid to the heater vessel resulting in less corrosion. The design of the heat recovery system has been modified based on this concept. A similar test to CBNC<sup>(1)</sup> on acid carry over was conducted in the pre-commissioning stage of THPAL. The amount of acid carry over was found to decrease by 60% to 70% compared with CBNC.

Other evidence can be seen in measurement of the thickness of the steam recovery pipe. The reduction in thickness of the pipe by erosion is remarkable in the half year operation at CBNC. However, the reduction rate is dramatically improved in the new design at THPAL. These pipes are inspected every half year in the scheduled shut down maintenance. The piping under the most erosive condition is normally subject to replacement within one year at CBNC. Although there are the occasional exceptions, the replacement of steam recovery pipes has not yet been carried out since the commissioning.

These improvements keep the plant from unscheduled shut down, and have contributed to high availability as shown in Figure 4. Although there are many mechanical issues such as poor installation and incorrect material selection in the initial stage of the ramp up, the THPAL plant achieves similar or rather higher availability of the plant compared with CBNC.

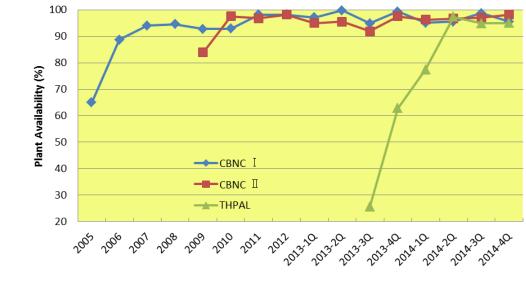


Figure 4: Comparison of Plant Availability

## **Recycle of Sulfur**

The recycle of sulfur was a solution for achieving completely integrated operation at SMM. High purity sulfur is purchased for synthesizing  $H_2S$  gas at the CBNC plant. Sulfur from MS is recovered as elemental sulfur by the MCLE process at Niihama. However, the sulfur recovered is not able to be refined to high purity because of contamination from other source. The elemental sulfur produced is sold at a relatively lower price than that of high purity sulfur. The price difference between the sulfurs was subject to settle. The synthesizing process for  $H_2S$  requires a catalyst at CBNC and deterioration of the catalyst is a major concern. Therefore, expensive high purity sulfur is fed to the process. The catalyst is also expensive and is required to be replaced every 5 years. In addition to this, the regular transportation between Taganito and Niihama can be used, if elemental sulfur produced at Niihama can be recycled to THPAL.

Thus, SMM investigated the "Girdler"  $H_2S$  gas generation process in which no catalyst is required. This process is common for  $H_2S$  gas production irrespective of minor impurities in recycle sulfur. The reaction takes place by bubbling hydrogen gas through liquid sulfur at 470°C and 700 kPaG. The catalysed process synthesizes  $H_2S$  at a temperature of 120°C at atmospheric pressure. It has been found that the high temperature process has advantages of cost saving in both capital and maintenance and tolerance to impurities. The "Girdler"  $H_2S$  gas generation process has been installed at the THPAL and the recycle of sulfur has been practiced since January 2015.

## **TECHNOLOGY IN THE FUTURE**

SMM has undertaken intensive research work in connection with the HPAL technology in last 15 years as described previously. The requirement of the process in the future appears to be the capability of treating still lower grade nickel laterite and recovery of other valuables contained in the laterite. In order to process lower nickel grade, loss of nickel and volume of leach residue must be minimized, that is zero emission in laterite development. The capital expenditure for the tailing storage facility is huge. Leach residue is constituted of mainly hematite, and utilization of the hematite in iron industry is an interesting subject for the development of a nickel laterite deposit.

Research work at SMM also focuses on products such as nickel hydroxide and nickel powder. SMM has produced electrolytic nickel, chemicals such as sulfate and chloride and ferro-nickel. A wide variety of products is advantageous for SMM's nickel business. These investigations have been conducted at pilot scale and the cost of the pilot facilities has been greater than 20 MUSD over the past few years.

## Hematite Residue

The study on the utilization of leach residue as a raw material for a steel mill commenced in 2007 in collaboration with a Japanese steel mill. However, the specification of chromium for the Japanese steel mill is at less than 0.01 % as shown in Table 4. Mineralogical investigation indicated that some chromium is dissolved in goethite matrix at more than 0.3 %. The chromium dissolved is not separated from the goethite phase and is transferred to the hematite phase during acid leach. Usage of hematite residue for blast furnace as raw material seemed to be difficult due to high chromium content. Another attempt was the application of a reduction furnace. It was technically possible, but the capital cost of a reduction furnace is high and requirement of natural gas limits the location of the plant. As a consequence, application of the reduction furnace was concluded to be unfeasible.

On the other hand a stainless mill is able to utilize the hematite residue, because of no limitation on chromium. Table 4 shows specification of raw material for steel mill and stainless mill. A bench scale study on leach residue successfully upgraded the iron content of the hematite residue. Subsequent to establishment of the basic flow sheet, a pilot plant was constructed at a cost of 6 MUSD in August, 2014. The pilot facility has a capacity to produce 10 k ton of hematite in a year. Hematite sample was first harvested in March, 2015. Typical analysis is presented in Table 4.

-					-
Element (%)	Fe	Si	Al	S	Cr
Steel Mill	56-63	<2.7	<1.4	<0.1	<0.01
Stainless Mill	>56	<2.6	1.3	-	-
Leach Residue	33.3	2.8	1.5	NA	1.5
Hematite Residue	62.4	1.7	1.1	1.4	1.2

#### Table 4 Specification of Raw Material for Steel Industry

## Chromite

Chromite is another value in lateritic ore. Recovery process for the chromite has been studied since 2009. The world market size for chromite is around 25 M ton. The price of chromite varies widely between 500 and 150 USD per ton. The studies have been advanced to pilot scale in August 2013 after developing the basic flow sheet. The pilot facility was constructed at a cost of 5.2 MUSD at the CBNC plant site. The facility was designed to produce 9000 ton of chromite annually. Subsequent to solving problems unknown in laboratory, the pilot test has advanced to producing chromite at semi-commercial level. Since then, 2500 ton of chromite have been produced by the pilot facility. Table 5 shows a typical assay of chromite shipped to a potential customer. The chromite content

and ratio of Cr/Fe are important specifications for a stainless mill. It is obvious that chromite produced at CBNC is well within both specifications.

Element (%)	$Cr_2O_3$	FeO	Cr/Fe	$AI_2O_3$	SiO <sub>2</sub>	MgO	S	Р
Stainless Mill	>40		≧1.2	≦17	≦3.5	≦12	≦3.5	≦0.02
THPAL	50.4	26.3	1.7	1.4	1.1	8.8	0.01	0.01

Table 5 Assay of Chromite Produced b	by the CBNC Pilot Plant Operation
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## Scandium

It was found that lateritic nickel contains trace scandium when Niihama Nickel Refinery processed MS supplied from Nonoc, Philippines. Electrolytic nickel and cobalt were produced in 1980'S from MS at Niihama and a small amount of scandium was detected in the process solution. Up to date, scandium has been obtained from various ores, tailings and residue at previously exploited mines as a by-product. The main application areas are high strength Al-Sc alloys and solid oxide fuel cells. The world market is 10-15 ton. However, the supply of scandium is very scarce and the price is around 2000\$ for 1 kg of  $Sc_2O_3$  at 99.9% purity. The presence of scandium at more than 50 ppm in laterite has the potential to be a prominent source.

Since commissioning of the CBNC plant, the investigation of the recovery of scandium from the process liquor was commenced in 2006. Test work at laboratory scale has been been conducted to develop the basic flow sheet. Following the trace of scandium content in ores for 2 years, a pilot facility was constructed in January 2014. The capacity of the facility is designed for the production of 100 kg of scandium annually.  $Sc_2O_3$  with 99.9% purity has been produced since January 2015. A typical analysis of scandium oxide is shown Table 6.

## Table 6 Typical Impurities of Scandium Oxide

Sc <sub>2</sub> O <sub>3</sub> (%)	Si (ppm)	Al(ppm)	Fe(ppm)	Ni (ppm)
99.93-99.94	30	20	20	120

## CONCLUSION

Two HPAL projects have been successfully operated by Sumitomo in the Philippines. The CBNC plant produced 24 k ton of nickel in 2014. The THPAL plant was commissioned in 2013 and the plant produced 26 k ton of nickel in 2014. Based on these successes, SMM has been continuously advancing the technology of HPAL to maintain competitiveness in the development of laterite resources.

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