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Recovery of Nickel and Cobalt From Low-Grade Domestic Laterites

UNITED STATES DEPARTMENT OF THE INTERIOR

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Recovery of Nickel and Cobalt From Low-Grade Domestic Laterites

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ILLUSTRATIONS

RECOVERY OF NICKEL AND COBALT FROM LOW-GRADE DOMESTIC LATERITES

by

R. E. Siemens,[1] P. C. Good,[2] and W. A. Stickney[3]

ABSTRACT

A process is being developed by the Bureau of Mines to selectively recover nickel and cobalt from low-grade domestic laterites. In laboratory evaluation of the process, the oxides in the laterite were selectively reduced with carbon monoxide at temperatures from 350° to 600° C. For material containing more than about 5 percent magnesia, pyrite additions or post-reduction heat treatments were necessary to achieve satisfactory nickel and cobalt extraction for this range of reduction temperatures. Multistage leaching of the reduced material at ambient temperature and pressure in the presence of oxygen, ammonium sulfate, and ammonium hydroxide extracted up to about 90 and 85 percent of the contained nickel and cobalt, respectively.

The nickel was selectively recovered from the leach solution by solvent extraction and was then stripped from the loaded organic with dilute sulfuric acid to provide a nickel-rich electrolyte. Treatment of the raffinate with hydrogen sulfide resulted in the recovery of cobalt as a sulfide. The only contaminant in solution was magnesium which was removed by ion exchange or precipitation. In this process the energy requirements are relatively low and except for minor operation losses, only coke and oxygen are consumed.

INTRODUCTION

Although sulfide ores have been the major source of nickel to date, the majority of known and anticipated reserves are lateritic deposits. However, the minimum economic grade limit for laterites is appreciably higher than for sulfide ores since the laterites are not amenable to concentration. The lower economic grade limit for laterites is determined by the process and varies from 1.4 to 2.0 percent nickel.

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[2] Reseach chemist.

[3] Research supervisor.

The current commercial processes used to treat laterites are reviewed and referenced in (2).[4] More specifically, pyrometallurgy processes such as those of LeNickel (2, 16) and Hanna Nickel Smelting Co. (2, 7) are limited to relatively high-grade (2.2 to 2.5 percent nickel for LeNickel; 1.4 percent for Hanna), low-iron silicate ores because of the large quantity of electrical energy required. High-temperature, high-pressure sulfuric acid processes (2, 5) are restricted to low-magnesia limonitic ores to avoid excessive acid consumption. The Caron process (2, 6), or modifications thereof, has a wider range of application but is subject to low recoveries when applied to silicate ores or ores with high magnesia content. In many of the current commercial processes, nickel is not produced in pure form. Most of the pyrometallurgy processes produce ferronickel while chemical methods such as the Caron process yield nickel oxide.

The need to develop new processes to economically treat the extensive laterite resources of the world to meet future nickel demands has stimulated active research in many countries. Most of the new processes proposed employ hydrometallurgical techniques or a combination of pyrometallurgical and hydrometallurgical processes. Only one of these processes to date has been considered to be sufficiently economical to warrant commercial application (4, 14-15). This process, developed by Sherritt Gordon Mines Ltd., is a modification of the Caron process and recovers cobalt and nickel separately by precipitation with the nickel subsequently recovered as metal powder by pressure reduction with hydrogen. Marinduque Mining and Industrial Co. will use the process to recover nickel from Philippine laterites. Pilot plant operation over a 28-day period resulted in 82.8 percent nickel recovery from feed containing 1.33 percent nickel and 0.12 percent cobalt (9).

The Bureau of Mines is concerned with developing domestic production of nickel. Previous research by the Bureau (1) demonstrated that ferronickel could satisfactorily be produced from Philippine laterite. However, because of the low grade of domestic laterites, new efforts were directed toward the development of an efficient, nonpolluting hydrometallurgy process with low energy requirements. This report describes a process that selectively extracts high percentages of nickel and cobalt from low-grade laterites in which the energy requirements are relatively low and all chemical reagents are recycled.

MATERIAL DESCRIPTION AND PREPARATION

The primary material investigated was from a 5-ton sample obtained from a pit in the Pine Flat deposit in northern California and southern Oregon. The sample is a typical laterite consisting primarily of limonite with lesser amounts of serpentine, altered olivine, a wadlike manganese oxide, and chromite. Petrographic and microprobe analysis of the material indicated that about 23 percent of the contained nickel and essentially all of the cobalt were in the manganese oxide while the remainder of the nickel was uniformly distributed in the laterite. The wad contained 28 percent manganese and

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[4] Underlined numbers in parentheses refer to items in the list of references at the end of this report.

13 percent of both nickel and cobalt. Most of the plus 5.08 cm (2 inch) serpentine, which contained 0.3 to 0.4 percent nickel, was removed from the sample. The remainder was then crushed to minus 6.35 mm (1/4 inch), split, and sampled. A chemical analysis of a representative portion contained, in weight-percent: Ni, 0.97; Co, 0.2; Fe, 36.1; Mn, 0.5; Cr, 1.87; MgO, 7.22; SiO_2, 21.5; Al_2O_3, 4.11; CaO, 0.08.

The free-moisture content of this sample was 21.3 percent. As is typical for laterites, a size analysis of the sample indicated little concentration of nickel or cobalt in any size fraction. The plus 2.362-mm (8 mesh, Tyler) fraction primarily consisted of low-value serpentine.

The material was pelletized on a disk pelletizer before reduction to control dust loss and then dehydrated at 350° to 600° C. At these low temperatures, there is essentially no decomposition of the silicates and the only dehydration, 13.5 percent, is that of the limonite.

BATCH TESTS

Reduction

Batch-reductive roasting of 100- to 300-gram charges of minus 6.35-mm (1/4-inch) pellets was done in a 4.45-cm-inside-diameter (1-3/4-inch) vertical tube furnace. Carbon monoxide of 99 percent purity (cylinder) or that prepared by passing oxygen through a coke bed at 1,100° C was found to be the most selective reductant and was particularly effective for the temperature range used. For a 300-gram charge, optimum extraction was achieved after a 40-minute reduction with a 600-ml per minute flow of carbon monoxide. The net reduction reaction was determined by differential thermal analysis to be slightly exothermic, producing about 11 to 14 kcal/kg (20 to 25 Btu/lb).

It is difficult to evaluate selective reduction in complex systems such as laterites because of inaccuracies in distinguishing between metals and oxides. The degree of selectivity was judged by the degree of chlorination extraction achieved because all the metals reduced in the laterites form soluble chlorides. This approach was found by Queneau (13) to be comparable to other methods. Maximum metal solubility was attained by sparging chlorine gas through an agitated slurry containing about 20 percent reduced solids at 80° to 90° C. The reaction was determined to be complete when the pH reached 1.8 to 2.0. On this basis the following percentages of the contained metals were found to be reduced under optimum extraction conditions: Ni (up to 94.6 percent); Co (up to 94.3 percent); Fe (10 to 15 percent depending on reduction temperature); Mn (97 percent); and Cr (<0.8 percent).

The magnesia content appears to influence the reduction conditions necessary to achieve good extraction. As shown in figure 1, nickel extraction with the chlorine leach was quite poor over the entire temperature range investigated without special treatment of the ore. However, nickel extraction can be improved significantly in this laterite with addition of pyrite. The effectiveness of pyrite in improving extraction was pointed out by Caron (6) for hydrated nickel silicates and more recently by the Bureau of Mines (3) for

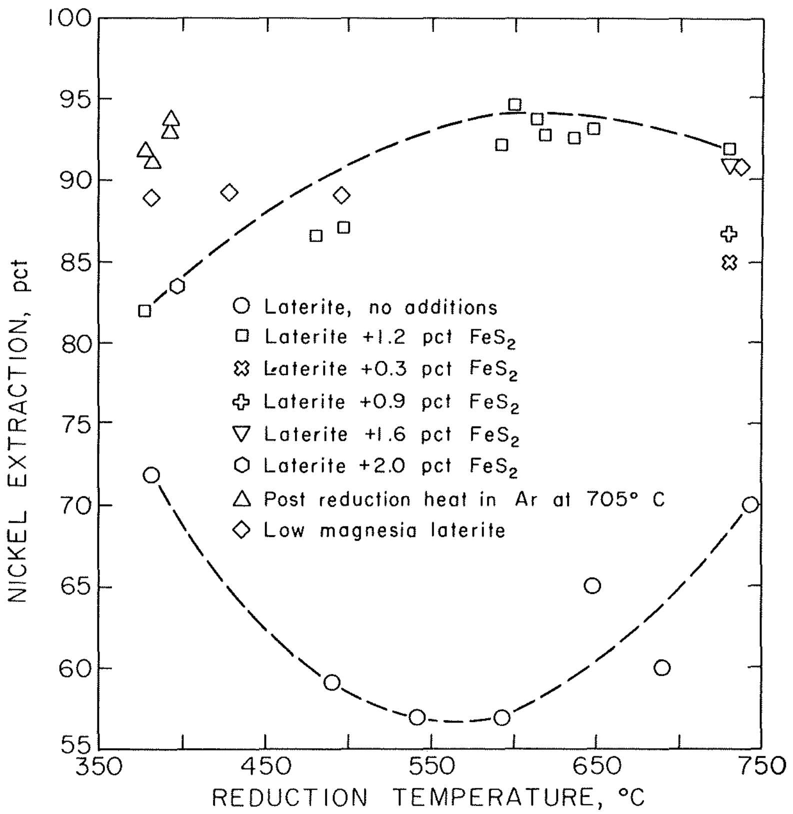

FIGURE 1. - Effect of reduction conditions on nickel extraction from Pine Flat laterite.

serpentine material, although neither suggested a mechanism and in particular did not relate the effect to the magnesia content. The reduction temperatures for the work of both (6) and (3) are higher than those used in this research. As shown in figure 1, reasonably good extraction (89 percent or better, depending on reduction temperature) can be achieved without pyrite additions for laterite essentially identical in content to Pine Flat laterite (Ni, 0.99 percent; Co, 0.28 percent; Fe, 39.9 percent; Mn, 0.47 percent; MgO, 5.37 percent) but containing about 2 percent less magnesia. These results suggest that with a magnesia content sufficiently high at a given reduction temperature, reduced nickel may be involved in a secondary reaction with magnesium and silicon that renders it inert in the subsequent leach. The sulfur from the pyrite apparently impedes this secondary reaction.

Further evidence of the interrelation of magnesia and pyrite content on nickel extraction was supplied by tests with other materials. For example (1) addition of 2 percent pyrite to Southern Oregon laterite (Ni, 0.75 percent; MgO, 6.6 percent; and Fe, 33 percent) resulted in an 8.75-percent increase in nickel extraction; (2) addition of 1.5 percent pyrite to saprolite (Ni, 1.3 percent; MgO, 28 percent; and Fe, 13 percent) resulted in a 250-percent increase in nickel extraction; and (3) addition of up to 10 percent pyrite to serpentine (Ni, 0.47 percent; MgO, 41 percent; and Fe, 6 percent) resulted in a 230-percent increase in nickel extraction. On the other hand, 95 percent of the contained nickel was extracted from Philippine laterite (Ni, 1.15 percent; Fe, 47 percent; and only 1.25 percent MgO) with or without pyrite additions during reduction.

Also of interest and illustrated in figure 1 is the attainment of better than 90 percent nickel extraction after reduction at only 380° to 395° C followed by postheating in an inert atmosphere at 705° C. This result was consistently achieved for either Pine Flat laterite or the material with lower magnesia content.

The results of chlorine leaching, figure 1, were useful as an analytical tool in evaluating the reduction process and in illustrating the optimum potential recovery to be expected from this laterite with the reduction conditions used. Although the extraction obtained with chlorine is excellent, the chlorine leach is not selective, the chlorine consumption is high, and subsequent impurity removal lowers the net recovery and results in the discharge of chloride contaminated effluents. Consequently, an oxidizing ammonia leach was employed, utilizing the same optimum reduction conditions established with the chlorine leach. Carbon monoxide was still used as the reductant since the high nickel and cobalt extraction obtained with its use with the chlorine leach seemed to be near optimum. Hydrogen-containing reductants such as producer gas proved to be not as effective as carbon monoxide when used for reduction of laterites at 350° to 550° C. Evaluation was made of the effect of carbon dioxide in the reduction furnace on extraction, since it is a reaction product. As shown in figure 2, nickel and cobalt extraction were slightly lowered when carbon dioxide was present in the reduction gas. These results were obtained with the oxidizing ammonia-ammonium sulfate leach applied to laterite containing 1.2 percent pyrite and reduced at 530° C. Similar results were obtained for reduction temperatures from 380° to 700° C. These results indicate than an excess of carbon monoxide would be necessary during reduction.

Since the reduced material is extremely pyrophoric, it was cooled in argon to below 200° C, then discharged under either water or leach solution.

Leaching

The leaching was performed in either a boiling flask agitated by a wrist-action shaker or in a modified commercial Waring blender.[5] Leach slurry batches consisted of 1/2 or 1-1/2 liters and contained about 20 percent solids and ideally, about 100 gpl ammonium hydroxide and 300 gpl ammonium sulfate. The leach vessel was sealed to prevent loss of ammonia and to obtain better control of the leach process. A thermocouple and sparger for introducing oxygen were extended to within about 1.27 cm (1/2 inch) of the vessel bottom. The oxygen

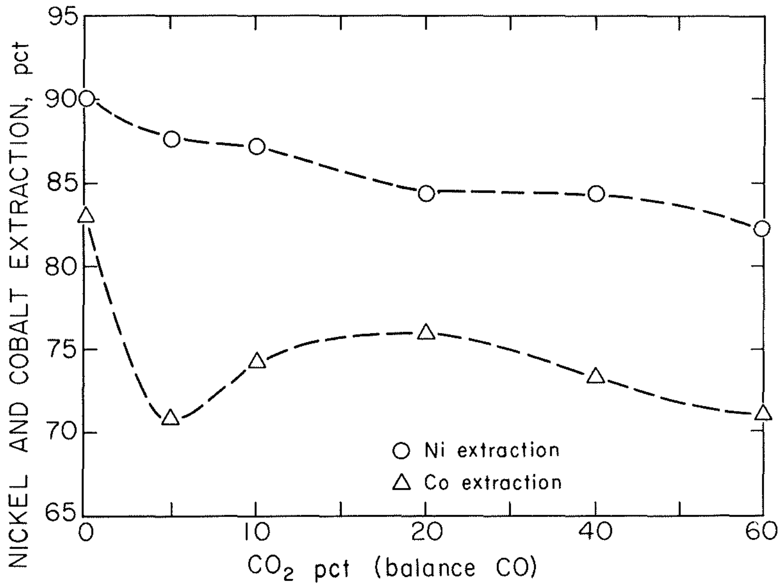

FIGURE 2. - Effect of carbon dioxide in reduction gas stream on nickel and cobalt extraction.

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[5] Reference to specific equipment (or trade names or manufacturers) does not imply endorsement by the Bureau of Mines.

was carefully metered with a flowmeter and the exit gas either passed through a bubbler or into a mercury manometer. The reactions during leaching were monitored by watching a chart recording of the thermocouple voltage. The reactions were slightly exothermic (~8° C temperature rise in a 1/2-liter slurry) and were determined to be complete when there was no further increase in temperature. The reaction also could be determined to be complete at the first sign of bubbles in the exhaust bubbler or by an increase in pressure in the manometer. During the reaction the pressure became lower than the ambient pressure which indicated that all of the oxygen introduced was consumed.

Dissolution or precipitation of the reduced metals in the oxygenated ammonia-ammonium sulfate solution proceeds by a process of oxidation wherein the iron initially dissolves as ferrous ions, the nickel and cobalt are oxidized and form soluble ammine complex ions, and most of the other metals form insoluble oxides. A minor amount of magnesium is dissolved as a sulfate. At a higher oxidation potential, the dissolved iron is oxidized and precipitates as ferric hydroxide. This precipitate occludes nickel and cobalt, a fact that emphasizes the importance of selective reduction. The following simplified equations illustrate the chemical reactions involved:

$$\mathrm{Ni} + 1/2\ \mathrm{O_2} + 4\mathrm{NH_3} + \mathrm{H_2O} + (\mathrm{NH_4})_2\mathrm{SO_4}$$

$$= [\mathrm{Ni(NH_3)_6}]\ \mathrm{SO_4}\ \text{(soluble)} + 2\mathrm{H_2O}.$$

(This particular equation illustrates the formation of nickel hexammine. A similar equation could be written for cobalt. In general for nickel, $Ni^{++} + xNH_3 = Ni(NH_3)_x{}^{++}$ where x = 2 to 6.)

$$\mathrm{Fe} + 1/2\ \mathrm{O_2} + \mathrm{H_2O} = \mathrm{Fe^{++}} + 2(\mathrm{OH})^-,\ \text{(soluble)}.$$

$$4\mathrm{Fe^{++}} + \mathrm{O_2} + 2\mathrm{H_2O} + 8\mathrm{OH^-} = 4\mathrm{Fe}\ (\mathrm{OH})_3\ \text{(insoluble)}.$$

The iron oxidation should take place in solution to avoid formation of a slime film on the surface of the particles that would interfere with the dissolution. The iron oxidation can be controlled to some extent by control of the oxygen feed rate; nevertheless, slimes seem to form on the surface of the particles. These slimes can be destroyed by repulping the filter cake. In fact the best extraction was obtained with controlled oxidation coupled with multistage leaching wherein the pulp was attritioned prior to each leach. For a 1/2-liter slurry containing 20 percent solids the best extraction was obtained with a three-stage leach with oxygen sparged through the slurry at 50 ml/min for 15 minutes each leach.

As with other ammonia leach processes, such as the Caron process and Sherritt Gordon's process, the oxidizing ammonia leach described in this report must be done in the presence of excess ammonia to prevent precipitation of nickel and cobalt as hydroxides. Numerous tests were made in which the ammonia and ammonium sulfate concentrations varied from 5 to 40 percent of the leach volume. The best results were obtained with the ammonium hydroxide concentration at 100 gpl and the ammonium sulfate concentration at 300 gpl. The nickel extraction remained high over a wide range of ammonia-ammonium sulfate concentrations but cobalt extraction dropped sharply with only slight changes in optimum concentrations.

Preliminary tests indicated that elevated leach temperatures (up to 80° C) were not beneficial to the extraction; therefore, leaches were conducted at ambient temperature and pressure. Leach pulps were maintained at 20 percent solids or less.

Most ammonia leach processes employ thickeners in liquid-solid separation. Although in batch tests filtration was more convenient, settling characteristics were evaluated. Equal proportions of 1 percent solutions of Polyhall M159 and Guartec 1018 flocculants were necessary to initiate settling. The initial settling rate of the leached solids was about 17.5 m/hr (57.5 ft/hr) and the overall rate to complete settling was 2.4 m/hr (7.9 ft/hr). No problems with filter blinding or excessive filtration time were encountered in separating the solids from the pregnant solution by filtration. Up to 1.5 gpl nickel and 0.30 gpl cobalt were contained in the first filtrate.

Solvent Extraction and Electrowinning

The general technology employed by SEC Corp. (8, 10-11) in recovering nickel and copper from a copper refinery bleed stream and the research investigations by General Mills Chemical Inc. (12) on liquid ion exchange recovery of nickel from ammonia-ammonium carbonate leach liquors were incorporated into the process.

Preliminary investigations on separating nickel and cobalt involved contacting the pregnant liquor with General Mills' LIX-64N dissolved in Napoleum 470, a Kerr McGee product. Adequate contact was achieved by stirring the mixture for 2 minutes in a high-speed mixer. The organic and aqueous phases were then separated in a separatory funnel.

The results of single-stage extraction studies on solutions containing 0.32 gpl nickel and 0.06 gpl cobalt are presented as a function of leach solution pH in figure 3. The pH was adjusted with sulfuric acid or ammonium hydroxide as required. The ordinate is the ratio of the nickel concentration in the organic to the sum of the nickel concentrations in the organic and in the raffinate. The organic-to-aqueous ratio is expressed as percent organic in the figure (the balance was aqueous solution). These results show that essentially all of the nickel can be recovered in two or three extraction stages at the initial solution pH. In fact, a two-stage extraction with 1.5 percent LIX-64N in a 1:1 ratio with leach solution provided a nickel barren raffinate (<0.001 gpl). The selectivity of the extraction with LIX-64N is also evident in figure 3. Little cobalt is extracted since the oxidizing ammonia leach dissolves cobalt as the trivalent hexammine. Although divalent cobalt will readily load on LIX-64N, trivalent cobalt will not. The nickel concentration was increased by a factor of 9.6 after a single-stage extraction from the leach solution of pH 9.6.

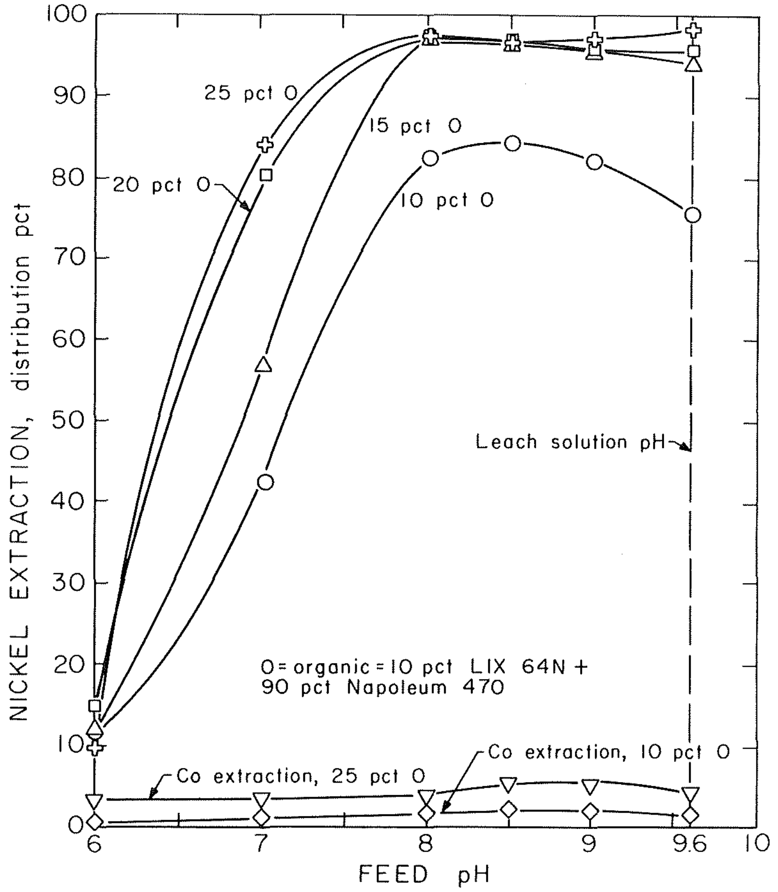

FIGURE 3. - Nickel extraction from ammonium hydroxide-ammonium sulfate leach solution.

The nickel was effectively stripped from the loaded organic in three stages with a solution containing only 1.5 gpl sulfuric acid.

Contact of the raffinate with hydrogen sulfide resulted in the recovery of cobalt as a sulfide. Minor concentrations of magnesium were then quantitatively removed by adding a small amount of phosphoric acid to form precipitate or by selectively removing the magnesium from solution with an ion exchange resin, Amberlite IRC-50.

Two experimental electrowinning cells have been tested with synthetic solutions containing 30 to 60 gpl nickel. The first cell has three nickel cathodes comprising 13.935 dm^2 (1.5 ft^2) and four lead anodes with an electrode separation of 4.216 cm (1.66 inches). Cell operation involved the principles used by the SEC Corp. (8, 10-11). A major difference is that the agitation is provided by air bubbles injected into the electrolyte from tubing lying lengthwise under the cathodes rather than by a directed flow of electrolyte. Electrolyte, consisting of nickel sulfate at pH 2.5, was automatically fed by a pH controlled monitor to keep the cell solution at pH 1.8 to 2.0. The electrolyte was maintained at 50° C by immersion heaters. The best test results to date, with a solution containing 60 gpl nickel, were obtained with a 5-hour run at 3.3 volts, for which the current efficiency was 66 percent and the power consumption 4.55 kW-hr/kg (2.1 kW-hr/lb). The current density was about 2.15 amp/dm^2 (20 amp/ft^2) and the deposition rate averaged 0.35 g/min. The spent electrolyte contained about 2 to 3 gpl less nickel than the feed solution. The specific resistivity of the electrolyte was 12.66 ohm-cm/cm^2 at 50° C and the voltage drop due to ohmic resistance was calculated to be 1.1 volts. After the 5-hour run, the measured anode potential was 1.65 volts and the cathode potential was minus 0.5 volt.

The other cell has only two nickel cathodes comprising 9.29 dm^2 (1 ft^2) and three lead-10 percent antimony anodes with an electrode separation of 6.35 cm (2.5 inches). The cathodes are isolated with woven polypropylene diaphragms. Preliminary runs with this cell, at nearly the same current density of the above cell but at 4.0 volts rather than 3.3 volts, resulted in a 36.2-percent increase in deposition rate, 22.4 percent less power consumption, and 25 percent greater current efficiency. Current efficiencies above 95 percent were obtained with the diaphram cell operating with the catholyte at pH 3.5 to 4.0. Under these conditions the power consumption was 3.74 kW-hr/kg (1.7 kW-hr/lb) at a current density of 1.9 amp/dm^2 (17.8 amp/ft^2) and the deposition rate was about 0.45 kg/day (1 lb/day) per square foot of cathode. For this pH range about 20 gpl boric acid was added to the electrolyte to control hydrolysis at the cathode. The spent electrolyte from the diaphragm cell contained about 5 gpl less nickel than the feed solution resulting in a more acidic solution for the strip step. Although evaluation of the two cells has not been completed, the diaphragm cell has the advantage of greater efficiency, lower power consumption, and greater deposition rate. The more acidic recycle is advantageous also since it provides more efficient stripping, thus requiring less stripping stages. An obvious disadvantage of the diaphragm cell is upkeep of the diaphragms.

Results and Discussion

Results of this investigation have demonstrated that nickel and cobalt can be recovered selectively from low-grade domestic laterites. The reduction step is the most important phase of the process. Ideally, all of the metal of interest must be reduced but the reduction must be selective so that subsequent processing results in high net recovery. Satisfactory reduction is achieved in a relatively low temperature range (350° to 600° C) with carbon monoxide as the reducing gas and with the use of pyrite additions or post-reduction roasts as required by the magnesia content.

An advantage of the oxidizing ammonia leach over other leach processes is the high selectivity obtained in the leach. The leach solution contains only nickel, cobalt, and a minor amount of magnesium. The effect of reduction temperature and pyrite additions on nickel and cobalt extraction are illustrated in figure 4. At temperatures below 500° C and without pyrite additions, cobalt extraction was less than 20 percent. Although these results follow the same trend as obtained with chlorine (fig. 1), recovery is slightly less. The reason for the lower extraction (maximum of 92 percent for nickel and 87 percent for cobalt) under the same reduction conditions is that some nickel (2.5 to 5 percent) and cobalt (7 to 11 percent) are occluded by precipitating iron hydroxide and manganese oxide. The cobalt extraction is affected more than the nickel extraction since essentially all of the cobalt in this laterite is closely associated with manganese whose precipitation has not been controlled as yet. On the other hand, most of the nickel is associated with iron that can be selectively reduced and whose precipitation is delayed by formation of soluble ferrous ions in the early stages of the leach. This occlusion of nickel and cobalt during the leach emphasizes the importance of selective reduction.

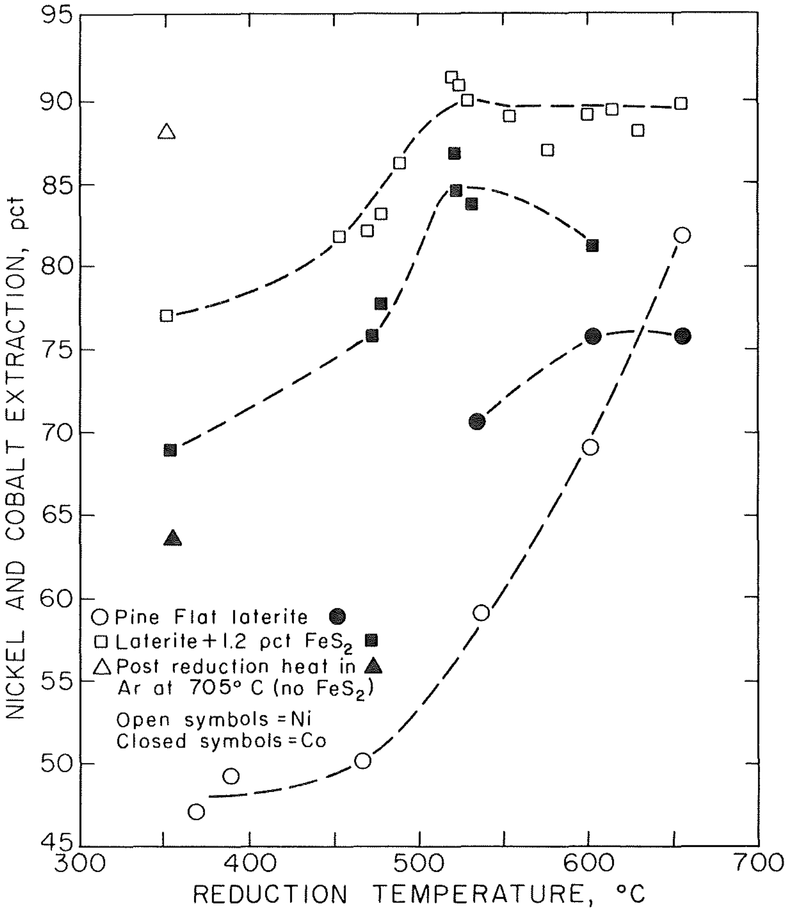

FIGURE 4. - Nickel and cobalt extraction from Pine Flat laterite with oxidizing ammonium hydroxide-ammonium sulfate leach.

CONTINUOUS PROCESSING

A continuous processing circuit is under development to provide engineering cost data for the entire process. Several sections are complete and are in preliminary operation stages. A flowsheet for the process is presented in figure 5. A continuous reduction system capable of handling 8 kg/hr passing countercurrently to a metered carbon monoxide flow is in the test stage. This system includes a feed hopper, rotary kiln for dehydration of the laterite, and a five-hearth, 20.32-cm-diameter rotary hearth furnace for the reduction. The reduced material is cooled to below 200° C in a water-cooled discharge screw. Preliminary operation of the unit has resulted in consistent extraction of 88 to 89 percent nickel and 77 percent cobalt under the following conditions: (1) A carbon monoxide-to-charge ratio of 90 liters/kg; (2) a roaster retention time of only 12 minutes (rabbling no faster than 1 rpm); and (3) with all the hearths at about 500° C. With only the feed rate changed (1.3 kg/hr), 91 percent nickel and 86 percent cobalt were extracted. The dehydration step prior to reduction is performed so that excess carbon monoxide and reaction-produced carbon dioxide can be recycled through a gas producer. To date, operation of a gas producer with carbon dioxide passed through a coke breeze bed containing 90 percent fixed carbon at 1,070° to 1,080° C resulted in a 99-percent conversion of carbon dioxide to carbon monoxide. The same producer could be used to make "typical" hydrogen-containing producer gas; however, the water produced from reduction with hydrogen would probably preclude recycle of the offgases. Furthermore, the extraction with such gases was generally found to be lower than with carbon monoxide as the reductant in the temperature range used in this process. A countercurrent leaching and filtering system is under construction to treat the reduced material. A countercurrent laboratory solvent extraction unit with 10 mixer-settler cells and capable of handling 250 ml/min total organic and aqueous flow has been installed and operated with batch leach solutions. This unit will provide the feed for the electrolytic cells. To date, up to

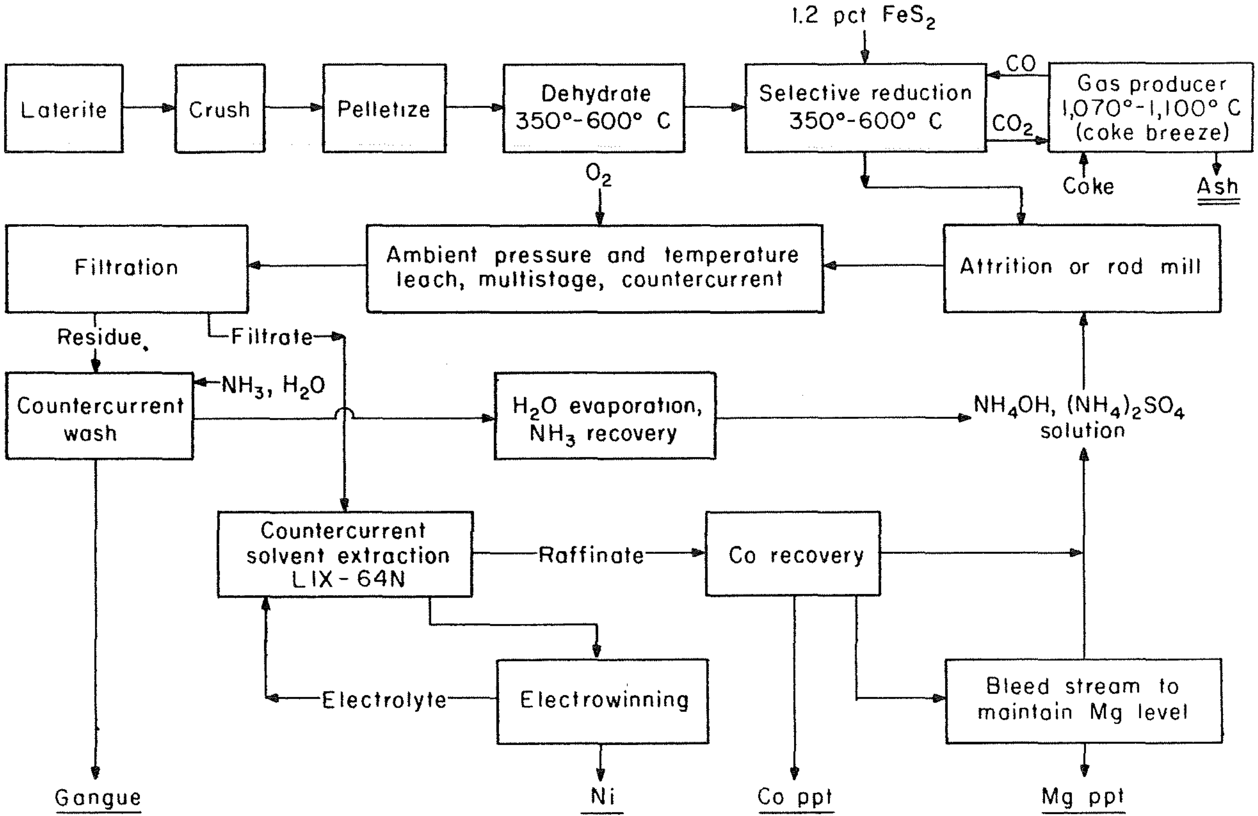

FIGURE 5. - Recovery of nickel and cobalt by an oxidizing ammonium hydroxide-ammonium sulfate leach.

98 percent of the contained nickel has been recovered from the leach liquor in a three-stage extraction with an organic-to-aqueous ratio of 1:1. The organic phase contained 10 percent LIX-64N and 90 percent Napoleum 470. The nickel-to-cobalt ratio has been increased from 5:1 in the leach solution to more than 700:1 in the loaded organic with less than 1 percent of the contained cobalt extracted. Work is now directed toward optimization of the aqueous-organic phase contact time to improve extraction and stripping of nickel. The organic phase is washed with dilute sulfuric acid prior to the nickel stripping step to remove entrained ammonia.

Entrained organic has been satisfactorily removed from the aqueous stream by employment of an inexpensive separation column. The column consists of a roll of polypropylene felt with a moisture barrier parallel to the surface. The contaminated aqueous phase is introduced into the center of the roll and must travel around a helical path from inside to outside of the roll (about 152.4 cm) past high-surface-area polypropylene fibers that collect the organic-phase droplets.

Although magnesium was not loaded on LIX-64N and did not adversely affect the leach in low concentrations, a scheme was demonstrated for its removal without adversely affecting the leach solution. A "bleed stream" could be treated as discussed in the batch testing to control the magnesium concentration.

In summary, advantages of the process are as follows: (1) The relatively low reduction temperature; (2) treatment of the leach solution by countercurrent solvent extraction and electrowinning so that the nickel and cobalt extracted in the leach are essentially totally recovered; and (3) the reagents are recycled. However, if the process were operated on a practical commercial scale, a minor ammonia loss (probably about 10 percent) could be expected.

PROCESS EXTENSION

An obvious extension of this process is to other ores that contain metals that will form soluble ammine complexes. Preliminary tests to date on dead-roasted chalcopyrite concentrate containing 36.3 percent copper resulted in 99 percent copper recovery for material reduced at 550° C and 98 percent copper recovery for material reduced at 350° C. One leach of a copper oxide ore containing 5.6 percent copper recovered 85 percent of the copper. About 88 and 74 percent of the contained copper (0.34 percent) and nickel (0.79 percent), respectively, were recovered from manganese sea nodules. Essentially all of the contained nickel (36.3 percent) and zinc (33.4 percent) were recovered from an oxidized, spent nickel-zinc catalyst material. These results are encouraging enough to warrant further study with a variety of materials, particularly since the process offers good recovery with relatively low energy and reagent consumption.

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