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DEPARTMENT OF ENERGY, MINES AND RESOURCES

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR-67-60

**INVESTIGATION OF A LOW-GRADE
NICKEL MINERALIZATION FROM
SUTHERLAND AND ASSOCIATES
BLIND RIVER, ONTARIO**

by

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MINERAL PROCESSING DIVISION

CENTRAL TECHNICAL FILES
C10/IR67/60
GEOLOGICAL SURVEY

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Investigation of Low-Grade Nickel Mineralization
From Sutherland and Associates, Blind River,
Ontario.

by

G.I. Mathieu*

SUMMARY OF RESULTS

In the sample received for investigation, only nickel (0.23%) was found in amounts of economic interest. Copper, zinc and cobalt were present in negligible quantities. No gold was detected and silver was found only in trace amounts.

Polydymite-violarite and pentlandite were the only nickel minerals observed in polished sections prepared from the head sample. The former was the most abundant. It occurred partly as free grains and partly as fine intergrowths with gangue and, to a lesser extent, with pyrite. The ultra-fine inclusions of nickel minerals in gangue accounted for 50% of the total nickel and averaged about 5 microns in size.

In several flotation tests on finely ground material under various conditions, nickel recovery never exceeded 51% in the rougher concentrates. When these concentrates were cleaned to a marketable grade (7 to 14% Ni), the overall nickel recovery was only about 30%.

Nickel concentration from flotation tailing by high-intensity magnetic separation was also tried, but without success.

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INTRODUCTION

Sutherland and Associates hold a mining property located in Montreal River Division, Temiskaming, Ontario. Considerable diamond drilling on this prospect had indicated a large tonnage of low-grade nickel mineralization. Prior to further development, the company wished to know if the material would be amenable to concentration by conventional mineral processing methods.

Shipment

On January 4, 1967, a shipment of split drill core weighing 512 lbs was received from Mr. W. Don Sutherland, Geological Engineer, P.O. Box 128, Blind River, Ontario. Mr. Sutherland said that the shipment was representative of 1080 ft of core samples which contained about 0.27% Ni with minor contents of copper, zinc, cobalt, gold and silver.

Sampling and Analysis

The drill-core sample was crushed to -1 in. from which a few representative pieces were selected for mineralogical examination. One half of the remainder was crushed to -10 mesh and a head sample was riffled out for chemical analysis.

TABLE 1

Chemical Analysis* of Head Sample

Nickel (Ni)	—	0.23 %
Copper (Cu)	—	0.02 "
Zinc (Zn)	—	0.02 "
Cobalt (Co)	—	0.01 "
Iron (Fe)	—	10.60 "
Sulphur (S)	—	0.22 "
Insoluble	—	47.46 "
Gold (Au)	—	none detected
Silver (Ag)	—	trace

* From Internal Report MS-AC- 67-168.

A spectrographic analysis on a portion of the head samples indicated the presence of the elements listed below in their approximate order of decreasing abundance.

TABLE 2

Spectrographic Analysis* of Head Sample

I	-	Si, Fe, Mg, Ca (>1.0%)
II	-	Al, Cr, Na, (1.0 - 0.5%)
III	-	Mn, Ni, Ti (0.5 - 0.1%)
IV	-	V, Sn, Cu (0.1 - 0.01%)
V	-	Co, In, Zn, Ag (<0.01%)

* From Internal Report MS-AC- 67-17.

Mineralogical Examination**

Several hand specimens and a portion of the -10 mesh head sample were sent to the Mineralogy Section of the Mineral Sciences Division for mineralogical examination.

The head sample was sized, and the -65+325 mesh fraction was separated into sub-fractions by means of heavy liquids having specific gravities of 2.96 and 3.33. Oil-immersion mounts were prepared from the 2.96 and 3.33 gravity float fractions, and polished sections were prepared from the 3.33 gravity sink fraction and from the pieces of drill core. The minerals were identified by microscopical and X-ray diffraction studies.

The sample consists of disseminated grains of ore minerals in a rock composed of feldspar, quartz, chlorite, a carbonate, and a trace of fluorite. The ore minerals are pyrite, chalcopyrite, sphalerite, violarite-polydymite, pentlandite, goethite, and hematite. Pyrite is the most abundant ore mineral. It occurs as irregular grains up to 1 mm in size, and contains minute inclusions and veinlets of chalcopyrite, violarite and galena.

The nickel-bearing minerals are violarite-polydymite and pentlandite. The former occurs as irregular grains up to 100 microns in size, and as inclusions and veinlets in gangue and pyrite. Although some of the inclusions are up to 50 microns in size, most of these are less than 10 microns.

The pentlandite occurs as minute inclusions in violarite. The chalcopyrite and sphalerite are present as small irregular grains, and as veinlets and inclusions in pyrite.

OUTLINE OF INVESTIGATION

Preliminary testwork consisted of rougher and cleaner flotation under various conditions, with the aim of finding the best practical technique for concentration of the nickel minerals.

** From Internal Report MS-AC-67-20 and Investigation Report IR-67-45

Then, in an attempt to produce a nickel concentrate of optimum grade and recovery, a test was done using the best conditions found previously.

Supplementary analyses, high-intensity magnetic separation, sizing tests and mineralogical examination were carried out on the flotation tailing to determine the cause of the poor recovery of nickel. When finding that most of the unfloated nickel was present as tiny inclusions in gangue (about 5 microns in size), flotation tests were made after stage grinding to -325 and -400 mesh.

DETAILS OF INVESTIGATION

Grinding Time, Tests 1-3

A 6000-g sample was cut from the -10 mesh material and divided into three equal fractions. After grinding these for 20, 30 and 40 min, separate rougher flotation was carried out on each fraction. The rougher concentrates were combined for cleaning.

All the products obtained were analysed for nickel. Using the weight of the products with these analyses, it was possible to compile with accuracy the nickel content of the original feed. This calculated head assay was used to calculate the nickel assays of the rougher concentrates. The same method for calculation was used in every subsequent series of tests.

Flotation procedure and results obtained are shown in Table 3 and 4. For an easier comparison, the results of rougher and cleaner flotation are tabulated separately.

TABLE 3

Reagents and Conditions of Flotation

Operation	Time min	Reagents	lb/ton	pH
Rougher flotation	12	Xanthate Z-6 Dowfroth 250	0.10 0.04	8.5
Scavenger flotation	6	Xanthate Z-6 Dowfroth 250	0.05 0.02	8.4
Cleaner flotation (twice)	7	Xanthate Z-6 Dowfroth 250	0.01 0.005	8.5
Recleaner flotation (twice)	5	Xanthate Z-6 Dowfroth 250	0.005 0.005	8.3

Note: The additions of reagents were made in stages.

TABLE 4

Flotation Results at Increasing Degree of Fineness

Test	Product	Weight %	Analysis* % Ni	Distribution % Ni
1 (62.7% -200m)	Ni rougher conc	7.6	1.06	36.6
	Ni scav conc	2.6	0.54	6.3
	Flot tailing	89.8	0.14	57.1
	Feed (calcd)	100.0	0.22	100.0
2 (83.7% -200m)	Ni rougher conc	7.1	1.21	39.2
	Ni scav conc	4.0	0.45	8.2
	Flot tailing	88.9	0.13	52.6
	Feed (calcd)	100.0	0.22	100.0
3 (94.0% -200m)	Ni rougher conc	6.1	1.11	30.8
	Ni scav conc	3.4	0.48	7.4
	Flot tailing	90.5	0.15	61.8
	Feed (calcd)	100.0	0.22	100.0
1-3 Cleaning	Ni recl conc	7.9	7.72	58.2
	Ni recl tailing	15.8	0.89	13.5
	Ni cl tailing	76.3	0.39	28.3
	Rougher conc (combined)	100.0	1.05	100.0

* By Sudbury Assay Office.

Reagents Concentration and Flotation Time, Tests 4-5

In these tests, made on 2000 g samples of ore ground to 87% -200m, the basic procedure of Table 3 was followed except that, in Test 4, reagent concentration was doubled and, in Test 5, both reagent concentration and flotation time were doubled.

As in previous tests, the rougher concentrates were combined for cleaner flotation and the results are given separately.

TABLE 5

Results with Higher Reagent Concentration and Longer Flotation Time

Test	Product	Weight %	Analysis % Ni	Distribution % Ni
4 (Higher reagent)	Ni rougher conc	8.2	1.28	43.6
	Ni scav conc	5.5	0.37	9.8
	Flot tailing	86.3	0.13	46.6
	Feed (calcd)	100.0	0.24	100.0
5 (Longer flotation)	Ni rougher conc	13.4	0.75	41.6
	Ni scav conc	11.9	0.32	15.8
	Flot tailing	74.7	0.14	42.6
	Feed (calcd)	100.0	0.24	100.0
4-5 (Cleaning)	Ni recl conc	7.6	7.89	59.8
	Ni recl tailing	9.8	0.99	9.7
	Ni cl tailing	82.6	0.37	30.5
	Feed (calcd)	100.0	1.00	100.0

Effect of pH, Tests 6 - 12

This series of tests was aimed at finding if nickel floatability could be increased by pH adjustment of the pulp. Neutral and acidic pH were controlled by use of sulphuric acid, while alkalinity was obtained by addition of either lime or soda ash. Chemicals used and corresponding pH values along with the results obtained are shown in Tables 6 and 7. The other conditions of flotation were similar to those used previously.

TABLE 6

Results of Flotation at Neutral and Acidic pH

Test	Product	Weight %	Analysis % Ni	Distribution % Ni
6 (H ₂ SO ₄ - 3.0 lb/ton pH -7.0)	Ni rougher conc	7.1	1.06	31.9
	Ni scav conc	3.6	0.50	7.6
	Flot tailing	89.3	0.16	60.5
	Feed (calcd)	100.0	0.24	100.0
7 (H ₂ SO ₄ - 6.0 lb/ton pH -6.6)	Ni rougher conc	7.3	1.24	37.5
	Ni scav conc	4.8	0.48	6.5
	Flot tailing	87.9	0.14	56.0
	Feed (calcd)	100.0	0.24	100.0
8 (H ₂ SO ₄ - 9.0 lb/ton pH -6.2)	Ni rougher conc	8.5	1.04	38.4
	Ni scav conc	3.4	0.45	9.7
	Flot tailing	88.1	0.15	51.9
	Feed (calcd)	100.0	0.24	100.0
6-8 (H ₂ SO ₄ - 1.0 lb/ton pH -6.5)	Ni recl conc	7.4	6.97	46.4
	Ni recl tailing	25.6	0.89	20.4
	Ni cl tailing	67.0	0.55	33.1
	Feed (calcd)	100.0	1.11	100.0

TABLE 7

Results of Flotation at High Alkalinity

Test	Product	Weight %	Analysis* % Ni	Distribution % Ni
9 (CaO-0.5 lb/ton pH - 9.0)	Ni rougher conc	5.9	1.26	32.3
	Ni scav conc	4.1	0.50	8.9
	Flot tailing	90.0	0.15	58.8
	Feed (calcd)	100.0	0.24	100.0
10 (CaO-1.5 lb/ton pH - 9.8)	Ni rougher conc	7.8	1.23	41.6
	Ni scav conc	2.9	0.33	4.2
	Flot tailing	89.3	0.14	54.2
	Feed (calcd)	100.0	0.24	100.0
9 - 10 (CaO-0.2 lb/ton pH - 9.5)	Ni recl conc	10.0	7.13	58.3
	Ni recl tailing	17.2	1.14	16.1
	Ni cl tailing	72.8	0.43	25.6
	Feed (calcd)	100.0	1.22	100.0
11 (Na ₂ CO ₃ -0.5 lb/ton pH - 8.9)	Ni rougher conc	8.3	1.04	37.6
	Ni scav conc	1.8	0.48	3.7
	Flot tailing	89.9	0.15	58.7
	Feed (calcd)	100.0	0.23	100.0
12 (Na ₂ CO ₃ -1.5 lb/ton pH - 9.6)	Ni rougher conc	7.5	1.22	39.8
	Ni scav conc	2.4	0.51	5.3
	Flot tailing	90.1	0.14	54.9
	Feed (calcd)	100.0	0.23	100.0
11 -12 (Na ₂ CO ₃ -0.2 lb/ton pH - 9.3)	Ni recl conc	8.0	8.15	59.3
	Ni recl tailing	15.7	0.86	12.3
	Ni cl tailing	76.3	0.41	28.4
	Feed (calcd)	100.0	1.00	100.0

* By Sudbury Assay Office.

Addition of Copper Sulphate, Tests 13 - 15

This series of tests was made on 2000 g samples ground for 30,60 and 90 minutes. The flotation procedure of Table 3 was followed, except that copper sulphate, in amounts of 1.0, 0.5 and 0.2 lb/ton of ore, was added to the rougher, scavenger and cleaner stages, respectively.

TABLE 8

Flotation Results with Copper Sulphate

Test	Product	Weight %	Analysis* % Ni	Distribution % Ni
13 (63.8%-200m)	Ni rougher conc	8.6	0.85	34.9
	Ni scav conc	2.8	0.45	6.0
	Flot tailing	88.6	0.14	59.1
	Feed (calcd)	100.0	0.21	100.0
14 (83.9%-200m)	Ni rougher conc	5.1	1.45	35.2
	Ni scav conc	3.7	0.47	8.3
	Flot tailing	91.2	0.13	56.5
	Feed (calcd)	100.0	0.21	100.0
15 (94.2%-200m)	Ni rougher conc	6.5	0.96	29.6
	Ni scav conc	4.2	0.54	10.8
	Flot tailing	89.3	0.14	59.6
	Feed (calcd)	100.0	0.21	100.0
13 - 15	Ni recl conc	6.3	10.81	67.9
	Ni recl tailing	14.1	0.48	6.7
	Ni cl tailing	79.6	0.32	25.4
	Feed (calcd)	100.0	1.00	100.0

* By Sudbury Assay Office.

A few tests were also made at higher concentration of copper sulphate, but no significant improvement of the results was achieved.

Use of Soda Ash with Copper Sulphate, Test 16

In this test, a 4000 g sample was ground to 83% - 200m and floated as before using 1.0, 0.5 and 0.2 lb per ton of both soda ash and copper sulphate in the rougher, scavenger and cleaner flotation.

TABLE 9

Effect of Soda Ash and Copper Sulphate

Product	Weight %	Analysis* % Ni	Distribution % Ni
Ni rougher conc	8.5	0.97	39.2
Ni scav conc	3.2	0.40	6.1
Flot tailing	88.3	0.13	54.7
Feed (calcd)	100.0	0.21	100.0
Ni recl conc	5.6	12.34	71.0
Ni scav conc	10.7	0.58	6.4
Flot tailing	83.7	0.26	22.6
Feed (calcd)	100.0	0.97	100.0

* By Sudbury Assay Office.

Flotation under Best Conditions, Test 17

A 6000 g sample of ore was ground to 84% - 200m and floated using the conditions shown in Table 10.

TABLE 10

Reagents and Conditions of Flotation

Operation	Time min	Reagents	lb/ton	pH
Rougher flotation	15	Soda ash	1.00	9.7
		Copper sulphate	1.00	
		Xanthate Z-6	0.15	
		Dowfroth 250	0.04	
Scavenger flotation	8	Soda ash	0.50	9.8
		Copper sulphate	0.50	
		Xanthate Z-6	0.08	
		Dowfroth 250	0.02	
Cleaner flotation (twice)	7	Soda ash	0.10	9.2
		Copper sulphate	0.10	
		Xanthate Z-6	0.01	
		Dowfroth 250	0.01	
Recleaner flotation (twice)	5	Copper sulphate	0.02	9.4
		Sodium silicate	0.05	

TABLE 11

Results of Flotation under Best Conditions

Product	Weight %	Analysis*			Distribution		
		%		oz/ton	%		
		Ni	S	Ag	Ni	S	Ag
Ni recl conc	0.4	14.53	21.30	3.07	26.6	36.6	84.1
Ni recl tailing	0.7	0.91	1.25	0.14	2.9	3.7	7.0
Ni cl tailing	6.6	0.26	0.33	0.02	8.0	9.4	8.9
Scav conc	4.7	0.45	1.00	Tr	9.8	20.2	—
Flot tailing	87.6	0.13	0.08	N.D.	52.7	30.1	—
Feed (calcd)	100.0	0.22	0.23	0.0.4	100.0	100.0	100.0

* Nickel determinations by Sudbury Assay Office.
Other analyses from Internal Report MS-AC- 67-345.

Because of the low recovery and because the sulphur content in the tailing appears insufficient to account for pentlandite (22% Ni, 36% S) and/or violarite-polydymite (55% Ni, 41% S), further studies were made to obtain more information about the nature of the nickel-bearing minerals.

A high-intensity magnetic separation test, using the Jones separator, was done on a sample of the flotation tailing. However, no concentration of either nickel or sulphur was obtained as shown by the following analyses:

Mag at 5 amp	—	0.16% Ni	and	0.06% S
Mag at 30 amp	—	0.16	" "	0.07 "
Non Mag (Jones tailing)	—	0.12	" "	0.09 "

A 500 g sample was then cut from the flotation tailing and sized using Tyler screens and an infrasizer apparatus. Each fraction was analysed for nickel and sulphur.

TABLE 12

Analyses of Sized Flotation Tailing

Size	Weight %	Analysis*		Distribution	
		Ni	S	Ni	S
+150 m	6.5	0.12	0.100	5.5	7.7
-150 + 200m	8.4	0.11	0.081	6.6	8.1
-200 m + 56 microns	9.7	0.11	0.074	7.6	8.6
-56 + 40 "	14.5	0.15	0.061	15.5	10.6
-40 + 28 "	15.5	0.13	0.057	14.3	10.5
-28 + 20 "	10.4	0.14	0.062	10.3	7.7
-20 + 14 "	6.8	0.12	0.068	5.8	5.5
-14 + 10 "	4.6	0.13	0.085	4.2	4.7
-10 "	23.6	0.18	0.130	30.2	36.6
Feed (calcd)	100.0	0.14	0.084	100.0	100.0

* From Internal Report MS-AC- 67- 574

The chemical imbalance between nickel and sulphur, particularly noticeable in the -56 to +14 microns fractions, prompted mineralogical examination of the flotation tailing to determine the nature of the nickel-bearing minerals. This study was conducted by Dr. W. Petruk of the Mineral Sciences Division and included microscopic examination as well as microprobe analyses. The details of the mineralogical work are given in Mines Branch Investigation Report IR-67-45 and only its conclusion will be quoted here.

"The nickel-bearing minerals identified in the tailing are violarite-polydymite and pentlandite. They are present as minute inclusions in gangue (about 5 microns in size) and attribute, at least in part, to the high nickel content of the tailing. The low sulphur:nickel ratio in the tailing indicates that some nickel is also present in a non-sulphide form. However, experiments aimed at determining whether the magnetite, carbonate minerals, and other gangue constituents contain significant amounts of nickel gave negative results. It is therefore inferred that the non-sulphide nickel is present as a trace element in gangue minerals (chlorite) in concentration too low to be detected by the probe microanalyser.

Because of the difficulty of liberating minute sulphide grains and the possibility of some nickel being present in the non-sulphide form, it is unlikely that high nickel recoveries can be achieved by conventional ore-dressing procedures."

Flotation of Ultra-Fine Ore, Tests 18 - 19

At the request of Mr. Sutherland, supplementary flotation

tests were made after stage grinding to -325 and -400 mesh in an attempt to liberate and recover additional nickel sulphides. The procedure shown in Table 10 was used and the results obtained are given in Table 13.

TABLE 13
Flotation Results After Grinding to -325 and -400 Mesh

Test	Product	Weight %	Analysis* % Ni	Distribution % Ni
18 (-325m)	Ni rougher conc	12.4	0.95	51.1
	Ni scav conc	5.0	0.27	5.9
	Flot tailing	82.6	0.12	43.0
	Feed (calcd)	100.0	0.23	100.0
19 (-400m)	Ni rougher conc	11.5	0.91	45.5
	Ni scav conc	5.2	0.33	7.4
	Flot tailing	83.3	0.13	47.1
	Feed (calcd)	100.0	0.23	100.0
18 - 19 (cleaning)	Ni recl conc	3.7	8.92	35.3
	Ni recl tailing	14.0	0.79	11.8
	Ni cl tailing	82.3	0.60	52.9
	Feed (calcd)	100.0	0.93	100.0

* From Internal Report MS-AC- 67-545.

Note: The sulphur content of the flotation tailing was 0.072% for Test 18 and 0.075 for Test 19.

SUMMARY AND CONCLUSION

Head analyses of the ore sample were as follows:

<u>Ni (%)</u>	<u>Cu (%)</u>	<u>Zn (%)</u>	<u>Co (%)</u>	<u>Ag</u>	<u>Au</u>
0.23	0.02	0.02	0.01	Tr	N.D.

Therefore, the only metal of economic interest was nickel.

Violarite-polydymite and pentlandite were positively identified in the ore, both as free grains and as inclusions in pyrite and gangue minerals. Studies of the nickel-sulphur distribution were indicative that some of the nickel might occur in a non-sulphide form.

In several series of flotation tests, the following points were established:

1. Grinding to about 84% -200 mesh was sufficient to float most of the recoverable nickel.
2. Higher reagent concentration and longer flotation time slightly increased the nickel recovery.
3. The pH of the pulp, varied over a range of 6.0 to 10.0, by sulphuric acid, lime and soda ash additions had no significant effect on either the grade or the recovery of nickel.
4. Addition of copper sulphate, with and without pH control by soda ash, improved the grade of the final nickel concentrate.

A typical flotation test gave a nickel recleaner concentrate assaying 14.5% Ni and containing 26.6% of the nickel. In an attempt to account for the low nickel recovery, further mineralogical and chemical studies were made on the flotation tailing. These led to the conclusion that some of the residual nickel (sulphide) was too fine for practical liberation and the remainder probably in non-sulphide form (i.e. chlorite and septachlorite) unrecoverable by conventional ore-dressing methods.

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