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**BENEFICIATION STUDIES OF NICKELIFEROUS ORES
FROM THE SHAMROCK MINE, JACKSON COUNTY, OREG.,
AND THE CONGRESS MINE, FERRY COUNTY, WASH.**

BY J. E. SHELTON

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SUMMARY

Flotation tests are described on samples of two lowgrade nickeliferous ores of Oregon and Washington. The sample from the Congress mine, Ferry County, Wash., contained 1.2 percent Ni, occurring chiefly as millerite. The sample from the Shamrock mine, Jackson County, Oreg., contained 2.20 percent Ni and 0.78 percent Cu occurring chiefly as pentlandite, nickeliferous pyrrhotite, and chalcopyrite.

The millerite in the Congress ore was concentrated by flotation with sulfydric collectors in a series of locked tests to recover 73 percent of the nickel as a 7-percent bulk concentrate. By successively regrinding and floating the bulk concentrate, it was further concentrated to 12.4 percent Ni at a recovery of 59 percent and 21.8 percent Ni at a recovery of 50 percent.

Attempts at selective concentration of the copper-nickel Shamrock ore were only partly successful because of the intimate association of pentlandite and chalcopyrite. The nickel concentrate, which contained 86 percent of the nickel, had an analysis of 4.42 percent Ni and 1.03 percent Cu. The copper concentrate had an analysis of 18.8 percent Cu and 1.96 percent Ni and contained 48.5 percent of the copper. A bulk sulfide concentrate contained 92 percent of the copper and 91 percent of the nickel and had an analysis of 4.17 percent Ni and 1.63 percent Cu.

INTRODUCTION

This work was started to investigate the application of ore-dressing techniques to nickel sulfide ores from the larger low-grade deposits of the Northwest. Some of these deposits contain as much as 1.0 percent nickel and, in some places, appreciable amounts of copper.

The primary objective of the test work was to produce a bulk sulfide concentrate that would be suitable as furnace feed to produce nickel alloys or ferro-nickel. A suitable grade appeared to be approximately 5.0 percent nickel. For those ores containing some copper, studies would be made to concentrate selectively the nickel and copper into separate products.

Two deposits were chosen as containing the desired minerals and ore of sufficient quantity to warrant study. One sample was obtained from the Congress mine in Ferry County, Wash., and the second from the Shamrock mine in Jackson County, Oreg.

CONGRESS MINE

Nature of the Ore

The Congress mine on Bridge Creek was discovered in the early 1880's. The present adit level was developed in 1904. There has been no production from the deposit.

The general geology of this immediate area has been described by Bancroft.^{2/} The schistose rocks are of both sedimentary and igneous origin, have been intruded by a large dike of porphyry, and are believed to be of Paleozoic age. The schistose rocks have a general northeast strike, and dip at angles ranging from 45° northwest to vertical. They are composed of apparently conformable strata of amphibolitic schists with interlaminated lime shale, mica schist, and rock that resembles sheared granite. Beds of limestone and quartzite also are included in this series.

Excerpts from a preliminary report by field engineers on the Congress mine state that the deposit appears to occur principally in the schist formation and the general strike is N. 60° E. The dip is 50° to 85° NW. The exact width and length of the deposit can not be determined from the work that has been done, but the adit level development and the mineralized float observed in the overburden indicate the deposit to be 6 to 80 feet wide and at least 1,200 feet long.

The nickel mineralization occurs as fracture fillings and disseminations in a gangue of bluish colored quartz. A strong gouge appears to form the foot wall of the deposit. Considerable faulting within the deposit appears evident.

Inferred ore reserves are estimated at 625,000 tons on the basis of an inferred width of 30 feet and depth equal to the length of the section or 500 feet. Because the ore is not blocked out in the underground workings, insufficient factual data is available for basis of an estimated ore reserve. The ore that may be ultimately developed is not limited to the above inferred tonnage because this ore section is open to possible extension.

Representative portions of the head sample were prepared and submitted for chemical and spectrographic analyses, with the results as shown in table 1.

TABLE 1. - Analyses of Congress-mine sample

Chemical analysis, percent					
Cu	Ni	Fe	S	Co	Cr
0.02	1.2	5.5	5.03	0.02	0.27
Spectrographic analysis					
Constituent			Approximate percent		
Si			Over 10		
Mg, Fe			5 to 10		
Ca, Ni			1 to 5		
Al, Co, Cr, Mn			0.1 to 1		
Ti			0.01 to 0.1		
Cu			0.001 to 0.01		

A representative sample of the ore was examined in the petrographic laboratory and was found to contain essentially quartz, with some associated dolomite and pyrite, small amounts of barite, serpentine, chromite, millerite, and nickeliferous chlorite, and very small amounts of calcite, limonite, and magnetite.

The nickeliferous chlorite has been tentatively identified by its optical properties as nepouite. An X-ray analysis of high-purity concentrate of this mineral did not yield any evidence that could be used in confirming the optical identification. Some of the millerite and nickeliferous chlorite is liberated in the

^{2/} Bancroft, Howland, Geology and Mineral Deposits of the Colville Indian Reservation: Geol. Survey Bull. 677, 1918, 186 pp.

minus-100-plus-200-mesh fraction. However, because of the intimate association of pyrite, millerite, quartz, and nickeliferous chlorite, liberation of the nickel minerals is not complete even in the minus-200-mesh fraction.

A screen analysis of minus-10-mesh roll-crushed material showed no significant concentration of the nickel or iron in any size fraction. The results of this screen analysis are shown in table 2.

TABLE 2. - Screen analysis of minus-10-mesh roll-crushed ore from the Congress mine

Product	Weight, percent	Analyses, percent		Distribution, percent	
		Ni	Fe	Ni	Fe
Plus-20-mesh	25.21	1.09	5.73	23.06	25.67
20/28-mesh	7.63	1.21	6.96	7.75	9.44
28/35-mesh	9.16	1.41	7.34	10.84	11.95
35/48-mesh	6.41	1.69	9.24	9.09	9.39
48/65-mesh	5.31	1.91	8.30	8.51	7.83
65/100-mesh	5.12	1.90	8.30	8.16	7.55
100/200-mesh	12.48	1.32	5.44	13.82	12.07
Minus-200-mesh	28.68	.78	3.16	18.77	16.10
Calculated head	100.00	1.19	5.63	100.00	100.00

Experimental Procedure and Results

Selective flotation tests were made in an attempt to float the millerite from the pyrite on samples of minus-10-mesh roll-crushed ore stage ground in the laboratory ball mill to 95 percent minus-200-mesh.

Anionic sulfonate-type promoters were successful in making a nickel concentrate with a grade of 4.08 percent nickel and 6.74 percent iron containing 74.4 percent of the nickel. Attempts to increase the grade of concentrate by cleaning lowered the recovery to less than 50 percent.

Selective flotation tests using sulfhydryc-type collectors in an alkaline circuit demonstrated that secondary butyl xanthate was selective in floating the millerite. Although pyrite was depressed, so was the unliberated millerite, therefore, to prevent the necessity of having to fine-grind the complete sample, bulk sulfide flotation was made in an acid circuit, and the subgrade concentrate was reground for selective flotation in an alkaline circuit. Bulk sulfide flotation tests made to determine optimum conditions showed that enough H_2SO_4 to maintain a pH of 6.0 was effective in depressing the siliceous gangue and suitable for activation of the millerite and pyrite. Cresylic acid produced a froth persistent enough to carry the heavy sulfides without excess gangue minerals. The bulk concentrates were reground to 88-98 percent minus-325-mesh, repulped, and the pH adjusted to 8.2 to 8.5 with NaOH, Na_2CO_3 , or CaO. Finer grinding of the cleaner concentrate was slightly beneficial in increasing both recovery and grade of nickel concentrate. Raising the pH to 7.5 or higher with NaOH or Na_2CO_3 was effective in depressing the pyrite with little or no effect on the free millerite. Excess CaO tended to depress the millerite. However, it was found that the addition of 0.1 to 0.3 pounds per ton NaCN to the flotation circuit aided in depressing the pyrite with no effect on millerite.

Results of the best test, in which the bulk sulfide concentrate was reground to 88 percent minus-325-mesh and Na_2CO_3 was used to maintain a pH of 8.2, are shown in table 3. Selective flotation tests were conducted using the reagent scheme

previously described on a bulk sulfide concentrate ground to 98 percent minus-325-mesh. Results of one of the tests are shown in table 4.

TABLE 3. - Metallurgical data for selective flotation of a bulk concentrate ground to 88 percent minus-325-mesh

Product	Weight-percent	Analyses, percent		Distribution, percent	
		Ni	Fe	Ni	Fe
Nickel concentrate	5.35	12.4	24.9	59.1	23.0
Pyrite concentrate	7.41	2.31	35.4	15.3	45.3
Rougher tailing	87.24	.33	2.10	25.6	31.7
Calculated head	100.00	1.12	5.79	100.0	100.0

TABLE 4. - Metallurgical data for selective flotation of a bulk concentrate ground to 98 percent minus-325-mesh

Product	Weight-percent	Analyses, percent		Distribution, percent	
		Ni	Fe	Ni	Fe
Nickel concentrate	2.40	21.8	25.8	50.0	10.1
Pyrite concentrate	7.70	2.20	41.3	16.2	52.1
Cleaner tailing	2.23	2.07	9.6	4.4	3.5
Rougher tailing	87.67	.35	2.4	29.4	34.3
Calculated head	100.00	1.05	6.13	100.0	100.0

Bulk sulfide flotation tests have shown that approximately 75 percent of the nickel could be recovered at a grade of 6.5 percent nickel. A locked series test was conducted to simulate continuous operation. The first sample was ground at 50 percent solids to 96 percent minus-200-mesh with 1.0 pound of Xanthate 301 per ton of ore. The ground pulp was conditioned with 0.3 pound of cresylic acid per ton of ore and H_2SO_4 to maintain a pH of 5.0. The concentrate was cleaned once at a pH of 5.9. The cleaner tailing was added to the ball mill and reground with the next sample. This was continued until 5 samples were processed with the final cleaner tailing kept separate. The concentrates and rougher tailings were combined for analyses.

A sample of the rougher tailing was sized on 100-, 200-, and 325-mesh screens with results as shown in table 5. The metallurgical data for this test and the reagent consumption are shown in table 6.

TABLE 5. - Screen analysis of rougher tailing from locked series test

Product	Weight, percent
Plus-100-mesh	0.20
100/200-mesh	3.49
200/325-mesh	23.44
Minus-325-mesh	72.87
	100.00

TABLE 6. - Locked series test on Congress-mine ore

Point of addition	Reagent scheme					
	Conditions		pH	Reagent consumption, pounds per ton		
	Time, min.	Solids, percent		X-301	H ₂ SO ₄	Cresylic acid
Grind	30	50		1.0		
Condition	5	35	6.0		3.0	0.03
Rougher float ...	5	24				
Cleaner float ...	3	12	5.8		.75	
Metallurgical data						
Product	Weight-percent	Analyses, percent		Distribution, percent		
		Ni	Fe	Ni	Fe	
Concentrate	10.71	7.25	42.6	73.26	71.58	
Cleaner tail68	1.29	5.8	.83	.62	
Rougher tail	88.61	.31	2.0	25.91	27.80	
Calculated head .	100.0	1.06	6.38	100.00	100.00	

Bulk sulfide flotations using fatty or resin acid were made in an attempt to improve recovery of the nickel without sacrificing grade. It was found that 86.56 percent of the nickel could be recovered in a concentrate that had an analysis of 3.02 percent nickel by using a 25-percent emulsion of 6 parts Liqro, 6 parts fuel oil, and 1 part Oronite L in an acid circuit utilizing two cleaning steps. Other types of fatty acid collectors, in either acid or alkaline circuits, failed to produce concentrates of equal grade.

Tailings from flotation tests using sulfhydic collectors contained approximately 25 percent of the nickel, which petrographic examination showed to be present as nickeliferous chlorite. Additional tests were conducted in an attempt to recover this nickel. Gravity concentration was not successful because the difference in specific gravity between the chlorite and gangue minerals was not great enough to permit separation by this means. Flotation using fatty acid or sulfonate-type collectors did not concentrate the nickel in either the concentrate or the tailing products.

Gravity tests showed that the chromite present in the rougher tailing could be concentrated to a grade of 14.2 percent Cr at a recovery of 18 percent. The table concentrate also contained 14.1 percent iron and 0.39 percent nickel with a recovery of 0.10 percent.

Discussion of Results

Flotation tests were conducted on a sample from the Congress mine with two objectives in mind: (1) To produce a nickel-iron concentrate and (2) to produce a high-grade nickel concentrate.

The results of locked series tests indicated that 73.3 percent of the nickel and 71.6 percent of the iron could be recovered in a bulk concentrate that contained 7.25 percent nickel and 42.6 percent iron.

It was found that regrinding the bulk concentrate followed by selective flotation of the millerite produced a concentrate that had a grade of 12.4 percent nickel and contained 59.1 percent of the total nickel.

Additional fine grinding of a bulk concentrate showed that 50 percent of the nickel could be recovered at a grade of 21.8 percent.

Attempts to further recover the nickel from the tailing were not successful because the nickeliferous chlorite did not respond to concentration by gravity methods.

SHAMROCK MINE

Nature of the Ore

As reported by Hundhausen,^{3/} the Shamrock copper-nickel mine was developed intermittently from 1900 to 1929 by the late Dr. W. P. Chisholm of Gold Hill, Oreg. During this period four adits were driven and a 60-foot inclined shaft was sunk. Oxidized copper-nickel showings in the shaft in an intermediate adit were not encouraging; the nickel content of the ore was not considered important. Subsequently, the shaft filled with water. Later, Chisholm's sons started the main adit to intersect the shaft and drain it, but this work was abandoned when hard rock was encountered 40 feet from the shaft bottom.

In 1945 R. D. Simon of Medford, Oreg., and his partner, Stanley Hyde of Hood River, Oreg., purchased the Shamrock claim from the Chisholm estate. They completed the main adit to the shaft in 1948. Nickel sulfide ore found at this elevation, between two flat faults, was drifted on for an additional 40 feet before work was suspended owing to lack of funds. The Bureau of Mines continued development in 1949 and 1950. The property has had no production.

Representative samples of this ore were prepared and submitted for chemical and spectrographic analyses, as shown in table 7.

TABLE 7. - Analyses of Shamrock mine sample

Chemical analysis, percent				
Cu	Ni	Fe	S	Co
0.78	2.20	28.1	16.1	0.01
Spectrographic analysis				
Constituent		Approximate percent		
Fe, Mg, Si		Over 10		
Al, Cu, C, Ni		1 to 5		
Co, Cr, Mn, Th		0.1 to 1		
Ti, V, Zn, Zr		0.01 to 0.1		
Hg		0.001 to 0.01		

Petrographic examination of a sample of the ore showed that it contained pyrrhotite and clinopyroxene, some biotite, amphibole, quartz, and feldspar, small amounts of chlorite, serpentine, and chalcopyrite, and very small amounts of pentlandite, zoisite, and graphite.

Detailed polished surface studies revealed that at least some of the chalcopyrite and pentlandite is intimately associated in minute microscopic veinlets and that complete liberation of these two minerals from each other would not take place even as a minus-400-mesh material. In this particular study it was not

^{3/} Hundhausen, R. J., Investigation of Shamrock Copper-Nickel Mine, Jackson County Oreg., Bureau of Mines Report of Investigations 4895, 1952, 12 pp.

definitely determined how fine the material would have to be ground to completely liberate the pentlandite from the chalcopyrite. However, examination of the sized products indicates that the bulk of the chalcopyrite and chalcopyrite-pentlandite admixture is liberated in the minus-100-plus-200-mesh fraction. The pyrrhotite also is liberated at this size.

8. A screen analysis of minus-10-mesh roll-crushed material is shown in table 8.

TABLE 8. - Screen analysis of roll-crushed material

Product	Weight-percent	Analysis, percent			Distribution, percent		
		Cu	Ni	Fe	Cu	Ni	Fe
Plus-20-mesh	28.04	0.69	2.08	27.1	23.40	26.76	26.68
20/28-mesh	15.04	.73	1.87	28.5	13.28	12.91	15.04
28/35-mesh	12.38	.73	1.82	29.2	10.93	10.34	12.69
35/48-mesh	10.37	.75	1.86	29.3	9.41	8.85	10.67
48/65-mesh	8.47	.70	1.99	29.8	7.17	7.73	8.86
65/100-mesh	6.79	.85	2.22	29.3	6.98	6.92	7.10
100/200-mesh	8.03	.80	2.76	29.2	7.77	10.17	8.23
Minus-200-mesh	10.88	1.60	3.27	28.1	21.06	16.32	10.73
Calculated head	100.00	.83	2.18	28.5	100.00	100.00	100.00

Experimental Procedure and Results

Previous metallurgical testwork on samples of ore from the Shamrock mine^{4/} showed that approximately 90 percent each of the copper and nickel can be recovered by flotation in a low-grade bulk sulfide concentrate.

Petrographic examination showed that liberation of the sulfide minerals from the gangue was not complete even in the minus-100-, plus-200-mesh fraction and the sulfides were intimately associated in the minus-400-mesh range.

Samples ground to 90 percent minus-200-mesh were treated by selective flotation of first the copper and then the nickel. The copper rougher concentrate was cleaned three times and the three cleaner tailings were combined with the rougher tailing for subsequent nickel flotation. Typical reagent consumption and metallurgical data are shown in table 9.

Bulk sulfide flotation tests were made using a combination of Minerec 27 and Xanthate 301. Sodium silicate, lime, or sodium carbonate was used to adjust the pH to 8.5 to 9.5. The rougher concentrates were cleaned once with no further reagent additions. The results of the best test, using CaO at a pH of 8.5, are shown in table 10.

^{4/} Hundhausen, R. J., Investigation of Shamrock Copper-Nickel Mine, Jackson County, Oreg. Bureau of Mines Rept. of Investigations 4895, 1952, 12 pp.

TABLE 9. - Selective flotation of Shamrock-mine ore

Reagent consumption							
Circuit	pH	Reagents, pounds per ton.					
		CaO	Na ₂ CO ₃	Minerex 27	Frother B-23	Xanthate 301	Na ₂ SiO ₃
Cu rougher	9.3	8	2	0.03	0.03		
Cu cleaner 1	9.0						
Cu cleaner 2	8.7						
Cu cleaner 3	8.5						
Ni rougher	8.5				.03	0.4	1.0
Ni cleaner 1	8.0						1.0
Ni cleaner 2	8.0						

Metallurgical data					
Product	Weight percent	Analyses, percent		Distribution, percent	
		Cu	Ni	Cu	Ni
Cu concentrate	2.41	18.8	1.96	48.5	2.5
Ni concentrate	37.63	1.03	4.42	41.5	86.2
Ni cleaner tail 2 ...	9.16	.55	1.31	2.7	3.1
Ni cleaner tail 1 ...	4.64	.19	.62	1.9	2.9
Ni rougher tail	46.16	.11	.22	5.4	5.3
Calculated head	100.00	.93	1.93	100.0	100.0

TABLE 10. - Bulk flotation of Shamrock-mine ore

Metallurgical data					
Product	Weight- percent	Analyses, percent		Distribution, percent	
		Cu	Ni	Cu	Ni
Concentrate	46.16	1.63	4.17	91.7	91.0
Cleaner tail	7.57	.29	1.06	2.7	5.2
Rougher tail	46.27	.10	.24	5.6	3.8
Calculated head	100.00	.82	2.11	100.0	100.0

Flotation tests conducted on samples of copper sulfide and nickel sulfide-bearing material from the Shamrock mine, Jackson County, Oreg., to produce separate copper and nickel concentrates by selective flotation indicated that as much as 48.5 percent of the copper could be recovered in a concentrate containing 18.8 percent copper and 1.96 percent nickel. A nickel concentrate could be made that recovered 86.2 percent of the nickel and 41.5 percent of the copper at a grade of 4.4 percent nickel and 1.03 percent copper.

Results of bulk sulfide flotation showed that 91.7 percent of the copper and 91.0 percent of the nickel could be recovered at grades of 1.63 percent copper and 4.2 percent nickel.

Much of the flotation work on this project was done by trainees Pedro Portaro Urdanivia and Carlos Angulo Saavedra from Peru and Nadda Bunyasiri from Thailand.

CONCLUSIONS

Selective flotation of millerite from pyrite, as found in the Congress mine ore, or pentlandite from chalcopyrite and pyrrhotite, as found in the Shamrock-mine ore, will produce nickel concentrates containing as much as 21.8 percent nickel, but it would be difficult to recover more than 50 percent of the nickel.

It is therefore concluded that bulk sulfide flotation to reduce the weight of product, before hydrometallurgical or pyrometallurgical treatment, offers the best method for utilizing the material.