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CONTINUOUS FLOTATION TESTS ON A NIOBIUM ORE FROM THE COULEE LEAD AND ZINC MINES LIMITED PROPERTY AT OKA, QUEBEC

by

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MINERAL DRESSING AND PROCESS METALLURGY DIVISION

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SUMMARY OF RESULTS

Continuous pilot plant flotation tests were carried out on a niobium ore from the Coulee Lead and Zinc Mines at Oka, Quebec. From approximately 120 tons of ore containing 0.28 to 0.32% Nb_2O_5 as pyrochlore, concentrates assaying from 3 to 20% Nb_2O_5 were produced. The best recovery obtained was about 90% at a grade of approximately 6% Nb_2O_5. The flotation reagent used as a pyrochlore collector was a proprietary mixture of a long chain amine and a diamine with a dispersing agent. The production of pyrochlore concentrates containing up to 10% Nb_2O_5 appears to be practical. More research is required to improve the process for upgrading the concentrates to 20% with reasonable recovery, and to reduce reagent costs.

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INTRODUCTION

Property

The property of the Coulee Lead and Zinc Mines Limited is located on the Ottawa River near Oka, Quebec. A joint owner of the property is Headway Red Lake Gold Mines Limited. Development of the process for recovery of the ore and treatment is being carried out by Columbium Mining Products, a holding company owned jointly by the Headway and Coulee companies.

The ore is a calcite-silicate complex containing niobium mainly as the mineral pyrochlore with minor amounts of niobium-bearing perovskite. Very little niocalite, a silicate containing niobium, has been found on this property. The gangue minerals are mainly calcite, diopside, apatite and mica, with minor amounts of magnetite and pyrite.

Personnel

The test work was under the supervision of Mr. H. L. Noblitt, Box 297, R.R. 5, Ottawa, Ontario, consulting metallurgist for Columbium Mining Products. He was assisted by Mr. R. J. Brisson and Mr. Dan Jacobs of Battelle Memorial Institute, Columbus, Ohio. Operating personnel and general supervision were provided by the Mines Branch, Department of Mines and Technical Surveys.

Object of Tests

Considerable test work had been done by Mr. Noblitt and Battelle Memorial Institute to develop a flotation process for the Coulee-Headway Oka ores. Laboratory work using a process patented by Mr. H. G. Burks of the Department of Geological Survey, Tanganyika, was very successful. However, pilot plant continuous work was required to investigate the operating variables of the flotation process, particularly to determine the effect of recycling cleaner tailings, since laboratory work had indicated that several cleaning stages would be necessary. Also, Battelle Memorial Institute required large samples of the concentrate to continue development of an extraction process.

Schedule of Tests

Preliminary tests were started on July 8 at Ottawa. After a grinding circuit had been arranged, flotation tests of about 5 hr duration were started. After Mr. Brisson and Mr. Jacobs arrived, 5 day continuous tests were started. About October 1, these tests were completed. Retreatment of the concentrates on both laboratory and pilot plant scale continued until November 28.

Ore Shipments

Two shipments of ore were received before the tests were started totalling approximately 55 tons and averaging 0.324% Nb_2O_5. During the tests, a third shipment of 66.5 tons was received which had an average grade of approximately 0.282%.

TEST WORK

(A detailed account of the test procedure with results is given in the report by Mr. H. L. Noblitt which is attached to the original of this report).

Crushing

When received the ore was crushed in the sampling plant 8 in. by 12 in. jaw crusher, followed by a 20 in. Symons cone and a Cie de Entreprises Industrielles gyratory crusher in closed circuit with a $\frac{1}{4}$ in. Tyrock screen. Preliminary tests on $-\frac{1}{2}$ in. ore proved that finer crushing was necessary to eliminate size segregation and resulting erratic performance of the grinding circuit feeder.

Grinding

The $-\frac{1}{4}$ in. ore was fed to the pilot plant rod mill which discharged to a 14 in. by 8 ft Duplex (DSFP) classifier. The overflow from the classifier, at approximately 30% solids, was the flotation plant feed. The classifier sands were ground in a small Hardinge mill (22 in. by 6 in.) and returned to the classifier. It was expected that this method would avoid overgrinding. However, both mills were high discharge types so some overgrinding resulted. The feed rate at which the circuit balanced was 460 lb/hr.

Flotation

The flotation test procedure was arranged not only to check the reagent combination for the process but to investigate as many operating variables as possible. Therefore, a number of flowsheets were employed. The standard flowsheet is shown in Figure 1, attached as an appendix to this report. The number of cells used varied in different stages of the test. In the final flowsheet pumps were used for all interstage pulp transfer to assist in froth control and to stabilize pulp levels.

The procedure normally employed a rougher circuit in which a rough pyrochlore concentrate was floated. The tailing, mainly calcite, went to waste. The pyrochlore concentrate was then cleaned in several stages with cleaner tailings recycled. At one time nine cleaning stages were employed. Only two products were made in this part of the procedure:- a cleaned pyrochlore concentrate and a tailing.

Several schemes were employed to clean up this pyrochlore concentrate, including reflotation with modifying agents such as sodium silicate, hot caustic treatment followed by washing and reflotation, magnetic separation, and gravity concentration. The magnetite was eliminated from the concentrate by magnetic separation. Sulphides and mica could be floated with cationic reagents such as Armac 12D but some pyrochlore was also lost. The best concentrates were made by tabling. Screening removed the large flakes of mica from the finished concentrate. It appears that any method of treatment should follow immediately after the initial flotation, particularly the flotation retreatment steps.

The reagent used in the primary flotation for collection of pyrochlore was a cationic amine mixture similar to the reagent combination patented by Mr. Herbert George Burks in the Union of South Africa. Mr. Burks' patented mixture is 0.6 lb Amine 220, 0.08 lb Duomeen T, and 0.3 lb Ultrawet 404 per ton of ore. Since Mr. Noblitt has found different proportions to be more effective and since the patent position is not clear in Canada the reagent mixture used in this test is referred to as Reagent A. In addition to Reagent A as collector, a bicarbonate was used as a modifying agent. This part of the process also is subject to patent claims. Potassium bicarbonate gave best results but due to its cost ammonium and sodium bicarbonates were substituted. Recovery with ammonium bicarbonate was good but the concentrate grade was slightly lower than with potassium bicarbonate. The carbonate was added with the feed water to mills and cells.

The Reagent A mixture tended to overfroth so air control on the cells was very necessary. This was never satisfactory as the improvised system of clamps and corks on the intercell passages lacked flexibility and the restricted passages were blocked by pieces of oversize feed, scale, cloth, wood, etc. Use of pumps to transfer concentrate from stage to stage helped to break up the froth.

The flotation pilot plant was operated on a three-shift continuous basis for a week at a time with shutdown on weekends. These operating periods were of sufficient length to establish equilibrium and to investigate the effect of return loads. No attempt was made to recirculate the water from the tailings but this could be done in a full scale plant. Reagent consumption was therefore somewhat higher than would be expected in final operation. Sulphuric acid was used in the first tests but its use was discontinued later.

During the investigation the laboratory flotation facilities were used frequently to check the metallurgy of the process. A large number of laboratory flotation and tabling tests were made during the investigation of retreatment processes for the primary concentrate.

Product analysis for Nb_2O_5 was done by the Mines Branch analytical laboratories using the calorimetric method described by G. H. Faye in Chemistry in Canada, Vol. 10, No. 4, p. 90, April 1958.

RESULTS

Details of results obtained are given in Mr. Noblitt's report to Coulee Lead and Zinc Mines Limited.

The best recoveries were obtained in the week September 22 to September 26, when from an average feed of 0.29% Nb_2O_5, 90% of the niobium was recovered in a concentrate averaging 3.73% Nb_2O_5, with an average tailing of 0.031% Nb_2O_5. This tailing composite was screened and infrasized and the fractions were assayed for Nb_2O_5. The results of this test indicated that although losses in the +100 mesh were high, the tailing losses in the -28μ material were much higher. The 13.4% of tailing which was -28μ represented 43.4% of the niobium loss. It would appear uneconomical to grind finer than the nominal -65 mesh obtained in the grinding circuit unless overgrinding could be prevented. In other test periods higher grades of primary concentrate were obtained, up to 6% Nb_2O_5 but with a sacrifice of about 10% in recovery.

No completely satisfactory method for upgrading the 3 to 6% Nb_2O_5 concentrate with a good recovery was developed during the tests. From a 6.91% Nb_2O_5 primary concentrate a final concentrate assaying almost 20% Nb_2O_5 was obtained at a 73% recovery. However, overall recovery would be about 50% from the original feed. Good recovery of an approximately 6% concentrate was made from a 3.25% primary concentrate.

DISCUSSION OF RESULTS

Very little can be added to the discussion of results given in Mr. Noblitt's report. The continuous testing confirmed that the Amine 220 Duomeen T flotation is a practical method of concentrating pyrochlore from the Oka ore. The grade of concentrate obtainable at various levels of expenditure can now be determined as the overall recovery at 80-90% is satisfactory. Operating details could not be worked out in the Mines Branch pilot circuit due to ineffective air control on the pilot plant Denver cells. However, this should not be a serious problem in mill equipment. Metallurgy should therefore be better in production.

Reagent expense would be quite high unless the tailing water can be reclaimed. It should be possible to grind to -65 mesh without so much overgrinding. Possibly low discharge mills and the use of two stage cyclones for classification would produce a better flotation feed.

More research work on the retreatment of the primary concentrate is necessary before this can be carried out in a continuous test.

The economics of the overall niobium metal production using this process for concentration will depend on the other steps of the process and on the future requirements and market price of niobium.

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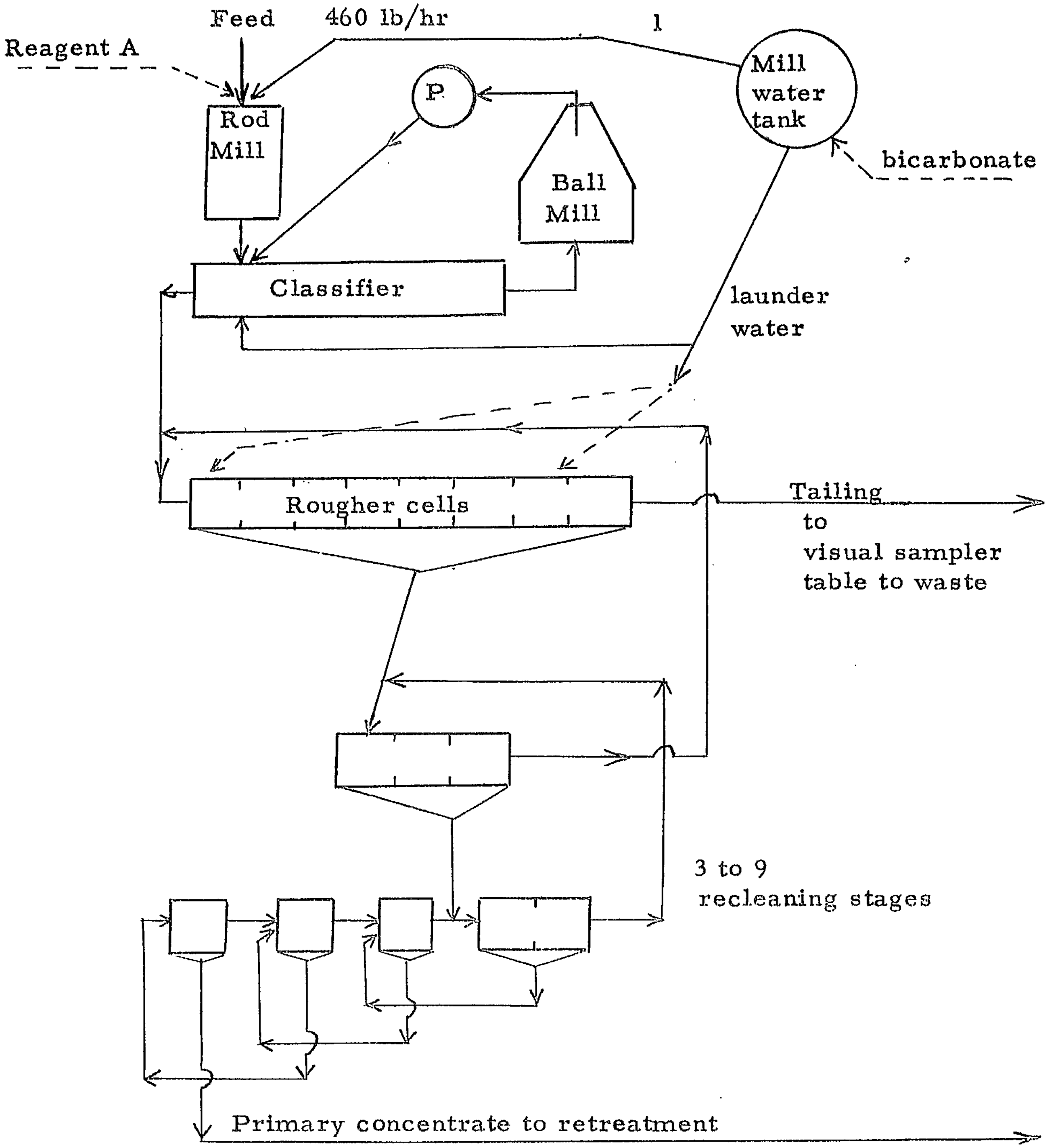

Figure 1 - Flowsheet for Continuous Flotation of Oka Ore.