MINING, ORE PREPARATION AND FERRONIOBIUM PRODUCTION AT CBMM

Octaviano de Souza Paraiso Filho and Raphael de Fuccio, Jr.

Companhia Brasileira de Metalurgia e Mineração
Rua Padre Joao Manoel 923 6
01411, Sao Paulo, SP
Brasil

Abstract

The paper presents a chronology of the history and development of the Araxa carbonatite complex with respect to the production of niobium products.

Mining and geology are described, including the background on methods used for defining reserves. The ore treatment technology and material flow patterns are described in detail, together with descriptions on impurity removal by calcination and leaching.

Ferroniobium is produced aluminothermically in simple, yet massive, reactors and details of the process are given.

Introduction

The pyrochlore deposit of Araxa, Brasil, is widely recognized today as the world's largest known niobium reserve. This mineral deposit, which represents more than 70 percent of the world's economically extractable known reserves, has been mined by CBMM (Companhia Brasileira de Metalurgia e Mineracao) since the beginning of the 1960's and, for the last fifteen years has supplied more than 60 percent of the world niobium consumption.

CBMM's niobium deposit occurs in the Araxa carbonatite complex, located about 6 km south of the city of Araxa in the state of Minas Gerais. Figure 1 shows the map of Brasil and location of Araxa, about 400 air kilometers north of the city of Sao Paulo and approximately 550 air kilometers northwest of Rio de Janeiro.

The purpose of this paper is to describe CBMM's present exploration of the Araxa deposit, its ore preparation and production of ferroniobium. Besides standard ferroniobium, CBMM has recently commenced production at Araxa of vacuum-grade ferroniobium and nickel niobium, as well as niobium oxide in different grades of purity, ranging from the high purity grade for metallurgical applications to especially pure oxides for optical and electronic applications.

History

The presence of pyrochlore in the Araxa carbonatite was first identified in 1954 by the eminent Brasilian geologist, Professor Djalma Guimaraes, who had at an earlier date, confirmed the presence of significant phosphate values in the deposit (1).

Aerial photography, geological mapping, and radiometric surveys, both ground and aerial, were carried out and defined the extent of the pyrochlore mineralization with its unique concentrations of niobium.

Beneficiation studies of the Araxa pyrochlore deposit began in 1958 when a 50-ton sample of ore was sent to the United States for evaluation. During 1959, beneficiation studies conducted on site in Araxa were successful in producing a concentrate containing 55 to 60 percent Nb_2O_5, with a total recovery of between 60 to 80 percent of the niobium values in the ore.

In March 1960, construction was started on the first beneficiation plant, with a treating capacity of 500 tons of ore per day; initial production of concentrates began in April, 1961.

Between 1961 and 1965, the company had difficulties with various impurities in the concentrate, the most significant being silicon, phosphorus and sulfur. These difficulties were overcome by reducing the silica and sulfur content in the flotation process and reducing phosphorus in a caustic soda leaching plant, built in 1964. Finally, by 1965, it became possible to meet all the specifications for steelmaking grade ferroniobium.

Later in the 1970's, customer attention rotated to the relatively high lead content in CBMM's ferroniobium. The company thus developed a new concentrate treatment process involving high temperature calcination with calcium chloride to volatilize lead chloride, followed by a hydrochloric wash to dissolve phosphorus and sulfur.

Figure 1 Map showing location of CBMM's Araxa mine.

Production of ferroniobium from leached and calcined concentrate, produced by either the caustic soda or the calcium chloride process, began in 1965, using the established aluminothermic reduction process, and at present, the majority of CBMM's supply of niobium is in the form of ferroniobium. At present, no pyrochlore concentrates are exported by CBMM for production of ferroniobium by other converters.

In January 1980, CBMM developed the capability to produce high grade niobium oxide in large quantities. The oxide plant's nominal capacity is 2,400 tons per year of niobium oxide (the equivalent of more than 5 million pounds), making this product extensively available for the world market.

Finally, during the present year CBMM developed a process, and has almost completed construction of a plant to produce especially pure grades of niobium oxide for optical and electronic applications.

Potential constraints on availability of niobium in higher purity forms have thus been dissipated, which could in turn stimulate interest in additional uses.

Geology and Mineralogy

The Araxa carbonatite is part of an important alkaline district where the presence of carbonatites has been established in seven complexes. These complexes are known by the names of Tapira, Araxa, Salitre I, Salitre II, Serra Negra, Catalao I and Catalao II.

Several important mineralizations predominate in these carbonatites: niobium in Araxa, Catalao I and II; apatite, a source of fertilizer phosphate in Araxa, Tapira and Catalao I; and titanium in Tapira, Catalao I, Serra Negra, Salitre I and II. Other less important mineralizations of vermiculite, barite, magnetite and rare earth minerals also occur, but are not yet commercially exploited.

The Araxa complex, approximately circular in shape and about 4.5 km in diameter, is distinguished by the unique extent of its niobium mineralization, with an average content higher than 2.5 percent Nb_2O_5 and isolated mineralized zones containing up to 5 percent Nb_2O_5.

The main geological features of the carbonatite, shown in Figure 2, comprise the Precambrian rocks of the Araxa Group arched in a domic structure by the alkaline intrusion which occured during the Upper Cretaceous period.

The Araxa complex is emplaced in quartzitic rocks with some interbedded schists, which are overlain by a unit of biotite muscovite schists and chlorite muscovite schists with some interbedded quartzites. The predominant quartzitic unit is in contact with the Araxa complex, forming a complete ring surrounding the intrusion.

The high niobium concentration of the deposit, located in the central part of the chimney, is the result of deep weathering and a strong residual enrichment of the original carbonatite rock. In the pyrochlore mineralized zone, also circular in shape and approximately 1,800 m in diameter, this weathering reaches depths of more than 100 meters, and in some places more than 200 meters before fresh rock is encountered.

Although the fresh carbonatite rock is also known to contain niobium mineralization, only the lateritic weathered mantle, the result of the

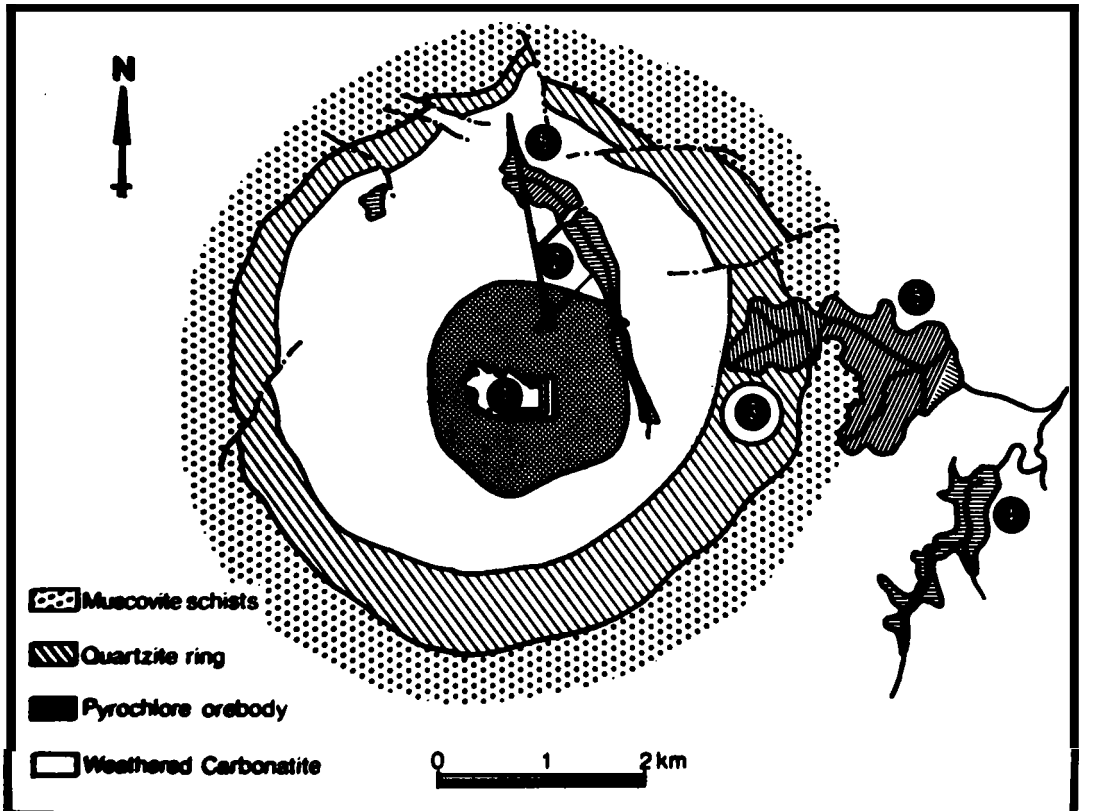


Figure 2. Geological map of the Araxá carbonatite. (1) Mine; (2) Plantsite; (3) New flotation plant; (4) Fresh water pond; (5) Tailings ponds.

residual enrichment of the deposit is included in the ore reserves. The ore is capped by a layer of dark red overburden, grading less than 0.4 percent Nb_2O_5 and varying in thickness from 0.5 meter to more than 40 meters.

The delineation between overburden and the ore zone is easily distinguished due to the sharp transition in color, from the dark red of the overburden to the light brown of the ore, this being an important feature to locate contact between overburden and ore during the mining operations.

The ore is composed principally of the minerals geothite, limonite, magnetite, barite, monazite, ilmenite, gorceixite, quartz and the niobium mineral pandaite, a bariopyrochlore.

The average mineralogical. composition of the in-place ore is shown in the following table (2).

Table I: Average Mineralogical Composition of the Ore

	% Weight
Pandaite (Bariopyrochlore)	4.6
Limonite, Goethite ($Fe_2O_3.nH_2O$)	35.0
Barite ($BaSO_4$)	20.0
Magnetite (Fe_3O_4)	16.0
Gorceixite (Ba, Ce) $(Al, Fe)_3(PO_4)_2(OH)_5 \cdot H_2O$	5.0
Monazite ($CePO_4$)	5.0
Ilmenite ($FeTiO_3$)	4.0
Quartz (SiO_2)	5.0
Others	5.4

Niobium occurs in the mineral pandaite, which is essentially a complex oxide hydro-oxide of niobium, barium, titanium, rare earths of the cerium group, and thorium. The ore is radioactive due to presence of thorium (0.12% in the ore) in the pandaite and monazite minerals. Table II shows a complete chemical analysis of a representative ore sample.

The pyrochlore group has the general formula $A_{2-m}B_2O_6(O,OH,F)_{1-n}pH_2O$ where A is represented primarily by sodium and calcium and B primarily by niobium to yield the usually-cited theoretical formula, $NaCaNb_2O_6(OH, F)$, (3). Appreciable amounts of thorium, lead, trivalent rare earths, zirconium, and uranium, may substitute for sodium and calcium in the A positions of the crystal lattice. In the B positions of the crystal lattice, appreciable amounts of tantalum, titanium and iron, may substitute for niobium.

Table II. Average Chemical Composition of the Ore

	% Weight
Nb_2O_5	3.0
Ta_2O_5	Traces
P_2O_5	3.32
TiO_2	3.60
SiO_2	2.38
ZrO_2	0.20
ThO_2	0.13
Al_2O_3	1.19
Fe_2O_3	46.52
$(RE_2)O_3$	4.44
SO_3	8.82
BaO	17.73
MnO_2	1.82
U_3O_8	0.008
LOI	6.02
TOTAL	99.178

The specific name of the niobium-bearing mineral in the Araxa ore is pandaite, a pyrochlore in which Na^{+1} and Ca^{+2} are virtually absent and have been partly replaced by Ba^{+2} in the A positions of the crystal lattice. The chemical analysis of the mineral corrected for impurities is given in Table III.

The name pandaite, honoring the Panda Hill carbonatite in Africa where this niobium mineral was first identified, was given by Jager, et al, in 1959 to those minerals of the pyrochlore group in which Ba^{+2} predominates over all other ions occupying the A positions of the crystal lattice (4).

More recently, the chemical designation bariopyrochlore was recommended as a replacement for Pandaite by the IMA Commission, but Pandaite is still in common use (3).

Table III. Complete Chemical Analysis of the Araxa Pandaite*

	%
Nb_2O_5	63.42
Ta_2O_5	0.15
BaO	16.51
CaO	0.44
$(RE)_2O_3$	3.29
ThO_2	2.34
MnO	0.16
FeO	2.37
TiO_2	2.30
PbO	0.42
SnO_2	0.10
H_2O	8.50

*Recalculated to 100% after deduction of impurities

Ore Reserves

Since the early days of its discovery, the Araxa pyrochlore deposit has been extensively studied as to the extension of its mineral reserves. First studies were carried out by the National Department of Mineral Production, with a drilling program that was later supplemented by CBMM. A total of 42 diamond drill holes were bored in the area, adding up to 5,700 meters drilled. Drill holes, in general, were stopped just after fresh rock was reached.

These studies confirmed the continuous vertical mineralization of niobium in all the lateritic mantle of the deposit above bedrock, and also supplied sufficient data for evaluation of the Araxa reserves. However, the program did not provide enough detailed information for mine development and ore extraction planning.

Thus, for mine planning and more detailed evaluation of the overall niobium reserves in Araxa, CBMM carried out an extensive exploration program which consisted of systematic digging of 1-m square test pits. The mineralized area was covered by test pits spaced in a 120m x 120m grid; test pits were dug to depths up to 45 meters, depending on the topography of the surface of the mineralized area. In areas of higher Nb_2O_5 concentration, the grid was later closed to a 60m x 60m grid, this also with the objective of aiding in future mine planning and for ore control in the mining operations.

Important additional information gained from this exploration program were precise definition of the contact between overburden and ore and information on the depth of the water table, in the mineralized areas.

241 such test pits were completed, totaling 8,084 meters. Because of the favorable nature of the ground which is very stable with no need for artificial timber supports, very little equipment was necessary to dig the test pits. Channel samples were taken and analyzed for each meter of the vertical length of the test pits. Average grades were computed at 10 meter intervals.

This work, combined with previous diamond drilling results, made possible using the criteria defined below, a re-evaluation of the Araxa reserves that are summarized as follows:

Table IV. Total Ore Reserves

	Thousands of Tons	$\%Nb_2O_5$
Measured	131,612	2.50
Indicated	41,793	2.49
Inferred	288,349	2.50
Total	461,754	2.50

Measured Reserves

(according to different cut-off values)

Cut-off Grade	Thousands of Tons	$\% Nb_2O_5$
3.0 %	35,700	3.54
2.5 %	65,000	3.15
2.0 %	98,300	2.88
1.0 %	131,600	2.50

Measured Reserves

The mineralized ore body was divided into benches 10 meters high with each bench, in turn, in 60m x 60m square blocks, so that each block has a volume of 36,000 cubic meters, which based on the ore's apparent density of 2.2, represents 79,200 tons of ore.

A weighted grade of Nb_2O_5, inversely proportional to the square of the distance between the sample point and the center of the block, was attributed to each block, according to the following standard formula:

$$t = \frac{\sum_{i=1}^{i=n} \frac{t_i}{d_i^2}}{\sum_{i=1}^{i=n} \frac{1}{d_i^2}} \qquad (1)$$

where,

t = grade of the block
t_i = grade of the test pit in a bench (average grade of the 10-meter interval of test pit in that bench)
d_i = distance between test pit and center of the block
n = number of test pits that influenced the block

The sum of all such blocks, to which confident grades of Nb_2O_5 could be attributed, is measured ore.

Indicated Reserves

Two considerations were made to calculate indicated reserves:

1. Since reliabiity of the average grade of Nb_2O_5 of a block is reduced for blocks located more than a certain distance ($d = 3/4 \times 120 \sqrt{2} = 127.3$ m) away from the nearest sample point, all such blocks were considered as indicated ore. Here it was not a question of discontinuity of the ore-body, but of a lack of confidence in the grade content.

2. Also, the first two blocks placed immediately below a vertical pile of measured reserve ore blocks were considered as indicated ore blocks. The summation of all blocks, as determined above, constitutes the indicated reserves.

Inferred Reserves

Since almost all drill holes went to depths that reached fresh rock, the contact between decomposed ore and fresh rock could be determined.

The total volume of the weathered mantle above fresh rock was then calculated and inferred ore was obtained by subtracting from this total volume, the volumes of overburden and of measured and indicated ores. As the assays of the drill cores did not indicate any reduction of Nb_2O_5 values, the average grade of the measured and indicated ore reserves was attributed to the inferred reserves.

In order to investigate the fresh rock underlying the weathered material, CBMM has just finished a deep hole drilling program. Six holes were completed to depths of 600 to 800 meters and results revealed high niobium values in the fresh carbonatite, averaging more than 1 percent Nb_2O_5; in some zones results as high as 3 percent grades were found. None of these drill holes went past the niobium mineralization zone.

Mining the Ore

Because of the altered and decomposed nature of overburden and ore materials of the Araxa open pit mine, no drilling and blasting are necessary in the stripping and mining operations.

Equipment used are heavy duty bulldozers, front-end loaders and dump trucks.

The ore is mined in benches 10 meters high; mining cut-off grade is kept at around 2 percent Nb_2O_5, and an average of 3 percent Nb_2O_5 is sought as concentrator feed material. Present mining rate is about 3,500 tons of ore, dry basis (3,900 tons, run-of-mine) per day, in one shift.

Starting in March, 1982, a 1.06-m (42") belt conveyor, driven by two 450-HP motors, will transport the ore to the concentrator, located 3.2 km from the mine site. The new belt conveyor will have a nominal capacity of 1,000 tons of ore per hour. All overburden, at present being used for dam construction, will also be carried by this conveyor in a second operation shift.

The feed station of the conveyor is being constructed near the border of the mineralized area, at an elevation 20 meters below the lowest mine bench being mined at the present, so that in the future loaded trucks will travel downhill a distance of only about 500 meters to the conveyor feed station.

Concentrator

New crushing and concentrating facilities were recently built by CBMM on a new site located at a distance of 3.2 km from the mine pit. The new concentrator was designed to treat 3,500 tons of ore per day and has replaced the old beneficiation plant, built in 1960 and expanded in 1965, and again in 1970. Rated annual production of the present mill, in the initial phase, is 42,000 tons of pyrochlore concentrates containing 60 percent of Nb_2O_5 (or the equivalent of 55 million pounds of contained Nb_2O_5).

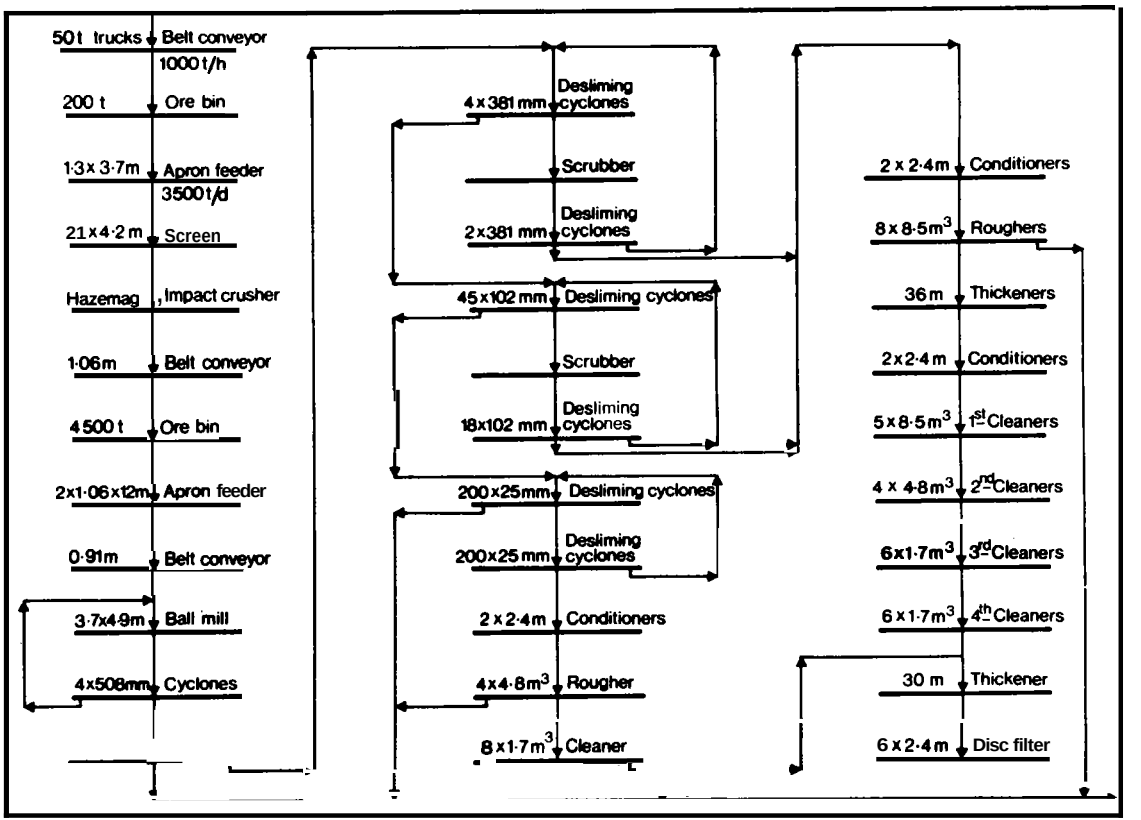
A second phase is planned in which the concentrator capacity will double to 84,000 tons per year (110 million lbs. of Nb_2O_5). Ore silos, concentrator building, and a second ball mill are already completed for this second phase. Also, crushing facilities and all necessary infrastructure were built for four times present concentrator's capacity.

The main steps of the concentration process are shown in Figure 3. The various stages are:

- Crushing
- Ball-mill grinding
- Magnetic separation
- Desliming
- Froth flotation

Crushing the Ore

Normal mine ore is almost completely desegregated and decomposed, and does not require heavy crushing. Crushing facilities consist of a 200-ton concrete ore bin, a 1.3m x 3.7m (54" x 12') apron feeder which delivers the ore to a 2.1m x 4.2m (7' x 14') vibrating screen having a 50mm (2") opening. The screen oversize, which is less than 10 percent, is delivered to a Hazemag type impact crusher. Crushed ore at -50mm (2") and screen undersize are discharged into a 1.06m (42") belt conveyor for delivery to the 4,500-ton capacity ore silo. Stockpiled ore is withdrawn from this silo, in two lines, by two 1.06m x 12m (42" x 39') apron feeders that discharge to a 0.91m (36") belt conveyor. An automatic weight balance weighs the ore in the belt conveyor and controls the speed of the apron feeders. The belt conveyor discharges directly into the grinding ball mill.


50 t trucks
Belt conveyor
1000 t/h
200 t
Ore bin
1·3 x 3·7m
Apron feeder
3500 t/d
21 x 4·2 m
Screen
Hazemag
Impact crusher
1·06m
Belt conveyor
4500 t
Ore bin
2x1·06x12m
Apron feeder
0·91m
Belt conveyor
3·7x4·9m
Ball mill
4x508mm
Cyclones
4 x 381 mm
Desliming cyclones
Scrubber
2x381 mm
Desliming cyclones
45x102 mm
Desliming cyclones
Scrubber
18x102 mm
Desliming cyclones
200x25mm
Desliming cyclones
200x25 mm
Desliming cyclones
2 x 2·4m
Conditioners
4x4·8m3
Rougher
8 x 1·7m3
Cleaner
2 x 2·4m
Conditioners
8 x 8·5m3
Roughers
36 m
Thickeners
2x2·4 m
Conditioners
5 x 8·5m3
1st Cleaners
4 x 4·8m3
2nd Cleaners
6x1·7m3
3rd Cleaners
6 x 1·7m3
4th Cleaners
30 m
Thickener
6 x 2·4 m
Disc filter

Grinding

Pyrochlore crystals of the Araxa ore are very small, seldom exeeding 1mm in size. Optimum liberation is achieved after grinding to 95 percent minus 104 microns (150 mesh). The grinding section consists of one overflow type 3.7m x 4.9m (12' x 16') ball mill, driven by a 1,250-HP motor, in closed circuit with 508mm (20") cyclone classifiers. Two identical grinding sections, each with a 1,250-HP ball mill, are installed, but only one section is at present being operated.

Size distribution and compositions of the ball mill feed and ball mill product are shown in Tables V and VI. The niobium content is seen to be higher in the finer material.

Table V. Screen Analysis of Ball Mill Feed

Microns	Mesh	% Weight	% Weight Cummul.	% Nb_2O_5	% Distrib.	% Distrib. Cummul.
+ 4,700	(+ 4)	6.3	6.3	1.19	2.4	2.4
+ 3,327	(+ 6)	3.0	9.3	1.73	1.7	4.1
+ 1,651	(+ 10)	7.7	17.0	1.60	4.0	8.1
+ 1,168	(+ 14)	2.7	19.7	1.87	1.6	9.7
+ 833	(+ 20)	6.7	26.4	2.03	4.4	14.1
+ 589	(+ 28)	6.2	32.4	2.19	4.4	18.5
+ 417	(+ 35)	5.8	38.4	2.39	4.5	23.0
+ 295	(+ 48)	5.5	43.9	2.68	4.8	27.8
+ 208	(+ 65)	6.3	50.2	2.71	5.7	33.5
+ 147	(+ 100)	8.1	58.3	3.18	8.2	41.7
+ 104	(+ 150)	6.6	64.9	3.16	6.7	48.4
+ 74	(+ 200)	4.9	69.8	3.44	5.4	53.8
+ 53	(+ 270)	4.5	74.3	3.73	5.4	59.2
+ 37	(+ 400)	4.4	78.7	4.29	6.1	65.3
- 37	(- 400)	21.3	-	5.07	34.7	-
		100.0	100.0	3.10	100.0	100.0

Table VI. Screen Analysis of Ball Mill Product

Microns	Mesh	% Weight	% Weight Cummul.	% Nb_2O_5	% Distrib.	% Distrib. Cummul.
147	100	-	-	-	-	-
104	150	6.3	6.3	2.31	4.5	4.50
74	200	12.7	19.0	2.42	9.51	14.01
53	270	18.0	37.0	2.60	14.51	28.52
37	400	18.0	55.0	2.81	15.66	44.18
-37	400	45.0	-	4.00	55.82	-
		100.0	100.0	3.22	100.0	100.0

Magnetic Separation

The Araxa ore contains 10 to 25 percent magnetite that is removed by low intensity (800-900 Gauss), double-drum magnetic separators. The non-magnetic pulp goes for desliming and the magnetic separator concentrates (MSC), containing about 67 percent Fe, are pumped to a separate stockpile area near the mill site for future use.

Desliming

Flotation of the pyrochlore mineral at Araxa is highly sensitive to the presence of slimes which, in certain quantities, may inhibit the flotation process. The very nature of the lateritic ore implies the existence of considerable amounts of minus 5-micron primary slimes, in addition to secondary slimes which are formed in the grinding circuit.

Although Table VI shows a concentration of Nb_2O_5 values in the minus 34-micron (40 mesh) fractions, sedimentation tests show that most of these values are in the plus 5-micron fractions; so desliming at a minus 5-micron split is an important step prior to flotation in that it eliminates about 12 percent of the weight, but only 5 to 7 percent of the Nb_2O_5 content of the feed.

Therefore, since the fractions between 37 and 5-microns are very high in niobium content and the minus 5-micron material seriously disturbs flotation, it is fundamental to the process that the desliming steps be very efficient. For this reason, three-stage desliming is used, with each stage followed by a re-desliming step.

First-stage desliming is carried out in 381mm (15") cyclones; cyclone underflows, at 70 percent solids, go to attrition machines for scrubbing before being re-deslimed in another battery of 381mm (15") cyclones. Overflows of the re-deslimniig cyclones return to the feed of the first-stage cyclones.

Overflows of the first battery of 381mm (15") cyclones feed another battery of 102mm (4") cyclones, for second-stage desliming, where a similar circuit of scrubbing and re-desliming is repeated. Both underflows of the re-desliming 381mm (15") and 102mm (4") cyclones are feed of the main roughers circuit.

In the third and final stage of desliming, the 102mm (4") cyclones overflows are deslimed and re-deslimed in 25mm (1") cyclones, this time without scrubbing. The underflows are flotation feed of a separate and independent set of roughers for fine-particle flotation.

Flotation Circuit

Concentration of the niobium mineral is by selective froth flotation of the pyrochlore. Deslimed pulp is conditioned for 15 minutes with an amine type cationic collector, a wetting agent and sodium fluorsilicate which is used as a pyrochlore activator. The flotation circuit is acidic with hydrochloric acid used to control pH at 2.5 to 3.5.

Rougher flotation consists of a bank of eight $8.5m^3$ (300 ft^3) flotation cells. Rougher concentrates, together with second cleaner tailings, are sent to a 36m (120 ft) thickener to adjust percent solids before the cleaning stages. Thickener underflow, at 25 percent solids, is again conditioned with

additional quantities of activator and collector reagents.

Cleaner flotation consists of four cleaning stages, each in closed circuit with the previous stage. The first cleaners consist of a bank of five $8.5m^3$ ($300 ft^3$) cells; second cleaners use four $4.8m^3$ ($170 ft^3$) cells and, finally, third and fourth cleaners have six $1.7m^3$ ($60 ft^3$) cells each.

A separate circuit is used for flotation of the fine particles from the underflow of the 25mm (1") cyclones which is the last stage of desliming. This circuit consists of one rougher flotation bank, with four $4.8m^3$ ($170 ft^3$) cells, a first-stage cleaner, with six $1.1m^3$ ($40 ft^3$) cells, and a second cleaner, with four $1.1m^3$ ($40 ft^3$) cells. Concentrate from the second cleaner step joins the fourth cleaner concentrate of the regular flotation circuit to form the final flotation concentrates.

Filtering

Flotation concentrates are thickened in a 30m diameter (100 ft) thickener and filtered in a 2.4m x 6 (8' x 6) disc filter; flotation concentrates, having 15 percent moisture, are then packed in 3,000 kg bag containers and transferred to the leaching plant.

Tailings Disposal and Concentrator's Water Supply

Except for magnetic separator concentrates which are stored in a separate stockpile, all concentrator tailings go to a large tailings pond for sedimentation of the solids. This tailing pond is contained by an earth-fill dam, situated in a valley approximately 600m from the plant site.

Water reclaimed from the settled solids is pumped for additional clarification to a second pond, formed as a result of a second earth-built dam further down in the valley. Clear water from the second lake is pumped back to the mill where it is re-used in the flotation process. All water used in the process is recycled and there is no effluent discharge. Make-up water is pumped from a fresh water lake, fed from a stream situated in another valley 2 km from the plant site.

Leaching Process

Flotation concentrates have the following approximate chemical composition shown in Table VII.

Because of its relatively high phosphorous, sulfur and lead contents, flotation concentrates are treated in a calcining and leaching plant, where the levels of those impurities are reduced to much lower levels prior to production of ferroniobium. Until 1976, only phosphorous was leached out of the concentrate by a process which consisted of baking a mixture of the flotation concentrate with 20 percent caustic soda in a rotary kiln at 200 C. Baking being followed by water leaching and filtering steps, in order to dissolve and eliminate phosphorous.

In 1976, a new process was introduced which simultaneously reduces the levels of phosphorous, sulfur and lead, and this is used at the present. This leaching process is shown in Figure 4, and consists of the following steps.

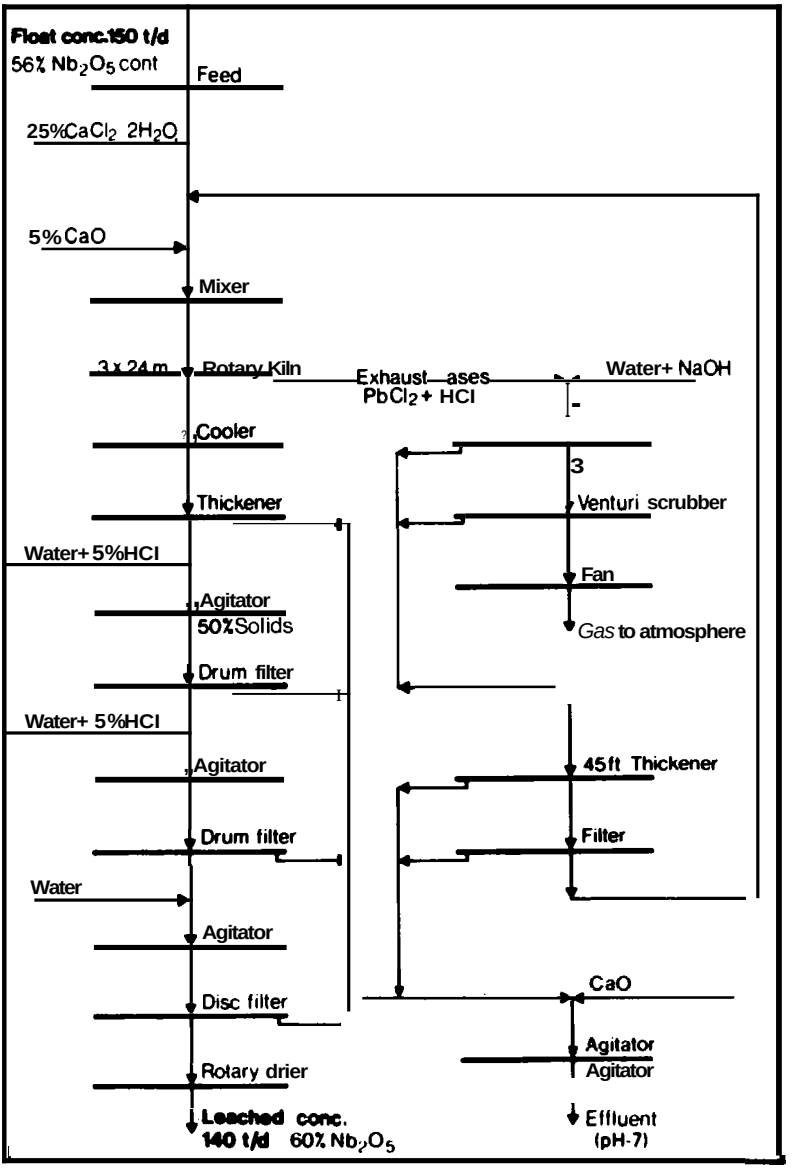


Figure 4. The leach plant flowsheet in its present form, capable of reducing the phosphorus, sulphur and lead in the concentrate to very low levels.

Table VII. Typical Chemical Composition of Flotation Concentrate

	%
Nb_2O_5	55 to 60
Fe_2O_3	2 to 8
SiO_2	0.1 to 0.5
ThO_2	2 to 3
U_3O_8	0.05 to 0.10
P	0.3 to 0.8
S	0.02 to 0.2
Pb	0.2 to 1
BaO	15 to 18
CaO	0 to 0.2
H_2O	5 to 7

1. Calcination of a mixture of flotation filter cake with 25 percent calcium chloride and 5 percent lime, in a rotary kiln at a temperature of 800 C to 900 C.

2. Cooling and leaching the calcined mixture, at 50 percent solids, with 5 percent hydrochloric acid, followed by filtration; this second step is done in two stages.

3. The concentrate is then washed with water, also at 50 percent solids, filtered, calcined and packed in containers, and is ready for ferro-niobium production.

During calcination, hydrochloric acid and lead chloride are formed; these are volatilized and picked up by a cooling tower, venturi and packed tower. All collected gases and dust are sent to a 13.7m (45 ft) thickener. Solids in the thickener underflow are filtered and return to the beginning of the process, and the overflow, containing hydrochloric acid and lead chloride, goes to a tailings pond.

Also during calcination, calcium replaces barium in the crystal structure of the pandaite mineral, and barium chloride is formed which is eliminated in the filtrate, together with soluble phosphorous.

The following, Table VIII, shows an analysis of the leached concentrate, with the typical ranges of composition; reductions in barium, phosphorous and lead, in comparison with flotation concentrates, are noticeable.

The leaching plant has a nominal capacity to treat 150 tons of concentrate per day, allowing an annual production of approximately 50,000 tons of leached concentrates.

Table VIII. Typical Chemical Analysis of Leached Concentrate

	%
Nb_2O_5	59 to 65
Fe_2O_3	2 to 8
SiO_2	0.1 to 0.5
ThO_2	2 to 3
U_3O_8	0.05 to 0.10
P	0.05 to 0.10
S	0.010 to 0.05
Pb	0.010 to 0.050
BaO	1 to 3
CaO	15 to 20
H_2O	0.1 to 0.3

All leaching plant effluents, namely, thickener overflows and filtrates are treated with milk of lime before going to the tailings disposal system.

The tailing disposal system presents no environmental problems in that it is a totally enclosed system with no effluent discharge. As with the concentrator facility located elsewhere on the property, water reclamation from the leaching plant's tailings pond is completely incorporated into the process with fresh make-up water added as necessary to compensate for evaporation from the pond.

Standard Ferroniobium Production

The major part of the niobium produced at Araxa is exported in the form of ferroniobium as this is the form in which most of the metal is used.

Batch wise, aluminothermic reduction is the standard process for making ferroniobium from pyrochlore concentrates. The Araxa ferroniobium batch operation is probably the largest aluminothermic reaction facility in the world in terms of reactor size, quantity of aluminum powder used and tons of ferroniobium produced by each reaction.

A typical reaction charge at CBMM is as follows:

Pyrochlore concentrate	18,000 kg (60% Nb_2O_5)
Iron oxide (as hematite)	4,000 kg (with 68% Fe)
Aluminum powder	6,000 kg
Fluorspar	750 kg
Lime	500 kg

The above charge will produce approximately 11 tons of 66 percent ferroniobium. CBMM has a capacity to fire six charges a day and is presently operating at the rate of 240 days/year. Recovery of niobium is of the order of 96 to 97 percent.

Each charge is mixed individually in a rotary mixer for 90 minutes and unloaded into steel containers to wait for reaction time.

The reactor vessel consists of a magnesite brick lined steel cylinder measuring 3.7 meters in diameter by 1.80m in height.

Reactors are placed on top of circular concave pits lined with a mixture of lime and fluorspar which have been previously prepared in a silica sand bed. The outside bases of the reactors are sealed with a mound of sand. Charges are poured into the reactor vessels and the exothermic reduction is initiated with a mixture of sodium peroxide and aluminum powder.

This "fuse" mixture can be ignited either with a flame or by pouring on a small quantity of water. The main reduction reaction is spontaneous and lasts about 15 minutes. The maximum temperature during the reduction reaches 2,400 C, and the slag and liquid niobium/iron alloy gradually separate into two layers with the slag floating on top of the metal. Most of the gangue impurities from the concentrate enter the slag, including all the thorium and uranium oxides. When the reaction is finished, the slag is tapped from the vessel and left in the sand bed to cool and solidify. The vessel is lifted leaving the metal in the sand.

Sixteen hours after the reaction is finished, the slag is removed from the sand bed and transferred to a 35-ton dump truck; the truck bed is protected against the excessive heat of the slag by a layer of earth. Slags are hauled to a disposal area.

The ferroniobium "button" is kept in the sand bed for several hours until it solidifies; it is then removed from the sand bed and after 12 more hours of cooling in air, the ferroniobium is crushed to minus 100mm in a jaw crusher, then sampled and stored in 1,000 kg steel containers.

The minus 100mm ferroniobium is later re-crushed, sieved and sized according to customers specifications, and packed for shipping.

Typical compositions of ferroniobium and slag are shown in Table IX.

It can be seen that while iron and phosphorous pass into the ferroniobium, other metals such as titanium, thorium, uranium and the rare earths are not reduced, and remain in the slag.

Personnel

The total work force of the mining and processing facilities of CBMM at Araxa, including administrative personnel, is 406 workers and 93 staff.

Conclusions

The development of the Araxa pyrochlore deposit has been underway for two decades. During this time, partly due to expansion of the market, CBMM has become the major world supplier of ferroniobium and niobium oxide. In the near future, additional forms of niobium will be offered to satisfy the growth in new areas of consumption.

Table IX. Typical Chemical Analysis of Ferroniobium and Slag

	Ferroniobium		Slag
Nb	66.0%	Al_2O_3	48%
Fe	30.5%	CaO	25%
si	1.5%	TiO_2	4%
Al	0.5%	BaO	2%
Ti	0.1%	RE_2O_3	4% (Rare Earth oxides)
P	0.1%	Nb_2O_5	Traces
S	0.04%	ThO_2	2%
C	0.08%	U_3O_8	0.05%
Pb	0.02%		

References

1. Guimarães, D., Djalma, "Relatório Sobre a Jazida de Pirocloro de Barreiro", Araxá, M. G.", Boletim 103, Divisao Fomento Producao Mineral - DNPM, Belo Horizonte, Brasil, 1957.

2. Jaffe, H. W., and Selchow, D. H., "Mineralogy of the Araxa Columbium Deposit", Union Carbide Ore Company Research Report No. 4., Research Center, Tuxedo, N. Y., March 14, 1960.

3. Hogarth, D. D., "Classification and Nomenclature of the Pyrochlore Group", The American Mineralogist, Vol. 62, p.p. 403 - 410, 1977.

4. Jager, E., Niggli, E., and Van der Veen, A. H., "A Hydrated Barium Strontium Pyrochlore in a Biotite Rock from Panda Hill, Tananyka", Mineral Magazine, Vol. 32, p.p. 10-25, 1959.