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## THE OUTLOOK FOR PYROCHLORE CONCENTRATES: A REVIEW

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THE OUTLOOK FOR PYROCHLORE CONCENTRATES:

A REVIEW\*

Abstract

World production of pyrochlore has recently outstripped that of columbite, previously the main source of niobium. Columbite is largely a by-product of tin-mining, but the new pyrochlore mines depend almost entirely for their success on an increasing market for ferroniobium. In spite of recent production and price cuts, it is envisaged that the longer term outlook for pyrochlore concentrates is good even if major breakthroughs in metal usage are discounted.

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## I. INTRODUCTION

The mineral pyrochlore, a fluoniobate of sodium and calcium, has been in commercial production as an ore of niobium for only eleven years. During that time the production of pyrochlore concentrates has increased rapidly and, in 1964, total output exceeded that of columbite, the traditional source of niobium, for the first time. These developments have had considerable impact on world markets for several reasons, namely, (1) the two largest operating pyrochlore mines lie in the western hemisphere, i.e., in Canada and Brazil; (2) world reserves of pyrochlore are much greater than those of columbite; (3) unlike columbite, which is normally produced as a by-product or co-product of cassiterite, pyrochlore is mined for its own sake; (4) the (alkaline) carbonatite ring complexes which contain large reserves of pyrochlore are compact bodies of small surface area, capable of being mined intensively.

With the advent of large pyrochlore producers, consumers of ferroniobium and niobium were assured of a constant and dependable source of supply, and the mining companies involved, unlike those producing columbite, depended almost entirely for the success of their operations on an annual demand for pyrochlore running into some thousands of tons.

## II. PRODUCTION OF NIOBIUM CONCENTRATES

The outputs of pyrochlore, columbite and columbite-tantalite from leading producer countries for the last twelve years are shown on Table A. The high production for the year 1955 was induced by heavy U.S. stockpile buying. Figures for subsequent years indicate an increasing trend of production which may be assumed to be more truly related to consumption.

Since 1960, prior to which Norway was the only pyrochlore producer, the pattern of output has been greatly modified by the rapid expansion of pyrochlore production in Canada and Brazil, and the displacement of columbite by pyrochlore as the major source mineral of niobium. In Nigeria, still the largest single producer of columbite concentrates, the grade of ore mined averages less than 0.025 per cent.  $\text{Nb}_2\text{O}_5$  as compared with greater

than 0.4 per cent or more  $Nb_2O_5$  for the operating sources of pyrochlore. These differences in grade must, of course, be balanced against the much lower costs of producing a high grade concentrate in Nigeria where columbite for the most part is a co-product of tin mining and can be recovered from alluvial sands by comparatively simple dressing methods.

In Canada the single producing mine at Oka, owned by St. Lawrence Columbiun and Metals, Ltd., turned out 1,324 short tons of  $Nb_2O_5$  in concentrate in 1965. Mill capacity is about 1,300 tons of ore per day averaging 0.45 per cent pentoxide. Recent reports state that Columbiun Mining Products Limited, which holds property neighbouring that of St. Lawrence Columbiun and Metals, are engaged on building a 1,000 ton per day concentrator and have contracted to supply 2.6 million lb. of niobium pentoxide per annum to Continental Ore Corporation over ten years. No production has yet been recorded, however. Other potential producers in a less advanced stage of development are Multi Minerals Ltd. at Nemegos, Dominion Gulf Ltd. at Lake Nemegosenda, Beaucage Mines Ltd. at Lake Nipissing and Consolidated Morrison Exploration which holds the rights over a recently discovered deposit, estimated to contain at least 40 million tons grading 0.52%  $Nb_2O_5$ , at St. James Bay, northern Ontario.

The mine at Araxa, Minas Gerais, Brazil, owned by Companhia Brasileira de Metalurgia e Mineracao (C.B.M.M.) produced 4,200 long tons of pyrochlore concentrates grading 58%  $Nb_2O_5$  in 1966. Plans for 1966 include an increase in the capacity of the mill to give an annual production figure of 5,000 tons.

The Norwegian concern, Norsk Bergwerk A/S, ceased operations in 1966, but the rights in the Sjøve deposit have been transferred to a partnership comprising Metallgesellschaft AG and Fangel and Co. A/S. These companies hope to recommence production at Sjøve in 1968.

It is estimated that, with the advent of production from the property of Columbiun Mining Products Limited, total world capacity should be in the

region of 13,000 tons of concentrate per annum or approximately 540 tons of pentoxide per month. Many companies, particularly in Canada, seem to be holding deposits while awaiting the turn of events. The U.S. General Services Administration stockpile must also be regarded as a large potential source.

### III. RESERVES

Pyrochlore and associated niobium minerals normally occur in alkaline-carbonatite complexes which generally take the form of ring structures or pipes and consist of a series of somewhat unusual silica-impoverished intrusive and replacement rocks ranging from pure carbonates to nepheline-syenites and fenites. About half of all carbonatite complexes are niobium-bearing (de Kun, 1962), and in about 20 per cent the grade of niobium is sufficiently high to make economic recovery feasible. In some deposits, however, particularly those in which the niobium is concentrated in residual zones of enrichment overlying carbonatite, the problems of beneficiation have so far defied solution. These difficulties obtain at Mrima Hill in Kenya and at Leushe in the Congo.

Some indication of world reserves is given in Table B. Other extensive deposits are known and have been investigated, particularly in Canada.

### IV. PRICES

The following are sample prices for pyrochlore concentrates and for niobium and ferroniobium, published on 1st January 1968.

#### Pyrochlore Concentrates

Canadian, f.o.b. mine or mill per lb. contained $Nb_2O_5$ (long term)	95¢
Canadian " " " " " " " (spot)	\$1.02-\$1.07
Brazilian, c.i.f. U.S. ports " " " (1 year contract/spot)	95.5¢
Comparable 1965 prices:- 1st quarter	\$0.75-\$0.92
4th quarter	\$0.93-\$1.02

Niobium Metal United States per lb.

99.5 - 99.8 per cent roundels, powder-metallurgical	\$11-\$22
-reactor	\$12-\$23
ingot - metallurgical	\$16-\$27
-reactor	\$17.50-\$28.00
according to quantity	

Ferroniobium

U.S. per lb. Nb, ton lots, standard grade	\$2.45-\$2.60
" " " " " " low alloy "	\$2.40-\$2.60
f.o.b. shipping point.	

V. QUALITY REQUIREMENTS

This is best established by comparison with the grades sold by current leading producers. When designing their plant, St. Lawrence Columbium and Metals Limited decided, on the basis of specifications of concentrates offered by traditional columbite producers, that concentrates assaying less than 50 per cent  $Nb_2O_5$  would not be acceptable to consumers. The company offers three grades the specifications of which are shown in Table C.

Similarly, Norwegian pyrochlore concentrates contained a minimum of 50 per cent  $Nb_2O_5$  but average grade increased from this level to 55 per cent in 1965. Brazilian concentrates of pyrochlore produced by DEMA average about 58 per cent  $Nb_2O_5$ .

VI. DEMAND

Table D indicates the trend of imports to the leading consumer countries over the past twelve years. High figures for 1965 derive from U.S. stockpile buying. The demand for pyrochlore concentrates is entirely controlled by demand for ferroniobium, and, to a much lesser extent for niobium metal and alloys.

Over 90% of all niobium concentrates, whether composed of pyrochlore or columbite, go towards the production of ferroniobium. This material,

usually about 65 per cent Nb in alloy in iron, is used as a key addition to various grades of steel. Used in amounts of about  $1\frac{1}{2}$  lb. to the ton the niobium reacts strongly with the carbon content of the steel to form niobium carbide, the presence of which effects a refinement in the grain structure, a gain of up to 20% in strength, a resistance to age hardening and an improvement in impact properties. Such niobium mild steels have become increasingly accepted in the structural steel and steel pipe manufacturing industries. In addition to this function, which itself has been largely instrumental in doubling demand for niobium concentrates since 1959, ferroniobium has been utilised as an addition to high-chromium stainless steels since the 1930's. In this application it is valuable in preventing intergranular corrosion and weld failure by inhibiting the formation of chromium carbide at grain boundaries.

Figures for breakdown of uses of ferroniobium in the United States for the years 1963-65 indicate that the quantity used in low alloy steels has doubled during that period, while that used in stainless and carbon steels has increased steadily but more slowly. Total consumption of ferroniobium in the United States in 1965 has been quoted as 2,198,744 lb. contained niobium. During the same year consumption in Britain was about 1.4 million lb.; this was 25 per cent greater than in the preceding year and three times as high as in 1960. A somewhat fluctuating factor in the pattern of demand is contributed by occasional shortage of vanadium, for which niobium can substitute.

In the long term, the demand for ferroniobium depends on the level of activity in the steel industry, and, discounting recessions in this field, may be expected to become higher with its increasing acceptance as an additive and with increasing use of steel.

By contrast, in the United States the consumption of niobium as metal, and as metal in refractory alloy, is said to have remained in the range

50,000 to 100,000 lb. until the end of 1965. It seems likely, however, that some increase in consumption may have taken place during 1966-67, for **accelerated research on niobium technology** has led to wider possible diversification in its use, though present consumption is largely based on research projects which may be ephemeral in the extreme. Some of the projects in which it is employed are mentioned below. In all of them except superconductivity niobium is liable to substitution, and none of them is likely to lead to a greatly increased demand within the next five years except possibly for alloys in which niobium, while not the main metal, forms an important part.

Predictions made in the late 1950's for a great increase in demand for the metal in the middle 1960's have not thus far been fulfilled, and it is perhaps pertinent to examine the reasons for this. These would appear to be:- (1) the availability and low comparative price of competitor metals, particularly molybdenum; (2) the difficulties which arise from the fact that the metal readily oxidises at temperatures over 500°C; (3) the low capacity uses to which the metal and its alloys can be put and (4) the inadequate information yet available on its exact properties. A number of large business organisations which are pursuing research in niobium technology are doing so, not because of the value of business which results from sale of the refractory metals, but because of the much greater value of possibly marketable products containing refractory alloys in essential parts.

The uses or possible uses of niobium may be broadly categorised as follows: (a) Refractory alloys; (b) Nuclear applications; (c) Superconducting coils and (d) other uses in alloy form in which refractoriness and workability are the important properties, such as wire inserts in other metals for purposes of reinforcement.

In the refractory alloy field, the direct competitors to niobium are tungsten, tantalum and molybdenum. All three metals have higher melting



points than niobium (see Table F) but the low mass of the last is likely to be of advantage in some engineering applications. Tungsten is more than twice as heavy, volume for volume, and is far more difficult to fabricate, while there seems little likelihood that tantalum, also considerably heavier than niobium, will ever be used in any but the most specialised applications because of its high price and poor availability. The properties of molybdenum, on the other hand, lie close to those of niobium, and its price is currently less than one-sixth that of the latter. Moreover, the mining, research and marketing of molybdenum has been carried out with efficiency for many years by a large, vertically-integrated industry on an international scale, with ready sales based on ferrous alloys, so that its capabilities are more widely known among engineers.

The refractory alloys are assumed to be those suitable for operation up to and beyond 1200°F. For temperatures of less than 1000°F alloys based on steel will generally suffice, and these may or may not contain small proportions of molybdenum or niobium. In general, the advantages of molybdenum over niobium are its higher melting point and its greater availability at much lower prices. Those of niobium are its lower density, its better oxidation properties, its unique malleability and greater ease of fabrication. For comparison of other properties, see Table F.

Both molybdenum and niobium are subject to rapid oxidation in the atmosphere at high temperatures, and in the earlier stages of research it was hoped that an alloy could be developed combining the advantages of fabricability, oxidation resistance and strength at high temperatures. These properties were found, however, to be closely interdependent and in giving priority to two of them, the third was lost. The alloy Cb-752, marketed by the Union Carbide Metals Co., was a case in point, for though it could be successfully welded to produce ductile welds at room temperature, and though the strength to weight ratio was exceptional at high temperature, oxidation resistance was poor. Recent attempts to solve this

problem have been concentrated on the development of coatings, particularly ceramic and intermetallic coatings, designed to protect the metal surface from atmospheric gases. Oxide ceramic coatings have proved effective for over 1,000 hours at high temperatures.

Such coatings have also, of course, been developed for molybdenum and its alloys, and at present it can only be supposed that niobium would be preferred largely in applications where lightness of structure is of paramount importance. (See Table E).

The niobium-based and niobium-containing alloys developed for the aerospace industry are too numerous to mention here. Of note is SU16 (83-92% Nb, 11% W, 3% Mo, 2% Hf, 0.08% C) reported to be stronger than any other niobium alloy so far produced, which is manufactured by the Kawecki Chemical Co. Among those in which niobium forms a significant addition are 713 C and 713 LC developed by International Nickel Co. for use in turbine rotor blades and turbine wheels. In the meanwhile, experiments on beryllium-niobium alloys are being pursued in the hope of combining the high-strength, low weight aspects of both metals.

Niobium possesses several attributes which make it attractive in the field of nuclear engineering, including low neutron capture cross section and corrosion resistance in the presence of liquid alkali metals, in addition to the properties given above. Since zirconium lacks high temperature strength the closest competitors in this context are molybdenum and stainless steels, though the latter would not be viable at temperatures of over 1000°F or in contact with liquid metals. The lower price of molybdenum has given that metal a considerable lead in reactor metallurgy and it is now widely utilised, at present to a greater extent in ferrous, than in non-ferrous, alloys. The only current application of niobium to nuclear equipment is as fuel cladding in the fast-breeder pile at Dounreay in Scotland, though it was used in a similar capacity in an experimental atomic generator intended for powering aircraft in the early part of this decade. It is

difficult to be certain if any future exists for niobium in nuclear furnaces; it is most applicable to the liquid metal-cooled type, but it is precisely in this venue that molybdenum competes most strongly. Furthermore, recent work on the fabricability of molybdenum has to a large extent overcome its most serious disadvantage with respect to niobium.

Pure niobium metal becomes superconductive at the comparatively high temperature of 8.5°K and the alloy Nb<sub>3</sub>Sn has the highest critical temperature known of 18°K. The latter substance is stated to remain superconductive in a field of 190 kilogauss at 4.2°K. Niobium-tin or niobium-zirconium, having proved the most promising for this purpose, will almost certainly be utilised in any commercial application of the property of superconductivity (e.g. to energise an M.H.D. generator). Various methods have so far been adopted to overcome the difficulty arising from their inherent brittleness in manufacturing coils of these alloys. Among these is the incorporation of fine wires of niobium-zirconium in thin copper strips, or the lamination of strips of niobium-tin foil between strips of copper. The Everett Research Laboratory of the Avco Corporation of the United States has built a superconducting magnet which uses 117 miles of niobium-zirconium wire in copper strip and is capable of storing  $5 \times 10^6$  joules of energy, and of developing at least 40 kilogauss.

This function is one in which, by the nature of its atomic structure, niobium has no real competitor, but commercial applications are at such an early stage of development that it is impossible to predict the amount of metal likely to find usage in superconducting coils. It is difficult to believe, however, that demand from this source would significantly affect the market for niobium concentrates within the next ten years.

There seems to be little future for niobium in wet applications which can make use of its high resistance to corrosion. In effectiveness it lies between stainless steel, titanium and zirconium, which are relatively cheap, and tantalum, which, though more expensive, is far more efficient.

It should be emphasized that the foregoing indicates some of the uses to which the refined forms of metal may be put and gives a measure of the research to which niobium, in its purer forms, is subject, rather than a sound manufacturing base which will lead to an inevitable growth in demand. The impression gained is that too many possibilities for substitution exist at present for predictions to be other than speculative. A recent report has indicated that consumption of metal has greatly increased during 1966 and it is to be borne in mind that if even one of the products of experiment were to be put to serious commercial production the requirement for refined metal would double or treble within a short space of time. A trebling of demand would lead to an increase of about 6-7% in the demand for concentrates, i.e., very approximately 700 tons per annum. It is doubtful if this would affect the price of concentrates in any significant degree. In the long term, therefore, the demand for pyrochlore concentrates is intimately connected with that for improved ferrous alloys and will probably depend on the level of production and research in the steel industry. Its acceptance as an additive, and the possibility that it may be used in greater proportions per ton of steel, suggests that there will be an increasing market for the concentrates. The signs of overcapacity which have recently developed in the industry, leading to cutbacks in production and 10% lowering of the price, are probably of short-term duration, and are unlikely to persist for more than a few months.

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Metal Bulletin  
Metals Week  
Mining Journal  
Mining World  
The Northern Miner  
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**TABLE A - Production of Niobium Concentrates, leading Countries**

Long tons

	1955	1956	1957	1958	1959	1960	1961	1962	1963	1964	1965	1966
Nigeria, columbite	3,146	2,604	1,923	806	1,588	2,047	2,345	2,264	2,011	2,339	2,548	2,221
Congo Rep. & Rwanda columbite & tantalite	778	611	388	351	318	279	111	177	127	<sup>a/</sup> 154	n.a.	n.a.
Malaya, columbite	236	276	142	159	120	93	95	110	88	56	46	67
Norway, pyrochlore	302	256	190	281	285	341	316	344	377	320	148	-
Canada, pyrochlore	-	-	-	-	-	-	53	852	1,202	1,885	2,013	<sup>b/</sup> 2,300
Brazil, pyrochlore	-	-	-	-	-	-	1,504	100	-	318	1,177	4,200

n.a. = not available

<sup>a/</sup> Rwanda only<sup>b/</sup> estimated

TABLE B - Pyrochlore Reserves

Country	Deposit	<u>Reserves</u> Millions tons	<u>Grade</u> %Nb <sub>2</sub> O <sub>5</sub>
Canada	Oka	62.6	0.4%
	Oka	100	0.3
	Oka	not released	-
	Nemegos	50	0.26
	Nemegosenda Lake	18	0.5
	Lake Nipissing	(0.617	1.06)
		(1.824	0.88)
		(2.695	0.69)
St. James Bay	40	0.52	
Brazil	Araxa	200	3.0
	Tapira	large	-
Tanzania	Panda Hill	(125	0.3
		( 3.8	0.79
Kenya	Mrima Hill	49 including 4½ at 1.25)	0.7
Uganda	Sukulu	200	0.20
Norway	Søve	11	0.35
Nigeria	Kaffo Valley	140	0.26
Congo	Lueshe	30	1.34
U.S.	Powderhorn	40	0.25

TABLE C - Grades of Pyrochlore Concentrate sold by St. Lawrence Colum-  
bium and Metals

	Type SA-64	Type SB-64	Type SD-64
Nb <sub>2</sub> O <sub>5</sub>	50% min	50-56%	52-58%
Ta <sub>2</sub> O <sub>5</sub>	0.1-1.0	0.1-1.0	0.1-1.0
SiO <sub>2</sub> max	4.0	4.0	4.0
SnO <sub>2</sub> max	0.02	0.02	0.02
TiO <sub>2</sub>	4.0-8.0	4.0-7.0	4.0-6.0
P max	0.25	0.06	0.06
S max	0.4	0.1	0.1

TABLE D - Imports of Niobium Concentrates, leading consumers (long tons)

	1955	1956	1957	1958	1959	1960	1961	1962	1963	1964	1965	1966
U.S. (Nb concen- trates)	4,291	2,544	1,495	1,141	1,516	2,255	1,240	2,255	2,638	2,054	2,184	4,142
U.K. (Nb Ta concen- trates)	268	744	438	386	369	618	576	485	745	1,444	1,635	1,558
W. Germany (Nb Ta concen- trates)	144	262	192	160	499	1,209	<u>b/</u> 650	<u>b/</u> 350	<u>b/</u> 650	<u>b/</u> 1,400	1,440	1,383
France (Nb Ta concen- trates)	n.a.	64	24	43	127	155	145	88	71	148	136	422
Japan (Nb concen- trate)	n.a.	3	77	6	3	86	55	11	76	300	279	553

b/ Estimated



TABLE E

Approximate decreasing order of merit of materials suitable for use in nuclear reactors

	1	2	3	4	5	6	7	8
Melting Point	W	Ta	Mo	Nb	V	Zr	ss	Mg
Thermal Neutron Capture Cross Section	Mg	Zr	Al	Nb	Mo.ss	V	W	-
(a) Fast Neutron Capture Cross Section	ss	V	Zr	Mo	Nb	Ta	-	-
Thermal Conductivity in Range 1200-1500°F	W	Mo	NbTa	V	ss	Zr	-	-
(b) Strength level at 2500°F	W	Mo	Ta	Nb	Zr.V	-	-	-

(a) Averaged over a broad section of fast neutron energy spectrum.

(b) Mo and W as wrought; Nb wrought and stress relieved, Ta recrystallised.

ss = stainless steel

TABLE F  
Comparison of Metals - Refractory and Otherwise

	W	Mo	Ta	Nb	Be	*Ti-6Al-4V
Yield Strength (Room t. KSI)	200	90	60	50	35-75	135
Yield Strength (1000°F KSI)	100	55	25	48	20-30	60
Melting Point °F	6170	4730	5425	4475	2345	
Density 16/cu.in.	0.70	0.37	0.60	0.31	0.066	0.16
Mod. of elasticity (p.s.i. x 10 <sup>6</sup> )	50	3.6	47	15	42	16
Price-strength index (room temp.)	2.45	2.00	65	27.9	18.8-8.80	0.38
Price/strength index (1000°F)	4.90	2.95	150	29.1	33-22	0.85
Weight/strength (room temp.)	$2.5 \times 10^{-3}$	$4.1 \times 10^{-3}$	$10.0 \times 10^{-3}$	$6.2 \times 10^{-3}$	$(1.89-0.88) \times 10^{-3}$	$1.19 \times 10^{-3}$
W/strength (1000°F)	$7 \times 10^{-3}$	$6.7 \times 10^{-3}$	$24.0 \times 10^{-3}$	$6.5 \times 10^{-3}$	$(3.3-2.2) \times 10^{-3}$	$2.67 \times 10^{-3}$

\*Note:- The alloy Ti-6Al-4V is that which is most widely used as a structural material in fast aircraft.