CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

MINES BRANCH

COLUMBIUM (NIOBIUM) AND TANTALUM

by

R. J. JONES

MINERAL RESOURCES DIVISION

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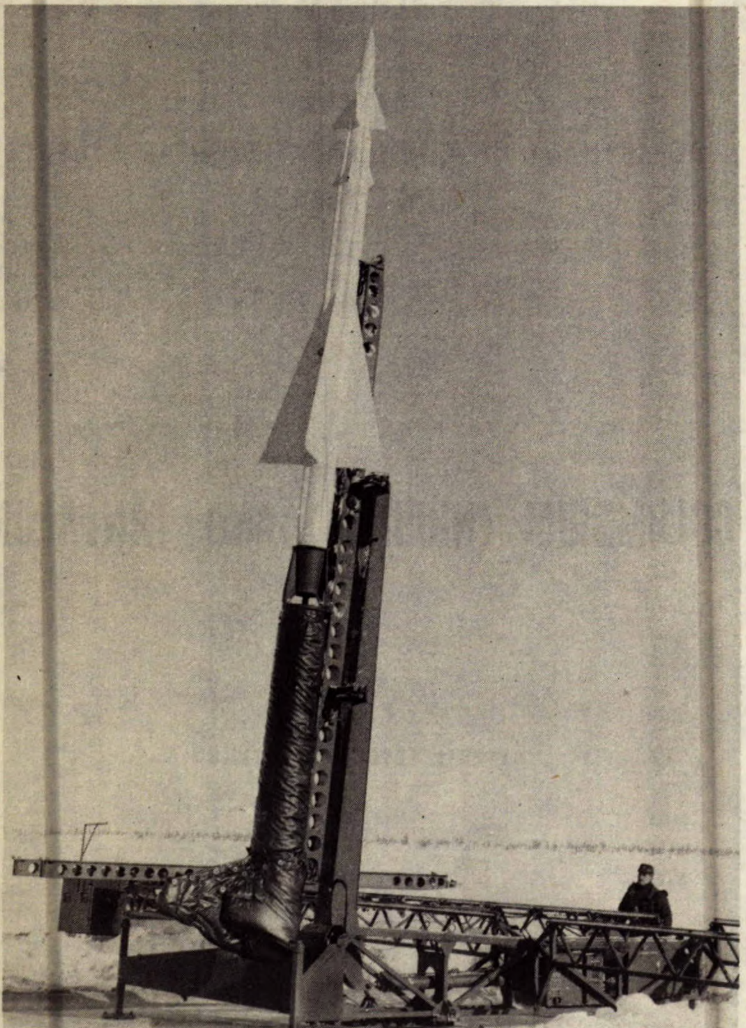

Figure 1 - A guided missile in launching position in northern Canada (Courtesy Dept. National Defence).

PREFACE

Columbium and tantalum have been known for more than a century and a half but only during the last twenty five years have they commenced to take their proper place in the metallurgical field. The advent of turbo-jet engines, guided missiles, rockets, and nuclear energy is hastening this development.

The search for uranium in Canada has brought about the discovery of large reserves of the columbium mineral pyrochlore. However, as with most new minerals, a considerable amount of joint research by both industry and government will be necessary before commercial production can be expected.

The Mines Branch is participating in such a program, and it looks forward with confidence to the eventual solution of the extractive and process metallurgy problems currently hampering the development of Canadian resources of columbium and tantalum. The Mines Branch research program includes mineralogical studies, liquid-liquid and acid extraction, chlorination, and smelting for the recovery of ferrocolumbium from low-grade pyrochlore ores.

John Convey,
Director, Mines Branch.
April 1957.

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COLUMBIUM (NIOBIUM) AND TANTALUM

by

R. J. Jones

CHAPTER I

GENERAL HISTORY

Charles Hatchett, the English chemist, in 1801 analyzed a specimen of rock which had been sent to the British Museum from Connecticut, U.S.A., and found that it contained a new element which he named columbium, after Columbia (America).

It is only natural that since columbium had been discovered in 1801 that its sister element should be found in short order, and in 1802 Anders Gustaf Ekeberg, the Swedish chemist, discovered tantalum in a sample of columbite. In 1809, Wollaston attempted to show that columbium and tantalum were actually the same element, but in 1844 the German chemist Heinrich Rose proved beyond doubt that columbium (which he called niobium from Niobe, daughter of Tantalus) and tantalum were different elements. Berzelieus in 1824 obtained a relatively impure form of tantalum metal, but it was not until 1903 that a ductile form of the metal was produced by a Pole, Werner von Bolton, in Germany.

Columbium metal was first prepared by Blomstrand who, in 1866, reduced columbium chloride with hydrogen, followed later by Moissan who reduced the oxide in an electric furnace with carbon and by Goldschmidt who reduced the oxide with aluminum powder.

The first use of von Bolton's small tantalum metal buttons was in the manufacture of fine wire which was used by Siemens & Halske, A.G. in its plant at Charlottenberg, Germany. Tantalum wire became the first metallic filament material to replace carbon in incandescent lamps, but tungsten soon took its place. Bolton also produced columbium powder in 1906.

In 1922, Dr. Clarence W. Balke of Fansteel Products Corporation of North Chicago, Illinois, perfected a process whereby tantalum bars up to 10 pounds could be made from pressed powder. During 1923, Fansteel placed on the market a charger for storage batteries and the Balkite charger in which tantalum was used as an electronic valve (or rectifier) and eliminated "B" batteries.

Siemens and Halske, A.G., which had patented a process for making pen nibs out of tantalum, also patented in 1927 an alloy of tantalum and nickel for making instruments and parts of chemical apparatus resistant to chemicals and to oxidation.

P.M. McKenna in 1921 received patents on an alloy steel adopted for high-speed tools and having a content of 16 to 20 per cent tungsten, 2.5 to 5 per cent chromium and 1 to 3 per cent tantalum and columbium.

Balke exhibited the first samples of columbium rod and sheet before the American Chemical Society in May 1929.

In 1930, Fansteel Products Corporation placed on the market a new hard cutting material under the trade name of Ramet which consisted of tantalum carbide with nickel used as a binder.

At the close of the 1920's, only Fansteel and Siemens & Halske, A.G. were producing columbium and tantalum metal from the ore, however Blackwell's Limited in Liverpool, England, commenced the production of ferro-tantalum-columbium at the beginning of the decade. Tantalum was being used extensively as a replacement for platinum in rayon spinnerets and various kinds of chemical-plant vessels and equipment even though the price of platinum was below $35 an ounce.

In 1933, Dr. F.M. Becket and associates in the Electro Metallurgical Company's (Union Carbide and Carbon Corporation) research laboratories made two important discoveries relative to the effects of columbium in plain chromium and chromium-nickel stainless steels. They found that when columbium was added to these steels in amounts of up to ten times the carbon content, it kept the high-chromium steels ductile without the loss of its corrosion resistance and resistance to oxidation at elevated temperature. The introduction of columbium to steels of the 18 per cent chromium—8 per cent nickel type prevented intergranular corrosion when the steel was exposed to high temperatures and chemical corrosion, and also prevented failure in weld and adjacent zones. The columbium in alloy form, i.e. ferrocolumbium, was added through the slag to a deoxidized steel bath just prior to pouring of the ingot. This development resulted in the Electro Metallurgical Company commencing to produce ferro-columbium at its Niagara Falls, New York, electric furnace plant, and the first major end-use for columbium.

Until this development, world production of tantalite ore was obtained mainly from alluvial material in the Pilbarra field of Australia, and producers were not paid for the columbium content of the tantalite ores. In 1936, forty tons of ferrocolumbium was sold in the United States from columbite ores mined in Nigeria. In Europe, the steel makers were using ferrotantalum-columbium.

In 1933 the Vascaloy-Ramet Corporation was formed to consolidate the tantalum-carbide manufacturing divisions of Fansteel Products Corporation and Vanadium Alloys Steel Corporation, and the name of the Fansteel corporation was changed to Fansteel Metallurgical Corporation.

In 1932, P.M. McKenna took out patents on a new carbide cutting material which combined tungsten and/or molybdenum with tantalum carbide.

Nigeria was the main supplier of columbite ores, production of which commenced in 1933 from tin gravels. Production of columbite-tantalite concentrates was also commenced in the Belgian Congo in 1934, the tantalum content being higher than that of Nigeria but lower than that of western Australia. The main producer in the Belgian Congo was the Compagnie Geologique at Minière des Ingenieurs et Industries Belges (Geomines) which discovered the mineral in the Manono tin deposits. In 1936, Geomines concluded an agreement with Fansteel whereby the latter corporation would treat the Congo concentrates and develop new uses for tantalum. In 1938, Geomines installed an electric furnace at Manono to experiment on ferrotantalum alloys. It also made some shipments to Belgium where the concentrates were converted to ferrotantalum-columbium by the Société Generale Metallurigque de Hoboken, near Antwerp.

From about 1935, the European steelmakers began to replace tantalum in stainless steels by columbium because it was found to be more effective, in greater supply, and less expensive.

In 1937, the Fansteel Metallurgical Corporation began mining a tantalite-lithium deposit in the Black Hills, near Tinton, South Dakota, and high-grade tantalite production was first reported from Brazil as a by-product of beryl and mica mining.

The great affinity of tantalum for all common gases led to its extensive use in the radio-tube industry, and with the advent of radar during World War II, the demand for tantalum for use as a "getter" increased. In 1940, Fansteel expanded its facilities at both North Chicago and at its subsidiary, Vascaloy-Ramet Corporation at Jersey City to take care of expanded demand, and in 1942 the Reconstruction Finance Corporation of the United States Government erected a government-owned plant next to that of Fansteel called Tantalum Defence Corporation to produce tantalum sheet, wire and fabricated electronic tube parts for the armed forces. The General Electric Company installed a subsidiary in Australia, Hard Metals Pty. Ltd. at Sydney, in order to process Australian tantalite for the manufacture of tool tips.

The collapse of European markets as a result of German occupation in 1940 meant that the Belgian Congo, Nigeria, Brazil and other African production was all diverted to the United States, with some material going to the United Kingdom. As a result of U-boat activity in 1942, arrangements were made whereby the United States Air Force transported tantalite-columbite concentrates from Africa and Australia. In 1943, about 237 tons was moved by air.

Murex Limited at Rainham, Essex, commenced production of ferro-columbium-tantalum and its sintered carbides.

Columbium and tantalum ores were placed under import control by the War Production Board in April 1942 in order that all available supplies would be directed toward the war effort.

The Metals Reserve Company of the United States Government commenced purchasing tantalite during 1942 at the following prices:

Per Pound Contained Ta_2O_5 $	% Ta_2O_5 Concentrate
1.25	40
1.75	50
2.15	60
2.60	70

In May 1943, the Metals Reserve Company began to purchase columbite containing a minimum of 50 per cent Cb_2O_5 and a maximum of 7.5 per cent TiO_2 and 5 per cent SnO_2 at 25 cents per pound of material, no payment being made for the tantalum content. It also raised its prices for tantalite ore containing not more than 3 per cent TiO_2 or 3 per cent SnO_2 to the following schedule:

Per Pound Contained Ta_2O_5 $	% Ta_2O_5 Concentrate
1.75	40
2.25	50
2.75	60
3.25	70

At the end of 1945, the Metals Reserve Company stopped buying tantalite and columbite and disposed of its stock of columbite on the market and transferred the tantalite to the Strategic Stockpile. The War Production Board control over importation and consumption was discontinued at the end of 1944.

The demand for tantalite dropped at the end of the war, but the demand for columbite continued at a high level owing to the development of the gas-turbine engine coupled with the fact that the tin gravel tailings reserve had been depleted during World War II.

At the end of the war, the consumers of columbite were offering 45 cents, 50 cents, and 55 cents per pound of contained Cb_2O_5 in a concentrate with a columbium-tantalum ratio of 2:1, 4:1, and 10:1, respectively. In 1948, the United States Government was actively purchasing columbite on the open market for the Strategic Stockpile and the price increased accordingly. The Metal Bulletin, London, noted that the price per unit of 50 per cent (Ta_2O_5 plus Cb_2O_5) concentrate increased in 1948 from 65 to 75 shillings, in 1949 from 75 to 115 shillings and in 1950 from 115 to 260 shillings.

Late in 1950, the National Production Authority of the United States Government placed columbium and tantalum under allocation and limited the amount of columbium that could be used in stainless steel. Schedule 5 of U.S. Control Order M-80 restricted the use of ferrocolumbium and ferrotantalum-columbium to steels going to direct defence, atomic energy and aircraft engines except for welding rods for low-carbon stainless steels stabilized with columbium, tantalum or titanium, or for non-ferrous nickel alloys and steels for power generating and chemical process industries which operate at continuous temperatures of 800-1,600°F. where other steels were not suitable. This restriction was kept in effect until June 30, 1953.

In May 1952, the Defence Materials Procurement Agency of the United States Government doubled the existing world price for columbite-tantalite ores until they had received 15,000,000 pounds of Cb_2O_5 plus Ta_2O_5. The price was an average of about $3.40 per pound of contained combined pentoxides in a 50 per cent concentrate. This program had two effects, it stimulated the prospecting and recovery of columbium ores and caused consumers to seek substitutes as the price of ferrocolumbium rose to $12.00 a pound of contained columbium. In May 1955, purchasing was terminated when it was announced that deliveries to the stockpile plus forward commitments equalled the objective. The immediate effect of this announcement was confusion on the part of both producers and consumers which resulted in an unstable market. Another important outcome of this premium price purchase program was the three-fold increase in world mine production, most of it as a by-product. Production may drop in certain localities owing to present lower prices, but the level of production should remain well ahead of that existing before 1952. It remains for research and development on the part of producers and consumers to lead columbium into its proper niche in the metallurgical field.

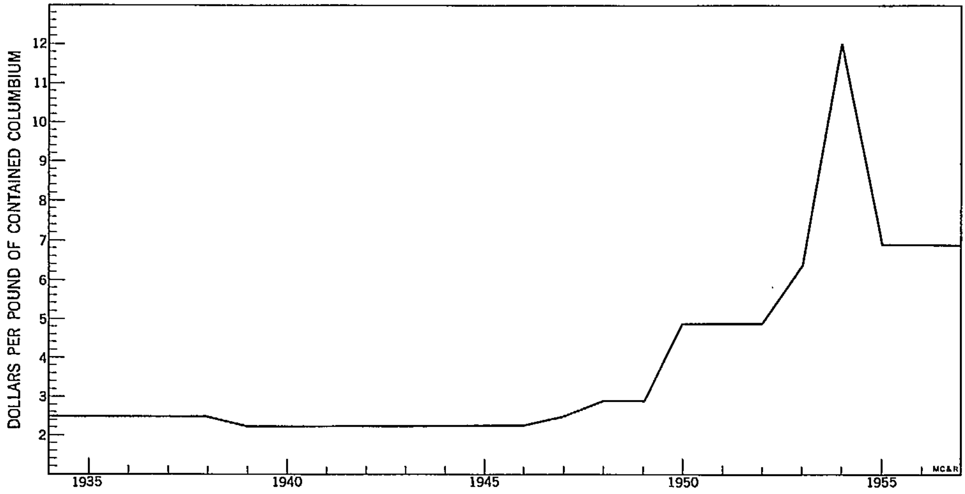


Figure 2 - Price of ferrocolumbium, 1934-1957.

In January 1956, Fansteel announced a $1,000,000 expansion at its North Chicago plant, and in July announced plans for a plant at Muskogee, Oklahoma, which will employ the U.S. Bureau of Mines liquid-liquid process developed at Albany, Oregon. Kawecki Chemical Company and the Kennametal Company commenced the production of pure tantalum and columbium metal in 1956. The Molybdenum Corporation of America became the second United States columbium and tantalum ferro-alloy producer during the year.

CHAPTER II

COLUMBIUM AND TANTALUM AND THEIR MINERALS

	Columbium	Tantalum
Atomic number	41	73
Atomic weight	92.91	180.9
Specific gravity	8.4	16.6
Melting point	2,500°C.	2,996°C.
Boiling point	3,300°C.	+4,100°C.
Colour	steel-grey	bluish-grey
Type of crystal lattice	Body Centered Cube	Body Centered Cube
Linear coefficient of expansion/°C	7.1×10^{-6}	6.5×10^{-6}
Electrical resistivity at 20°C., microhms/cm	13.1	15

Columbium is a malleable and ductile metal and is extremely resistant to corrosion and most types of chemical attack. It is completely soluble in sulphuric acid.

Tantalum is very ductile and malleable under special treatment and highly resistant to almost all forms of corrosion -- in fact it is similar to glass and is attacked by hydrofluoric acid. Tantalum has a higher melting point than all other metals except tungsten and rhenium. Tantalum and columbium absorb hydrogen when heated and also combine with other gases as oxygen and nitrogen. Both metals will not oxidize at room temperatures but when heated in air become coated with oxide.

Columbite and Tantalite

The present important commercial minerals of columbium and tantalum are columbite and tantalite which are respectively, a columbate and tantalate of iron and manganese. Pure columbite, $Fe\ Cb_2O_6$, contains 82.7 per cent Cb_2O_5, and pure tantalite, $Fe\ Ta_2O_6$, contains 86.1 per cent Ta_2O_5. Columbium pentoxide and tantalum pentoxide contain respectively, 69.99 per cent columbium and 81.93 per cent tantalum.

Tantalite and columbite occur in granite pegmatites and in residual or alluvial deposits derived from such rock. Being closely related minerals and difficult to distinguish from one another, they require precise chemical analysis for their differentiation, although specific gravity methods can be successfully used. High-grade tantalite usually occurs in the form of stout, more nearly equidimensional crystals than those of columbite, has a compact texture, with conchoidal fracture and high lustre, is purple-black in colour, and has a higher specific gravity than columbite.

Crystals of columbite generally have a thinner, bladed or platy form, and often occur in radiated groups. The mineral has a steel-grey colour, lower lustre, granular texture and uneven fracture. However since the minerals grade from the pure extremes, the above visual characteristics are at best only generally indicative of composition. Tantalite is produced chiefly in Australia, Brazil and the Belgian Congo while the important source of columbite is Nigeria.

Pyrochlore

This mineral occurs in alkali rocks and is the source of columbium production from Sove, near Ulefoss in Norway, and from Freiberg, Germany. It is a complex columbate of cerium, calcium, sodium with some titanium, thorium, fluorine and a little tantalum. The Cb_2O_5 content ranges to about 73 per cent. The mineral has a hardness of 5 - 5.5 and a specific gravity of about 4.3 and generally has a brown colour. In Canada, there is generally some radioactivity associated with pyrochlore deposits, and this fact has been responsible for large discoveries in recent years such as in British Columbia, Ontario and Quebec. Extraction of columbium from these deposits still presents metallurgical difficulties owing to the extreme fineness of the grains and a specific gravity not very different from those of some of the associated minerals.

Other minerals of the pyrochlore group are koppite, hatchettolite, microlite, betafite and perovskite.

Fergusonite

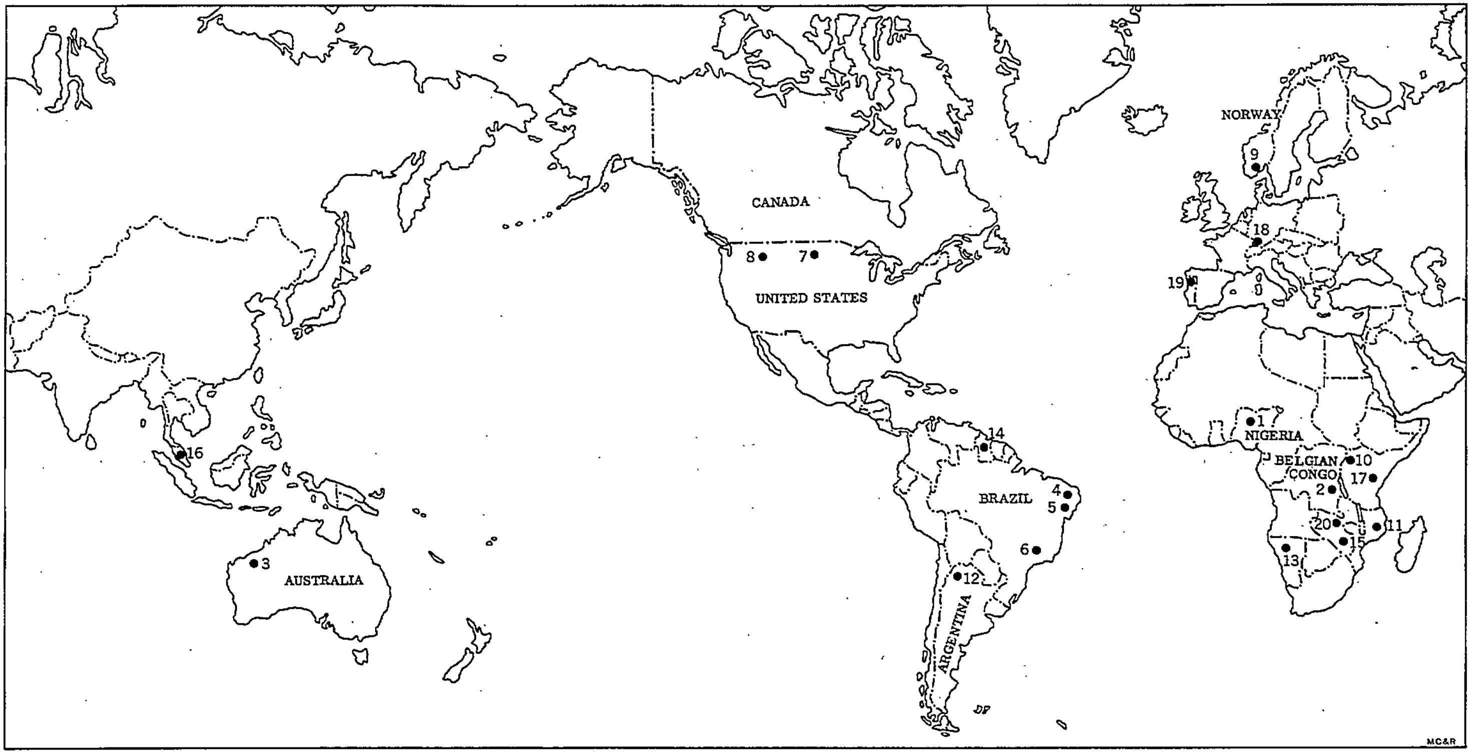
This mineral is a tantalate and columbate of yttrium, erbium, and cerium.

Samarskite

This mineral is a complex tantalate and columbate of iron, yttrium, calcium and cerium.

Euxenite

This mineral group comprises tantalates and columbates of yttrium, erbium, cerium and uranium. Other minerals of the group are polycrase, eschyrite and lyndochite.



1. JOS PLATEAU
2. MANONO
3. PILBARRA
4. RIO GRANDE DE NORTE
5. PARAIBA
6. MINAS GERAES
7. BLACK HILLS, SOUTH DAKOTA
8. BEAR VALLEY, IDAHO
9. ULEFOSS, NORWAY
10. UGANDA DEPOSITS
11. MOZAMBIQUE DEPOSITS
12. ARGENTINA DEPOSITS
13. SOUTHWEST AFRICA DEPOSITS
14. BRITISH GUIANA DEPOSITS
15. BIKITA, SOUTHERN RHODESIA
16. MALAYA DEPOSITS
17. KENYA DEPOSITS
18. KAISERSTULL, GERMANY
19. CAMINHA, PORTUGAL
20. NORTHERN RHODESIAN DEPOSITS

Figure 3 - Key Map of World Mine Production of Columbium and Tantalum

CHAPTER III

WORLD MINE PRODUCTION

Production of columbium and tantalum ores has, up to the present, been derived from tin-bearing gravels and eluvial and alluvial pegmatites mainly from Africa, Brazil and Australia.

Production from Canada has been negligible, coming from pegmatite dykes in the Northwest Territories. Any concentration of columbite-tantalite which may have existed from the weathering of these and other dykes was removed during the glaciation period.

However, the intense search for uranium ores in Canada has led to the discovery of large radioactive pyrochlore deposits in the provinces of Quebec and Ontario. The successful exploitation of these deposits is dependent upon an economic extraction process and the market for the metals at such time. However the strategic location of the deposits compared with present sources of supply will no doubt be of considerable significance in the future.

Nigeria

Nigeria is by far the world's most important source of columbite. Production of tantalite is negligible. The mineral is widely distributed in the alluvial tin deposits in northern Nigeria, and most of the mineral is mined as a co-product of the tin mining industry. Columbite occurs as a widely disseminated primary accessory mineral in the weathered biotite granites. At Kuru, in an area mined by Jantar Nigeria Company Limited, the columbite-tin ratio is about 1:25. In the Liruei Hills, Kaou province, the columbite-tin ratio is high at about 1:3.

The bonus price offered by the United States Government in 1952 resulted in a very substantial increase in production from newly mined gravels and from old tailings and dumps and in intensive exploration for new deposits.

TABLE I

World Production of Columbium and Tantalum Mineral Concentrates by Countries

(Pounds)

<table>
<tr><th rowspan="2">Country 1/</th><th colspan="2">1946-50 (average)</th><th colspan="2">1951</th><th colspan="2">1952</th><th colspan="2">1953</th><th colspan="2">1954</th><th colspan="2">1955</th></tr>
<tr><th>Columbium</th><th>Tantalum</th><th>Columbium</th><th>Tantalum</th><th>Columbium</th><th>Tantalum</th><th>Columbium</th><th>Tantalum</th><th>Columbium</th><th>Tantalum</th><th>Columbium</th><th>Tantalum</th></tr>
<tr><td>Argentina</td><td colspan="2">485</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td>2/11,023</td><td>---</td><td>2/10,800</td><td>2/6,614</td></tr>
<tr><td>Australia</td><td>---</td><td>6,856</td><td colspan="2">5,125</td><td colspan="2">16,108</td><td colspan="2">18,124</td><td colspan="2">117,767</td><td colspan="2">3/125,000</td></tr>
<tr><td>Belgian Congo (incl. Ruanda-Urundi) 4/</td><td colspan="2">317,903</td><td colspan="2">209,437</td><td colspan="2">231,042</td><td colspan="2">623,902</td><td colspan="2">967,819</td><td colspan="2">947,978</td></tr>
<tr><td>Bolivia (exports)</td><td>1,367</td><td>---</td><td>1,043</td><td>---</td><td>---</td><td>---</td><td>3,366</td><td>---</td><td>---</td><td>---</td><td>2,350</td><td>---</td></tr>
<tr><td>Brazil</td><td>5/17,316</td><td>5/57,765</td><td>5/11,200</td><td>5/8,960</td><td>5/4,480</td><td>5/53,760</td><td>5/67,200</td><td>5/40,320</td><td>2/124,460</td><td>2/255,533</td><td>2/233,012</td><td>2/221,834</td></tr>
<tr><td>British Guiana</td><td>---</td><td>---</td><td>---</td><td>---</td><td>2,000</td><td>---</td><td>11,200</td><td>---</td><td>4,480</td><td>---</td><td>6,720</td><td>---</td></tr>
<tr><td>Canada</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td>90</td><td>77</td><td>42</td><td>390</td></tr>
<tr><td>French Equatorial Africa</td><td colspan="2">4,460</td><td colspan="2">---</td><td colspan="2">3,527</td><td colspan="2">3,514</td><td colspan="2">6,261</td><td colspan="2">2,672</td></tr>
<tr><td>French Guiana</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">13,228</td><td colspan="2">28,250</td><td>---</td><td>2/23,085</td></tr>
<tr><td>Germany, West</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td>2/267,957</td><td>2/62,865</td><td>2/849,310</td><td>2/594,030</td></tr>
<tr><td>Madagascar</td><td colspan="2">---</td><td>8,598</td><td>---</td><td>5,732</td><td>---</td><td>8,377</td><td>---</td><td>36,596</td><td>---</td><td>38,801</td><td>---</td></tr>
<tr><td>Malaya</td><td>3,584</td><td>---</td><td>56,000</td><td>---</td><td>105,280</td><td>---</td><td>116,480</td><td>---</td><td>248,640</td><td>---</td><td>52,910</td><td>---</td></tr>
<tr><td>Mozambique</td><td colspan="2">1,738</td><td colspan="2">6/11,257</td><td colspan="2">32,652</td><td colspan="2">6/58,133</td><td colspan="2">94,031</td><td colspan="2">82,884</td></tr>
<tr><td>Nigeria</td><td>2,546,432</td><td>5,233</td><td>2,419,200</td><td>6,720</td><td>2,896,320</td><td>2,240</td><td>4,388,160</td><td>---</td><td>6,527,360</td><td>22,400</td><td>7,047,040</td><td>35,840</td></tr>
<tr><td>Norway</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td>2/40,367</td><td>---</td><td>392,419</td><td>---</td><td>675,930</td><td>---</td></tr>
<tr><td>Portugal</td><td>---</td><td>5/7/3,009</td><td>---</td><td>5/4,526</td><td>---</td><td>2/35,428</td><td>2/68,121</td><td>2/154,323</td><td>2/148,732</td><td>2/86,279</td><td>2/168,362</td><td>2/6,614</td></tr>
<tr><td>Rhodesia and Nyasaland, Fed. of:</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td>Northern Rhodesia</td><td colspan="2">---</td><td colspan="2">---</td><td>---</td><td></td><td colspan="2">---</td><td>---</td><td>1,252</td><td>---</td><td>---</td></tr>
<tr><td>Southern Rhodesia</td><td>---</td><td>14,572</td><td>---</td><td>---</td><td>1,120</td><td>10,360</td><td>5,100</td><td>27,060</td><td>18,060</td><td>14,300</td><td>12,240</td><td>4,660</td></tr>
<tr><td>Sierra Leone</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td>8,960</td><td>---</td><td>8,960</td><td>---</td></tr>
<tr><td>South West Africa</td><td colspan="2">3,689</td><td colspan="2">3,974</td><td colspan="2">4,400</td><td colspan="2">17,634</td><td>22,439</td><td>3,868</td><td>8,299</td><td>2,924</td></tr>
<tr><td>Spain 2/</td><td colspan="2">---</td><td colspan="2">---</td><td>---</td><td>741</td><td>4,410</td><td>---</td><td colspan="2">---</td><td>2,525</td><td>11,276</td></tr>
<tr><td>Sweden 2/</td><td colspan="2">---</td><td colspan="2">---</td><td colspan="2">---</td><td>16,713</td><td>4,242</td><td>---</td><td>19,251</td><td>---</td><td>---</td></tr>
<tr><td>Uganda 8/</td><td colspan="2">5/5,390</td><td colspan="2">5/42,560</td><td colspan="2">5/9,094</td><td colspan="2">23,542</td><td colspan="2">23,117</td><td colspan="2">34,003</td></tr>
<tr><td>Union of South Africa</td><td>---</td><td>1,600</td><td>---</td><td>6,000</td><td>---</td><td>8,000</td><td>---</td><td>38,000</td><td>---</td><td>46,000</td><td>---</td><td>22,000</td></tr>
<tr><td>United States (mine shipments)</td><td colspan="2">1,871</td><td colspan="2">925</td><td colspan="2">5,385</td><td colspan="2">14,867</td><td colspan="2">32,829</td><td colspan="2">12,440</td></tr>
<tr><td>World total (estimate)</td><td colspan="2">2,990,000</td><td colspan="2">2,800,000</td><td colspan="2">3,430,000</td><td colspan="2">5,770,000</td><td colspan="2">9,590,000</td><td colspan="2">11,250,000</td></tr>
</table>

1/ Frequently the composition (Cb_2O_5 - Ta_2O_5) of these mineral concentrates lies in an intermediate position, neither Cb_2O_5 nor Ta_2O_5 being strongly predominant. In such cases the production figure is centered.
2/ United States imports.
3/ Estimate.
4/ In addition, tin-columbium-tantalum concentrates were produced as follows: 1945-50 (average), 1,224,876 pounds; 1951, 2,597,019 pounds; 1952, 2,813,070 pounds; 1953, 3,575,861 pounds; 1954, 5,970,057 pounds; 1955, 3,941,825 pounds; columbium-tantalum content averaging about 10 per cent.
5/ Exports.
6/ In addition to figure shown, 176 pounds of samarskite were produced in 1951, and 132 in 1953.
7/ Average for one year only, as 1950 was first year of commercial production.
8/ In addition, tin-columbium-tantalum concentrates were produced as follows: 1950, 1,210 pounds; 1951, 336 pounds; 1952, 3,248 pounds; 1953, 4,480 pounds; 1954, 6,720 pounds.

(Source: U.S. Bureau of Mines, Washington, D.C.)

There are many companies and individuals operating on the cassiterite-columbite deposits which together produced 3,152 long tons of columbite concentrates in the year ended March 31, 1955. The main producers with their production were as follows: Amalgamated Tin Mines of Nigeria Limited, 616 tons; Keffi Tin Company Limited, 433 tons; Bisichi Tin Company (N) Limited, 339 tons; Tin & Associated Minerals Limited, 293 tons; and Jantar Nigeria Company Limited, 227 tons. Kennecott Copper Corporation in 1956 purchased a controlling interest in Tin and Associated Minerals Limited. The first commercial production was recorded in 1933 and the following table showing both columbite and cassiterite production illustrates well the increase since the beginning of World War II.

TABLE II

Production of Cassiterite and Columbite in Nigeria, 1939-1955

Year	Cassiterite	Columbite
	Long Tons	Long Tons
1939	13,003	431
1940	16,568	396
1941	16,638	402
1942	17,107	865
1943	17,463	802
1944	17,258	2,055
1945	15,482	1,571
1946	14,252	1,550
1947	12,597	1,286
1948	12,740	1,096
1949 & 1st Qtr. 1950	15,383	1,144
1950-1951	11,333	899
1951-1952	11,596	1,115
1952-1953	11,758	1,406
1953-1954	10,910	2,100
1954-1955	11,026	3,152

Estimated reserves of columbite in 1955 were 62,500 tons. Pyrochlore has been found in the Kaffo Valley granites where inferred reserves of pyrochlore-bearing granites are of the order of several hundred million tons. The Cb_2O_5 content is about 0.26 per cent with some uranium.

Nigerian columbite assays 60 to 68 per cent Cb_2O_5 and 10 to 15 per cent Ta_2O_5 reaching 73 per cent combined oxides. The proportion of TiO_2, FeO, MnO and SnO_2 average around 3.0, 18.0, 2.0, and 2.5 per cent respectively.

Belgian Congo

The Belgian Congo ranks next to Nigeria in the production of columbium and tantalum minerals. Production is derived as a co-product with tin in the sluicing of gravels and also from the Manono tin smelter slags which are shipped to the United States for recovery of the tantalum and columbium.

The largest producer is Geomines which commenced production in 1939. Other important producers are Symetain, Minetain, Somuki, Sermikat and Minière Grands Lacs which operate mines in the Congo and Ruanda-Urundi. Material from the Congo is an intermediate grade of tantalite averaging about 35 per cent Ta_2O_5 and 30 per cent Cb_2O_5. Since 1947, a tin-columbium-tantalum concentrate has also been produced containing about 10 per cent Ta_2O_5 plus Cb_2O_5.

TABLE III

Production of Tantalite-Columbite in Belgian Congo
(Metric Tons)

1940	268	1948	145
1941	208	1949	116
1942	127	1950	135
1943	151	1951	96
1944	294	1952	220
1945	198	1953	283
1946	168	1954	633
1947	157		

Brazil

The world's principal production of high-grade tantalite (60 per cent Ta_2O_5) is derived from the States of Rio Grande de Norte and Paraiba in north-east Brazil and from the State of Minas Geraes in the south.

Production of tantalite and beryl are co-products from pegmatite dykes which have been mined since 1937. The properties are generally worked by primitive methods.

Other promising localities are Ramalhite near Pecanha, Rio Dore Valley and Santana de Suassui, State of Minas Geraes.

Australia

Prior to World War II and the increase in production from Brazil, Australia was the leading producer of high-grade tantalite with a Ta_2O_5 content of 65 per cent and less than 10 per cent Cb_2O_5. Production is mainly obtained from the Wodgina pegmatite dyke about 70 miles south of Port Hedland in western Australia. Other producing areas are Strelley, 40 miles from Port Hedland; Tabba Tabba, 40 miles southeast of Port Hedland; Pilgongoora, 75 miles southeast of Port Hedland; and Greenbushes, 160 miles south of Perth. Production was commenced in 1905 and has been more or less continuous to the present.

TABLE IV

Production of Tantalite in Australia

(Short Tons)

1942	0.85
1943	13.70
1944	12.09
1945	0.52
1946	0.40
1947	0.05
1948	0.01
1949	1.75
1950	7.38
1951	2.56
1952	8.05
1953	9.06
1954	58.88

Norway

Norsk Bergverk in cooperation with Norsk Hydro are operating a pyrochlore deposit (koppite) about 65 miles southwest of Oslo near Ulefoss in Telemark county. The columbium mineral is contained in a limestone with an apatite content of from 6 to 10 per cent. During the last war, I.G. Farbenindustrie carried out diamond drilling and beneficiation research with the object of producing agricultural limestone, and phosphate and columbium alloys. The average columbium pentoxide content of the ore is 0.2 to 0.3 per cent.

A typical analysis of ore from the Cappelen vein, which has a higher columbium content than other deposits in the area, is as follows:

	%
CaO	43.5
MgO	1.2
FeO	1.1
MnO	0.5
CO_2	36.4
3 (3 CaO. P_2O_5) CaF_2	8.5
$Cb_2O_5 + Ta_2O_5$	0.30
Fe_3O_4	5.0
FeS_2	0.5
Silicates, etc.	3.0

Ore reserves of the area are in the neighbourhood of 11,000,000 tons and said to contain 17,000 tons of Cb_2O_5 and 680,000 tons P_2O_5.

Production was commenced in 1953 with the assistance of a United States Government loan, and production of columbium concentrate is presently about 30 tons per month.

Germany

Production of columbium is derived from a metamorphic limestone containing koppite at Kaiserstull near Freiberg in southwest Germany. A company, Niob-Bergbau G.m.b.H., owned by the Government of Baden and by Fabriques de Produits Chemiques de Thann et Mulhouse, S.A., have been mining the deposit since 1953. Part of the output is refined by Fabriques de Produits Chemiques at Thann in France and the remainder is exported to the United States.

Portugal

Production in recent years has been obtained from the retreatment of alluvial tin tailing dumps from Caminha. The tin deposits contain gold in addition to cassiterite and columbite. Columbite also occurs in the wolframite deposits at Viano do Costelo.

Uganda

Production of tantalite-columbite commenced in 1936, mainly from pegmatite dykes west of Lake Victoria and east of Lake Edward associated with tin and gold.

Large quantities of pyrochlore have been discovered in a red soil calcereous complex of the Sukulu plug near Tororo. Average grade of the material is 13.1 per cent P_2O_5 and 0.20 per cent Cb_2O_5. Associated minerals are apatite, baddeleyite, zircon, tremolite, ilmenite and magnetite. Other promising areas are at Nampeyo Hill north of Kampalo, Okollo in West Nile, Buswale in south-east Uganda.

The United States firm of Colin Mathieson Chemical Corporation together with Frobisher Limited and Uganda Development Corporation are exploiting these deposits under the name of Tororo Exploration Company with a view to production in 1958. Production of apatite concentrates in a pilot plant commenced in October 1956.

Northern Rhodesia

Anglo American Corporation are examining the possibilities of extracting pyrochlore from limestone at Nkombwa Hill in the Isoka district.

The Corporation has also discovered another similar deposit in the Feira district in the Central Province about 150 miles from Lusaka.

Mozambique

Production of tantalite is derived from eluvial and alluvial pegmatitic deposits and from a uranium bearing mineral, samarskite, which is mined in the Tete district. Production of samarskite was commenced about 1948 and has been increasing ever since.

United States

The United States is the largest consumer of columbite ores and also one of the smaller producers. It depends upon imports for practically all its requirements.

Columbite production from the Black Hills, South Dakota, and New Mexico has been intermittent over a number of years from pegmatite mines principally as a by-product of mica, feldspar and beryl mining. Fansteel Metallurgical Corporation have operated a tantalite-lithium deposit near Tinton, South Dakota, since 1937.

Large reserves of columbium are known to occur in bauxite deposits and in titanium mineral deposits of the Magnet Cove area, Arkansas. Tests have shown that columbium is concentrated in an ilmenite fraction of the bauxite black sand. The ilmenite fraction contains as high as 0.86 per cent columbium. The red-mud waste from bauxite mining and processing contains a large reserve of columbium. Placer deposits in Bear Valley and Cascade, Idaho, containing around 0.25 pounds of columbium per yard, have been examined by the U.S. Bureau of Mines. The deposit at Bear Valley was producing during 1956 as a result of a purchase contract with General Services Administration for 1,050,000 pounds of $Cb_2O_5 + Ta_2O_5$ over a five-year period.

Argentina

Columbite occurs in pegmatites in the Provinces of Cordoba, Catamarca, La Rioja and Salta near the border with Bolivia. The deposits in Salta are the more important.

Southwest Africa

Tantalite-columbite minerals occur in pegmatitic alluvial deposits in the Erongo Mountains, Karabib-Usakos district and Omaruru district.

British Guiana

Large deposits of columbite exist in the valleys of the Rumong-Rumong and Morabisi Rivers about 100 miles southwest of Georgetown. Columbite occurs in alluvial and eluvial deposits associated with pegmatites. Ilmenorutile occurs in the Rumong-Rumong area together with about 4 1/2 pounds of columbite in a cubic yard of material. Kennametal International S.A. are developing a deposit in the area.

Deposits in the Morabisi River area average about 2 pounds of columbite per yard.

Southern Rhodesia

Production of tantalite is derived from the Bikita area, 100 miles east of Gwelo. Tantalite occurs in rich "spots" in pegmatitic areas.

Production is relatively small and generally amounts to less than 10 tons of concentrate per year.

Malaya

Columbite occurs in the large alluvial tin deposits of Malaya. The columbite content is very small. A few tons were produced by the Japanese during 1942-1945. The large slag dumps of the tin smelters contain recoverable quantities of the mineral.

Kenya

An extensive deposit of columbium mineralization, known as the Mrina Hill deposit, occurs 10 miles from the coast, near the Tanganyika border.

The Geological Department of Kenya has estimated the content at 26,000,000 tons running 0.78 per cent Cb_2O_5 together with monazite and rare earths. The Anglo American Corporation of South Africa Limited is investigating the deposit.

CHAPTER IV

CANADIAN OCCURRENCES

(Reference numbers following sub-headings refer to numbers on Figure 4)

Production of columbium and tantalum minerals in Canada has so far been confined to a very small output following the end of World War II and in the years 1954 and 1955 from the Yellowknife area of the Northwest Territories. Known occurrences of this area are contained in pegmatites which lend themselves to difficult sampling and estimation of reserves. The operations which produced the minerals did not succeed and indicates that the columbite-tantalite content was small in relation to overall costs in this remote area.

British Columbia

Bugaboo Creek (1)

The property of Quebec Metallurgical Industries Limited extends along the head of Bugaboo Creek, about 25 miles by road from Spillimacheen and covers occurrences of post-glacial gravels containing uranian pyrochlore derived from erosion of the Bugaboo granite stocks.

During 1955, the gravel was tested by churn-drilling and, during the summer of 1956, a plant was installed at Bugaboo Creek to mine gravel and to produce a gravity concentrate. These concentrates were shipped to the company's research laboratories near Ottawa and are now being processed to produce high-purity columbium oxide, columbium alloys and columbium sponge.

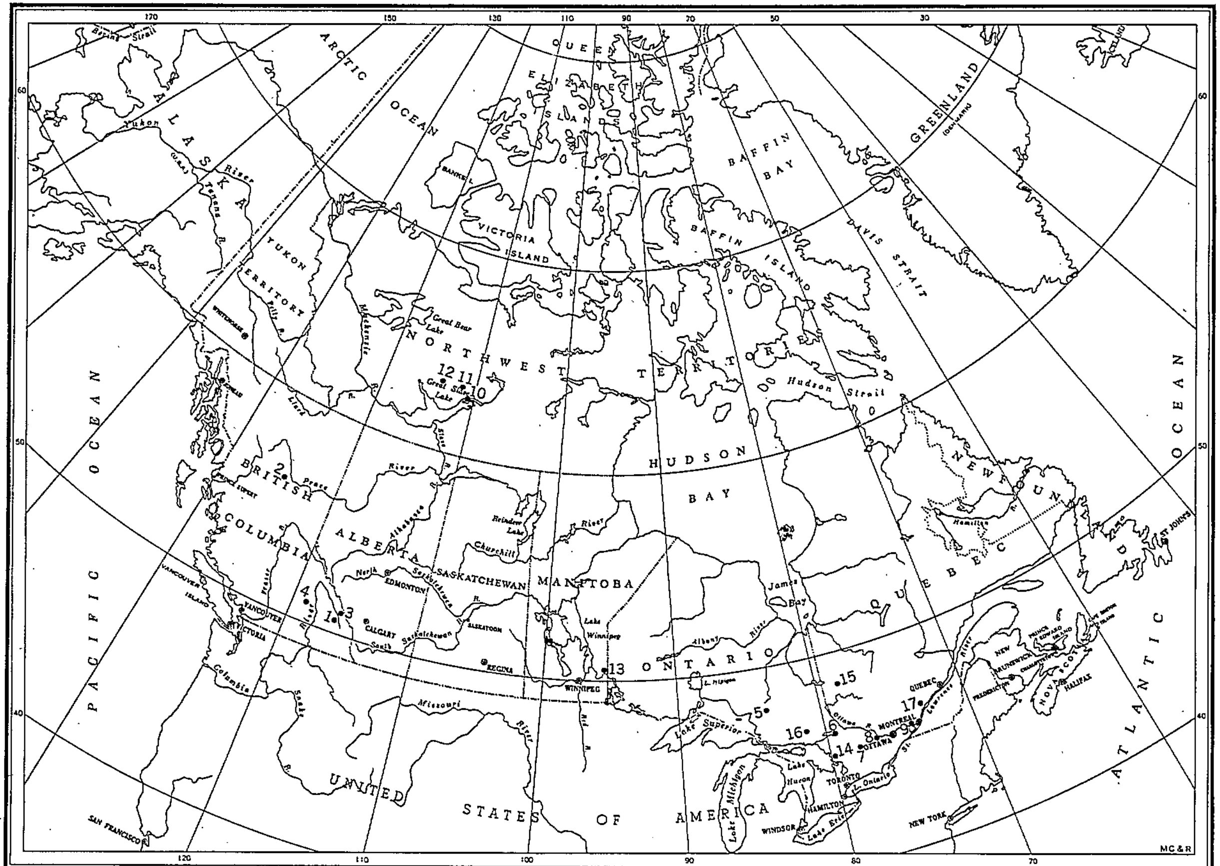


Figure 4 - Key map of Canada showing columbium-tantalum occurrences.

Proven and potential columbium-bearing gravel reserves amount to 65,000,000 cubic yards. Concentration tests show that 0.11 pounds of columbium per cubic yard of gravel can be recovered in a black sand concentrate. This suggests a recoverable potential of 7,150,000 pounds of columbium at the property.

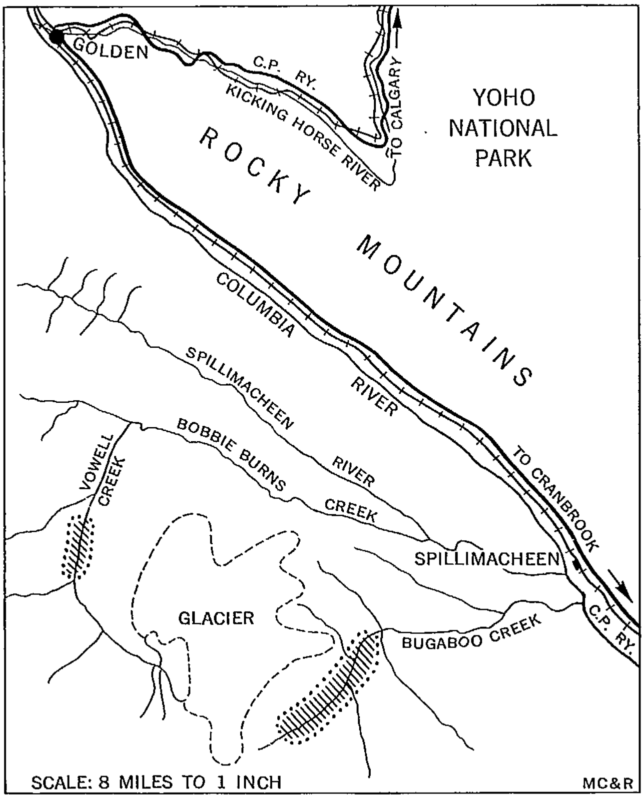


Figure 5 - Index map showing location of Bugaboo gravel deposits.

Granite Creek (2)

Northwestern Exploration, Limited optioned eight claims, called the Lonnie group, which are situated on the southeast side of Granite Creek, a tributary of Manson River and are about 1 1/2 miles from the Fort St. James-Manson Creek road.

The claims were located in 1954. The mineralization consists of columbium and uranium-bearing minerals, ilmenorutile, columbite and uranian pyrochlore in a band of carbonate and alkaline rocks which is bordered by hornblende gneiss. Columbium is present in the minerals ilmenorutile, columbite and pyrochlore.

During 1955, the mineralized zone was explored by trenches and hydraulic stripping. Northwestern Explorations, Limited have since dropped their option on the property.

Moose Creek (3)

Radioactive and columbium-bearing mineralization occurs between Sharp and Helmet Mountains near the head of Moose Creek, a tributary of the Beaverfoot River. The columbium occurs in pegmatites with calcite, biotite, pyroxene, magnetite-ilmenite, and schorlomite, with minor amounts of pyrite, pyrrhotite, nephelite, and other accessories.

Samples taken from different sections of pegmatites ran from 0.016 per cent to 0.08 per cent columbium.

Lempriere (4)

Uranium-bearing pyrochlore was discovered in 1950 in a carbonate rock adjacent to the Canadian National Railways tracks, 4 miles south of Lempriere station. In 1952, St. Eugene Mining Corporation Limited optioned the property and carried out some geological work and prospecting. Uranium-bearing pyrochlore was found with other minerals in crystalline, dolomitized limestone interbedded with gneisses.

It is understood that no ore of any importance was found on these claims.

Manitoba

Lac du Bonnet Mining Division,
Oiseau River Area (13)

Red feldspar dykes about 9 miles above Pointe du Bois contain columbite-tantalite with beryl and some uraninite.

Lac du Bonnet Mining Division, Rush Lake (13)

A bulk sample from the Odd claim indicated 0.23 per cent combined Cb_2O_5 plus Ta_2O_5. The columbite-tantalite is contained in a pegmatite dyke carrying cassiterite.

Ontario

Sudbury Mining District, McNaught and Lackner Townships (5)

Multi-Minerals Limited own a property approximately 7 miles northeast of Nemegos Station on the main line of the Canadian Pacific Railway. The property is about 14 miles from Chapleau.

Two major deposits outlined by diamond drilling contain 50,000,000 tons of material with an average columbium pentoxide content of 0.26 per cent with local concentrations of undetermined extent assaying more than 1 per cent Cb_2O_5. Included in this tonnage is a 2,000,000-ton calcitic body of a type similar to that at Sove, Norway.

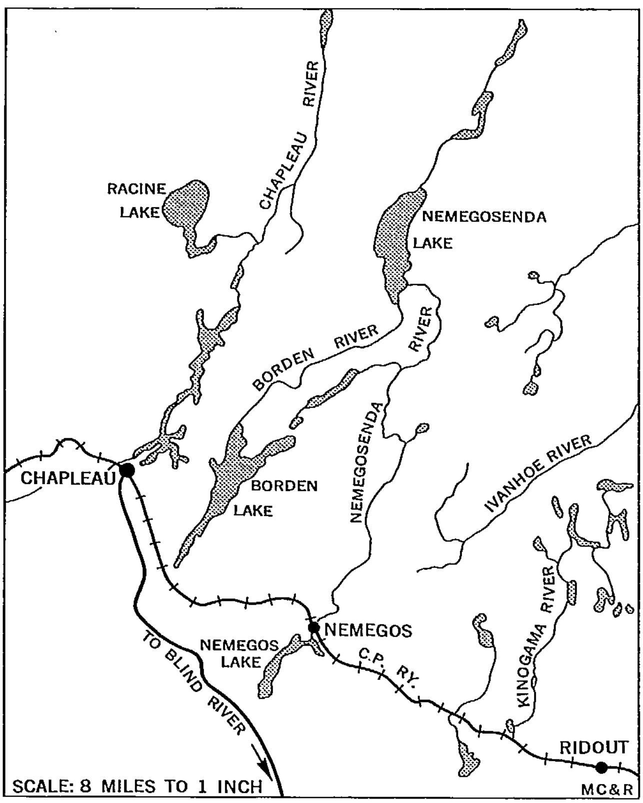


Figure 6 - Index map of Nemegos area.

The columbium-bearing minerals are members of the pyrochlore group associated with magnetite and apatite. The pyrochlore has a resinous lustre and ranges in colour from light yellow to reddish-brown and occurs chiefly as irregular aggregates in feldspar and nepheline. In the calcitic body of material, pyrochlore occurs as octahedral crystals and rounded grains embedded in calcite and other gangue minerals.

Mining and processing of the columbium deposits are contingent on the operation of adjacent magnetite-apatite deposits and on market conditions.

Sudbury Mining District, Chewett Township (5)

Dominion Gulf Company have outlined two areas of columbium mineralization on their property at Nemogosenda Lake, 17 miles northeast of Chapleau.

Extensive diamond drilling on one area has indicated over 20,000,000 tons of material averaging 0.5 per cent columbium pentoxide. In addition there is a substantial tonnage averaging down to 0.3 per cent Cb_2O_5. The columbium mineral is pyrochlore.

The other area has been drilled only sufficiently to identify the presence of ore zones. From this drilling, a possible tonnage of 30,000 tons per vertical foot, or 15,000,000 tons above the 500-foot level, have been indicated.

Field work has been terminated and the company is working on an extraction process.

Nipissing Mining District, Lake Nipissing (6)

The Beaucage Mines Limited property is an area of some 8,100 acres, five miles west of North Bay and including the Manitou Islands with a land area of 328 acres.

Closely spaced surface diamond drilling and underground development have been concentrated in the zone to the east of Newman Island. Tonnage and grade estimates on underground development within this zone, not including any material between the lake bottom and the 300-foot level are as follows:

Tons	Tons/ Vertical Foot	% U_3O_8	% Cb_2O_5
2,695,500	6,990	0.042	0.69
1,824,000	4,560	0.05	0.88
617,000	1,540	0.075	1.06

The columbium mineral is pyrochlore which occurs in a metamorphic rock consisting of gneisses, crystalline limestone and intrusives typical of the Grenville province together with acmite, pyroxene, apatite, pyrrhotite, magnetite, pyrite and marcasite as the principal gangue minerals.

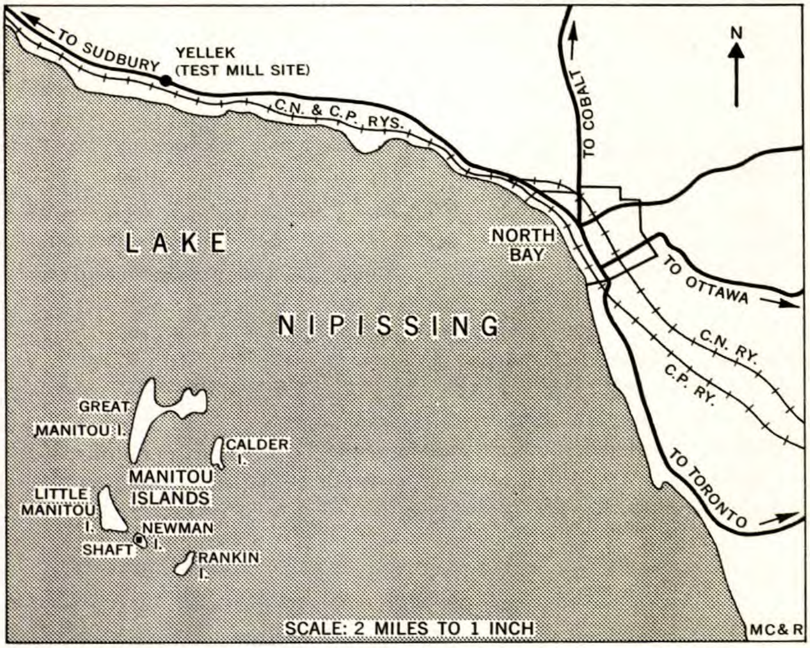


Figure 7 - Index map showing location of Manitou Islands in Lake Nipissing.

A 50-ton test mill has been erected and is being operated at Yellek on the shore of Lake Nipissing, five miles west of the city of North Bay to check the laboratory findings of Battelle Memorial Institute. This process consists of three basic steps (a) Flotation to eliminate pyrite and calcite which are reagent consumers in succeeding stages, (b) Chemical removal of the columbium from the flotation underflow, and (c) Leaching of the residue to remove the uranium.

Parry Sound District, Conger Township (14)

Samarskite occurs with urananite in a feldspar dyke on Lots 9 and 10, Concession IX, near Blackstone Lake.

Sudbury District, Dill Township (16)

Toddite occurs in a feldspar dyke on Lot 4, Concession III, north of Lake Huron.

Hastings County, Faraday Township (7)

Silver Crater Mines Limited has carried out exploration work on a calcite body containing crystals of betafite ranging in size from less than one inch to three inches. The betafite which contains 41.5 per cent Cb_2O_5, 1.4 per cent Ta_2O_5 and 21.4 per cent U_3O_8 is associated with mica, apatite and biotite-scapolite gneiss.

Hastings County, Monteagle Township (7)

The MacDonald and Woodcox mines, Lots 18 and 19, Concession VII near Hybla, have been operated for feldspar. Occuring with the feldspar are cyrtolite and ellsworthite, a radioactive mineral of the pyrochlore group.

Renfrew County, Lyndoch Township (8)

Euxenite minerals occur with beryl on Lot 23, Concession XV, in pegmatites. It is doubtful whether these minerals will be found in economic quantities in these pegmatites. They also occur in corundum-bearing rocks in Brudenell and Raglan Townships.

Quebec

Oka District, Two Mountains County (9)

A columbium-tantalum-rare earths deposit was discovered in 1953 by Molybdenum Corporation of America. Since that time, a considerable amount of exploration work has outlined large columbium-bearing areas associated with tantalum, rare earths, radioactive elements and magnetite-ilmenite minerals. The ore minerals have been identified as pyrochlore, perovskite, betafite, britholite, magnetite and ilmenite, and, in addition, a new mineral species called niocalite, a calcium columbium silicate, has been found in significant quantities. Niocalite contains 19 per cent columbium pentoxide.

The main companies in the area are: Quebec Columbium Limited jointly owned by Molybdenum Corporation of America and Kennecott Copper Corporation; Columbium Mining Products Limited jointly owned by Coulee Lead and Zinc Mines Limited and Headway Red Lake Gold Mines Limited; Oka Rare Metals Mining Company Limited; and St. Lawrence River Mines, Limited.

The Quebec Columbium Limited property is reported to contain an estimated 30,000,000 tons averaging 0.6 per cent Cb_2O_5 in one zone and 25,000,000 tons averaging 0.35 per cent Cb_2O_5 in another zone.

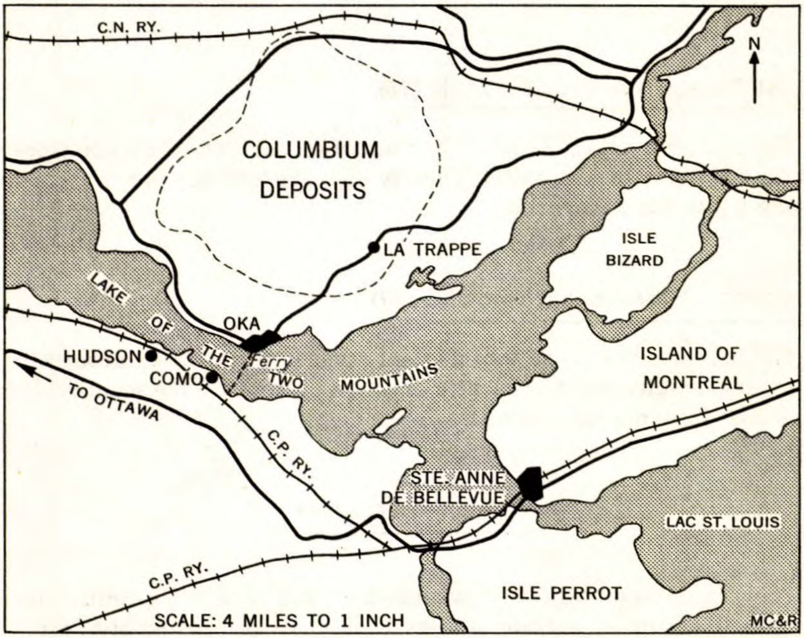


Figure 8 - Index map showing location of columbium deposits, Oka district, Two Mountains county.

Columbium Mining Products Limited has outlined some 30,000,000 tons of material averaging 0.35 per cent Cb_2O_5 in an area south of Quebec Columbium Limited.

These zones are all above the 1,000-foot level of depth and occur in the contact zone between the Grenville limestone and the Monteregian intrusive rocks.

Abitibi-East County, Preissac Township (15)

The Aldous property, Lot 53, Range VII, five miles southeast of the village of Preissac contains tantalite-columbite in a white feldspar, quartz and muscovite pegmatite.

Abitibi-East County, Figuery Township (15)

The Moneta property, Lot 12, Range II, two miles from the Amos-Malartic highway contains small quantities of tantalite in a dyke similar to the Aldous property.

Abitibi-East County, Lacorne Township (15)

The Lacorne mine, 22 miles northwest of Val d'Or, is a producer of molybdenite and bismuth. A small quantity of columbite and beryl occurs on the property but is not recovered.

Berthier County, Maissoneuve Township (17)

The Maissoneuve mine was a small muscovite producer around the year 1900. Samarskite and fergusonite crystals occur with the muscovite in a microcline and white quartz pegmatite.

Northwest Territories

The pegmatites of the Yellowknife-Beaulieu area are granitic and contain columbite-tantalite in addition to beryl, spodumene, and amblygonite. The presence of columbite-tantalite is particularly noted in the pegmatites of the Hearne Channel-Buckham Lake and Ross Lake areas. In general it might be said that the known occurrences are too low-grade to be profitable at present.

Moose, Tan and Best Bet Groups (10)

The Moose, Tan and Best Bet groups on Hearne Channel, Great Slave Lake, about 72 miles east of Yellowknife, have been operated by various companies, the latest being Boreal Rare Metals Limited which mined the Moose Dyke by open-pit method, erected a 100-ton mill in 1953 and constructed a refinery at Cap de la Madeleine, Quebec, to produce columbium and tantalum pentoxides. A small quantity of these oxides were produced during 1954 and 1955, and exported. In 1956 the company closed the refinery.

Lita Group (11)

The Lita claims are situated on the northwest shore of Buckham Lake. Columbite-tantalite occurs with beryl and spodumene.

Peg Group (12)

This group of claims lies between Ross and Ridout Lakes about 44 miles east-northeast of Yellowknife. A 50-ton mill was erected in 1946 but was operated for a short time only. Columbite-tantalite occurs with small quantities of beryl.

CHAPTER V

EXTRACTION AND METALLURGY

Concentration and Beneficiation

Columbite has a high specific gravity, and in the preliminary concentration of alluvial or tailing deposits it is removed together with cassiterite, etc., by means of sluice-boxes or jigs.

The concentrate from the riffles is taken to a dressing plant and is separated into its constituent minerals by a variety of processes employing gravity concentration, pneumatic tabling, magnetic separation and electrostatic separation.

In magnetic separation, the ilmenite and magnetite are removed first. The remainder is a mixture of magnetic cassiterite, columbite, zircon and monazite. The magnetic cassiterite is removed on the air flotation tables. The monazite and zircon are removed from the columbite by electrostatic separation leaving a marketable columbite product.

Extraction

Columbium and tantalum are extracted simultaneously from their concentrated ores by fusion methods. The tantalum and columbium salts are then separated from impurities and the metals from each other.

The concentrate is fused with hot caustic soda or caustic potash. The fused product is digested with water to remove silica and then treated with hydrochloric acid to remove iron, manganese, and other impurities, leaving the tantalum and columbium as hydrated oxides. These oxides are thoroughly washed with water, heated, and then dissolved in hydrofluoric acid. After cooling, potassium tantalum fluoride, K_2TaF_7, crystallizes out as needle-like crystals leaving potassium columbium oxyfluoride, K_2CbOF_5, in solution for subsequent recovery. The potassium columbium oxyfluoride is treated with potassium carbonate to remove the remaining tantalum. Tungsten and tin are removed by treatment with caustic soda. Columbium is finally recovered as columbium oxide by treating potassium columbate with hydrochloric acid.

The potassium tantalum fluoride crystals can be reduced to metal powder by two processes. The process used by Siemens & Halske, A.G. consists of mixing the dried fluoride with metallic sodium which are then placed in a covered steel crucible. The crucible is heated and an exothermic reaction commences which reduces the tantalum salt to metal powder. The remaining fused salts and impurities are removed by washing with water and acid. At the Fansteel Metallurgical Corporation, North Chicago, Illinois, the potassium tantalum fluoride crystals are electrolyzed in an iron pot with a graphite rod anode. Tantalum pentoxide is added to the melt and tantalum metal is produced in the bath. The electrolyte is then crushed, ground, and the tantalum powder separated by mechanical operations such as tabling. The powder is washed with water and acid to leach out the remaining traces of salts.

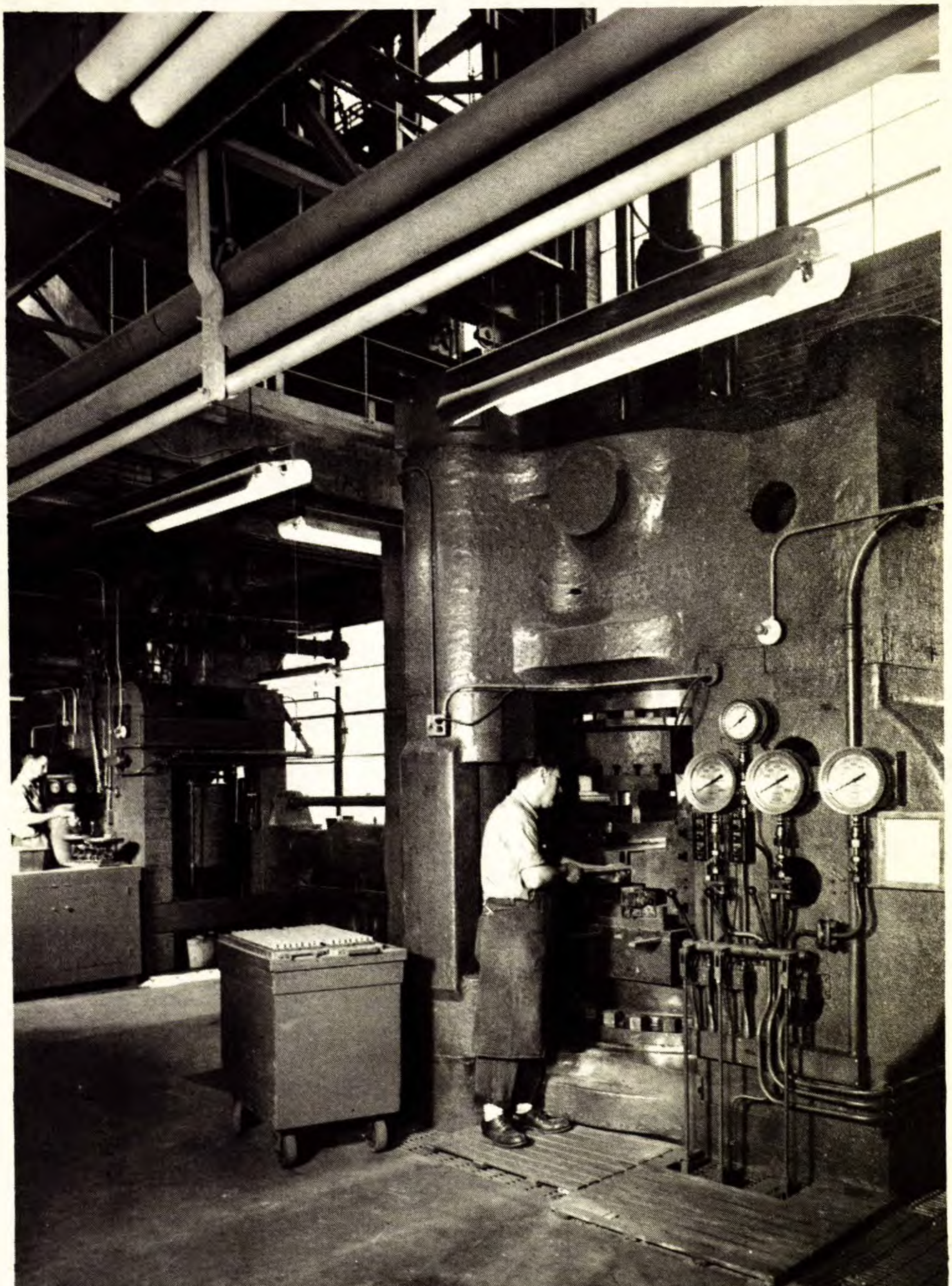
In order to produce columbium powder from columbium pentoxide, the pentoxide is mixed with columbium carbide. The mixture is pressed into bars which are then heated in a vacuum until carbon monoxide ceases to be evolved. The porous columbium metal is then fed to a ball mill and ground to a fine powder.

Casting of bars or ingots of tantalum and columbium is not practical owing to the high melting points of the metals and their reaction with atmospheric gases. It is necessary, therefore, to heat the metal powders in a high vacuum. At Fansteel, the powder is first subjected to high pressure in molds to form fragile bars which are fired in a hydrogen atmosphere for a short period. The bars are then sintered in high-vacuum electric furnaces by passing an electric current through the bar. The current heats the tantalum powder close to its melting point and causes the powder particles to weld together. The bars are then cold rolled or forged and heated again in the high vacuum to produce a dense and malleable bar.

Rolling, swaging, drawing and forming operations are then carried out on the cold metal to form sheet, rod and wire. The bars can withstand a considerable amount of deformation before becoming work-hardened and if necessary, annealing is carried out in a high vacuum at high temperature.

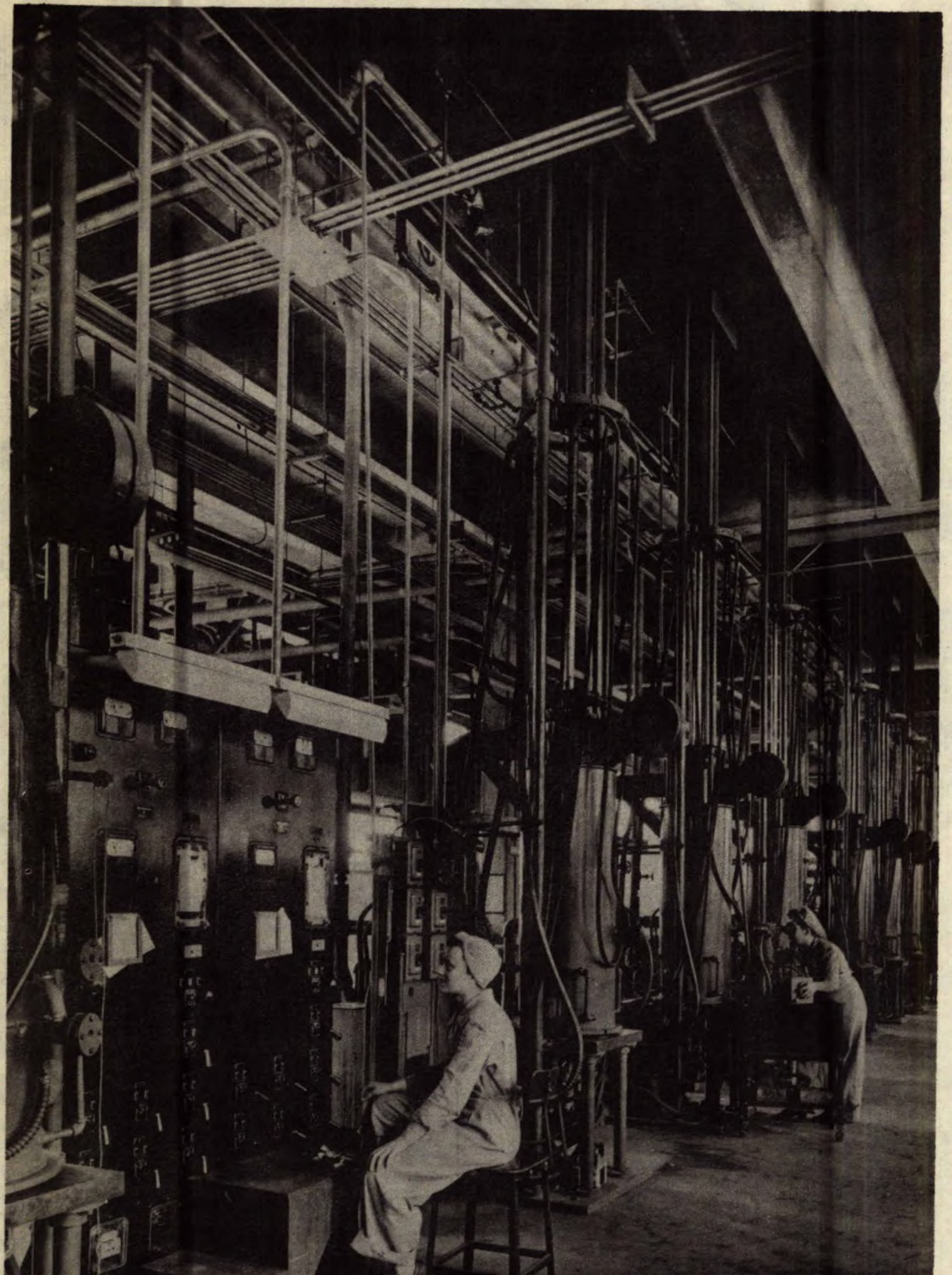
In the United Kingdom and Germany, columbium and tantalum metal powder are converted to massive metal by a different technique. The pressed bars of powder are heated in a special vacuum furnace having a series of long tungsten rods arranged as a cylinder with copper electrode rings cast on both ends. The pressed bar which is surrounded by the tungsten rods, is sintered into massive metal. The bar is then subjected to an electrical current in another high vacuum furnace which produces ductile metal.

Tantalum sponge metal has been produced by the U.S. Bureau of Mines by the reduction of tantalum chloride with magnesium in an inert atmosphere (Kroll Process).



Courtesy Fansteel Metallurgical Corporation

Figure 9 - Compacting of tantalum powder in a hydraulic press.



Courtesy Fansteel Metallurgical Corporation

Figure 10 - Sintering of bars compacted from tantalum powder in high-vacuum furnaces.

Production of Ferrocolumbium

Ferrocolumbium and ferrotantalum-columbium are produced in electric furnaces by aluminum or silicon reduction of columbite ores. These alloys are sold in a number of crushed sizes. Ferrocolumbium contains from 50 to 60 per cent columbium, a maximum of 8 per cent silicon and a maximum of 0.40 per cent carbon, while ferrotantalum-columbium contains about 40 per cent columbium, 20 per cent tantalum, 25 per cent iron and has a maximum carbon content of 0.30 per cent. These alloys are added to steel after deoxidization and about one-half hour before tapping. About 90 per cent of the columbium is recovered in the finished steel.

Liquid-Liquid or Solvent Extraction

The United States Bureau of Mines has developed a liquid-liquid extraction process for the separation of tantalum and columbium from their combined concentrates. Geomines tin slag was successfully treated by the process.

The slag having an analysis of 9.2 per cent Ta_2O_5 and 9.8 per cent Cb_2O_5 is first ground to -200 mesh and leached with concentrated hydrochloric acid to remove iron, manganese, aluminum, chromium and other acid-soluble impurities. In this step, 46 to 50 per cent of the original weight is removed. Silica, which comprised 28 per cent of the slag, is then removed by chlorination of the leached product. The tantalum-columbium chlorides are then hydrolized to hydrated oxides. Hydrofluoric acid and hydrochloric acid is added and the solution is contacted with methyl isobutyl ketone which extracts the tantalum, leaving columbium in the acid solution. Hydrochloric acid is added and the mixture recontacted with fresh ketone to extract the columbium. The tantalum pentoxides and columbium pentoxides are stripped from acid solution in the above steps by virtue of the fact that the ketone will not mix with the acid solution.

Fansteel Metallurgical Corporation will employ the process at its new refinery at Muskogee, Oklahoma.

The Mines Branch of the Department of Mines and Technical Surveys has proposed improvements in the acid mixture which may lead to a better separation of the columbium and tantalum and to the elimination of impurities.

Norwegian Practice

The Sove ores containing koppite are dressed by a method based on gravity concentration and leaching with nitric acid.

The ores, containing about 0.30 per cent Cb_2O_5, are crushed, ground and classified. The sized product is tabled, producing a pyrochlore-apatite concentrate and a residue consisting mostly of carbonates. The pyrochlore-apatite concentrate is treated with nitric acid, producing a 50 per cent Cb_2O_5 concentrate and a nitric acid-phosphoric acid solution which is further treated to produce phosphate fertilizer.

The above process was chosen by Norsk Bergverk in 1952 in order that commercial production could start in 1953. However, research by Norwegian firms led to the development of another process which was not used because of the larger capital expenses involved. In this process, the ore is crushed and ground to -6 mesh and calcined in fluid-bed roasters. The calcine is then air-slaked to remove lime and magnesia. The air-slaked calcine is then slaked in water, producing hydroxide milk, which is further processed, and a low-grade carbonate fraction which consists of from 6.5 to 11.5 per cent of the original weight of the feed. This material is tabled to remove mica, etc., and the heavy fraction is magnetically separated into magnetite and a non-magnetic fraction consisting mainly of apatite and columbium minerals. The columbium-apatite concentrate is then treated with nitric acid to produce a marketable columbium concentrate and a nitric acid-phosphoric acid solution which is used to produce phosphate fertilizer. Tests have shown that a separation of the columbium and apatite can be made by using an electrostatic separator instead of the nitric acid leaching step. Electrostatic separation increases the recovery of apatite.

CHAPTER VI

CONSUMPTION AND USE

The uses of columbium as a pure metal have not been developed as has been the case with tantalum. Apart from its use as a "getter" in vacuum tubes and as a sheath in nuclear power reactors, columbium is used in the form of ferrocolumbium, ferrotantalum-columbium and columbium carbide.

Tantalum metal on the other hand has important use as a construction material for acid-proof equipment in the chemical industry. It also is used as a "getter" in vacuum tubes.

Tantalum and columbium are both used in the carbide form either alone or in combinations with tungsten and titanium carbides. Potassium tantalum fluoride, columbium oxyfluoride and potassium columbate are used as starting chemicals in the preparation of other tantalum and columbium compounds.

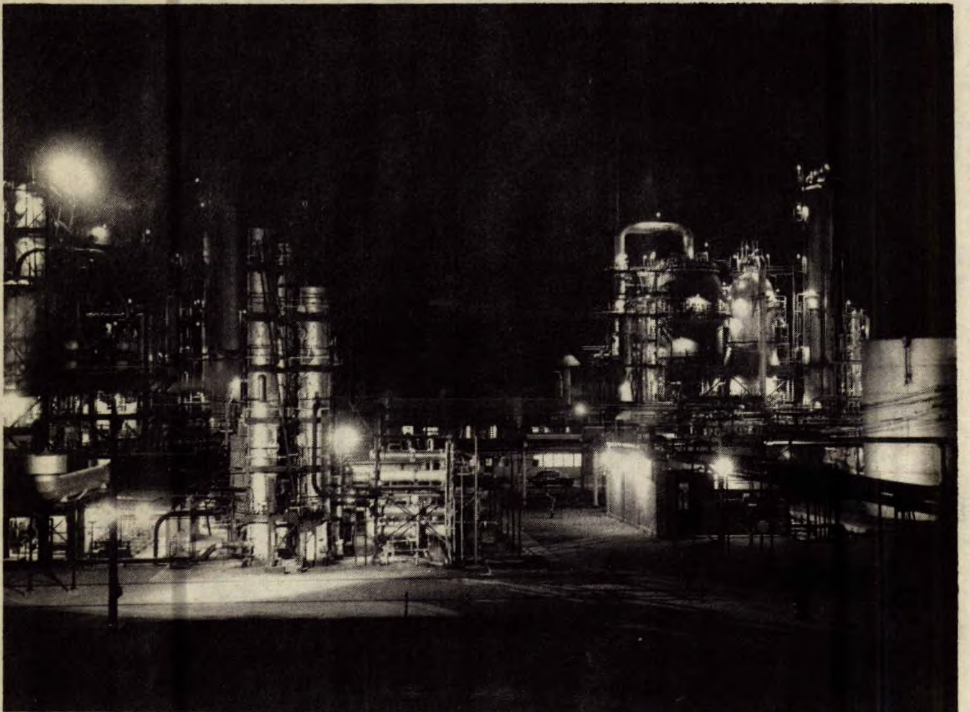
The more important consumers of tantalum and columbium ores and their products are the following:

1. Fansteel Metallurgical Corporation, North Chicago, Illinois.	Tantalum and columbium powder, sheet, rod, tubing, carbide, oxide. Potassium tantalum fluoride, columbium oxyfluoride, potassium columbate, tantalum-tungsten alloy.
2. Electro Metallurgical Company, Niagara Falls, N.Y.	Ferrocolumbium, ferrotantalum-columbium.
3. Murex Limited, Rainham, Essex, England.	Ferrocolumbium, ferrotantalum-columbium, metal powder, rod, sheet, wire.
4. Blackwell's Metallurgical Works, Ltd., Liverpool, England.	Ferrocolumbium.
5. Société Generale Metallurguique de Hoboken, near Antwerp, Belgium.	Ferrocolumbium, ferrotantalum-columbium and carbides.
6. Kennametal, Inc., Latrobe, Pa.	Carbides, tantalum and columbium metal.
7. Metallwerke Plansee, Reutte, Austria.	Tantalum and columbium carbides.
8. Kawecki Chemical Co., Boyertown, Pa.	Tantalum, columbium and chemicals.
9. Molybdenum Corporation of America.	Ferrocolumbium and ferrotantalum-columbium.
10. Fansteel Metallurgical Corporation, Muskogee, Oklahoma (proposed plant)	Tantalum and columbium metal.
11. Fabriques de Produits Chemiques de Thann et Mulhouse, S.A., Thann, France.	Columbium acid and metal.
12. Herman C. Starck, Goslar, Germany.	Tantalum metal, powder and carbide, columbium metal, powder and carbide.
13. Heraeus Quarzschmelze G.m.b.H., Hanau, Germany.	Columbium and tantalum metal.

14. Electric Furnace Products Company Ltd., Sanda, Norway.	Ferrocolumbium.
15. Quebec Metallurgical Industries Ltd., Ottawa, Canada.	Columbium oxide, alloys and sponge.
16. Kovametalli Oy, Helsinki, Finland.	Columbium and tantalum carbide.

Stainless Steel

The largest use of columbium is in the form of ferrocolumbium (50-60 per cent Cb) and ferrotantalum-columbium (approx. 40 per cent Cb, 20 per cent Ta, minimum 60 per cent Cb plus Ta) in the manufacture of stabilized austenitic stainless steels of the 18-8 type. This type of steel without the addition of a carbide stabilizer is adversely affected by heating to a temperature of from 400° to 850°C. Carbide precipitation takes place in the vicinity of the crystal



Courtesy Imperial Oil Limited

Figure 11 - An oil refinery at Sarnia, Ontario. Columbium bearing steel is used in such large field installations.

boundaries. It is also found that in welding these steels that the metal on either side of the weld seam is less resistant to corrosion after welding. In order to prevent this precipitation of carbide, an addition of columbium equal to at least ten times the amount of carbon present eliminates this defect in the steel.

The development of very low-carbon ferrochrome and heat treatment of stainless steels has obviated the use of columbium to a large degree except for certain definite end uses. For instance, when it is impractical or too expensive to dissolve carbides by heat-treatment, columbium-bearing steel is used -- an example being large field-erected welded equipment for the chemical, oil and food industries and exhaust systems of aircraft engines. The columbium content of such steels is about one per cent.

Columbium-bearing electrodes are used for welding columbium-stabilized stainless steels. Stainless steel castings stabilized with columbium are used in conjunction with wrought alloy stabilized steels. These castings bear the designation CF-8C of the Alloy Casting Institute, New York, and call for a minimum amount of columbium equal to 8 times the carbon content with a maximum of one per cent columbium or a minimum of columbium plus tantalum equal to 10 times the carbon content with a maximum of 1.35 per cent columbium plus tantalum. These castings may be welded without subsequent heat treatment.

Tantalum is similar to columbium in the formation of stable carbides. However about twice as much tantalum is required to provide the same result as columbium. Whereas about 90 per cent of the columbium in ferrocolumbium is recovered, only 70 per cent of the tantalum is recovered when using ferro-tantalum-columbium.

During the Korean Emergency, substitution of A.I.S.I. Type 347 (columbium stabilized) by A.I.S.I. Type 321 (titanium stabilized) was made by manufacturers of aircraft exhaust components because of the high price of ferrocolumbium.

Consumers of columbium alloys in the production of stainless steels in Canada are: Atlas Steels, Limited; Shawinigan Chemicals, Limited; Fahralloy Canada, Limited; Sheepbridge Engineering (Canada), Limited; Hayward Tyler of Canada, Limited; and Massey-Harris-Ferguson, Limited.

TABLE V

U.S. Production of A.I.S.I. Type 347 Stainless Steel Ingots

Year	Short Tons
1952	13,265
1953	10,679
1954	10,466
1955	10,042

TABLE VI

U.S. Production of A.I.S.I. Type 321 Stainless Steel Ingots

Year	Short Tons
1952	70,204
1953	46,150
1954	27,994
1955	35,944

TABLE VII

U.S. Consumption of Ferrocolumbium by Steel Industry

Year	Short Tons
1951	424
1952	352
1953	268
1954	280
1955	303

Source: American Iron and Steel Institute

TABLE VIII

U.K. Consumption of Ferrocolumbium by Steel Industry

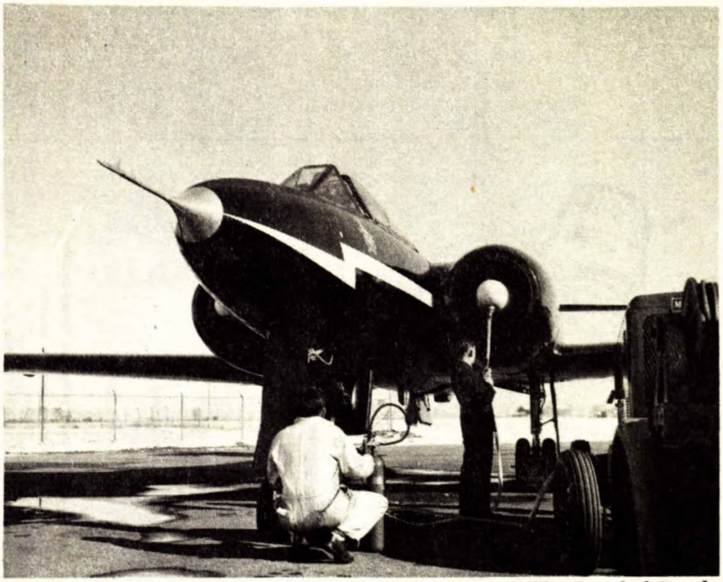
Year	Long Tons
1954	100
1955	100

Source: British Iron and Steel Federation

High-temperature Alloys

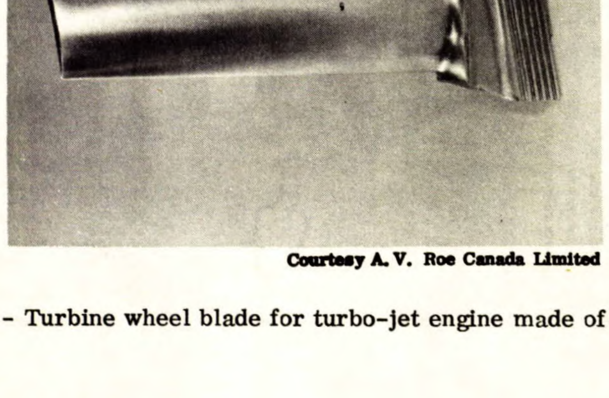
Columbium is used for imparting creep resistance to certain alloys used in high-temperature applications such as turbine wheels and buckets of jet aircraft engines.

It is added to austenitic steels in amounts less than 5 per cent plus other additive elements such as chromium, nickel, cobalt, tungsten and



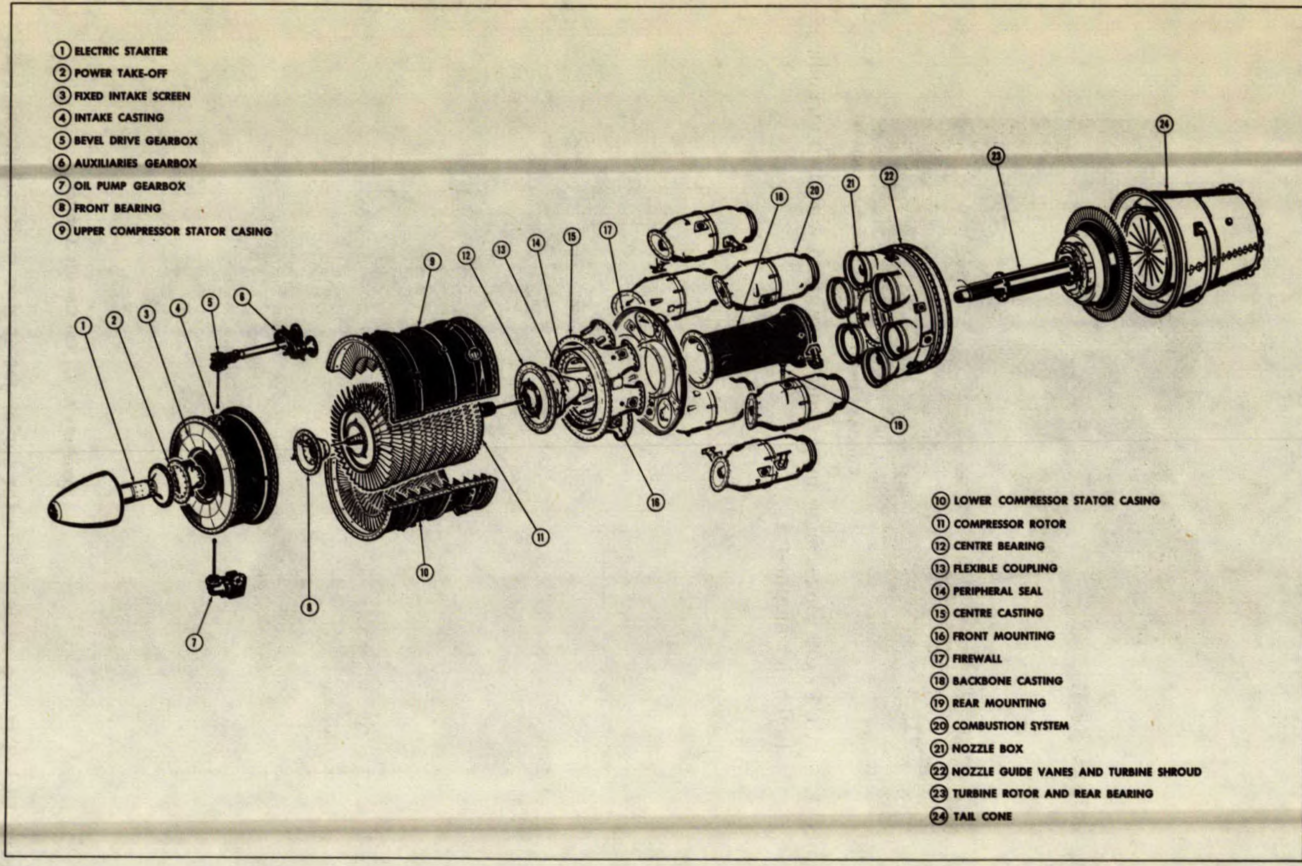
Courtesy National Film Board of Canada

Figure 12 - A CF-100 long-range interceptor equipped with two Orenda turbo-jet engines.



Courtesy A. V. Roe Canada Limited

Figure 13 - Turbine wheel blade for turbo-jet engine made of Inconel-X.



Courtesy A. V. Roe Canada Limited

Figure 14 - Components of gas-turbine aircraft engine.

molybdenum. Hardening and creep resistance is brought about by precipitation of complex carbides. For use at still higher temperatures, iron is practically eliminated from the composition of the so called "super alloys". Chromium-nickel alloys or chromium-nickel-cobalt alloys together with columbium, titanium, etc., are used.

These high-temperature metals are difficult to hot-work but because of their exceptionally high strength they are not subject to serious cracking provided proper hot-working conditions are maintained. The advent of guided missiles and rockets will probably require an ever increasing quantity of columbium.

Aluminum Casting Alloys

Small amounts of columbium, cerium, chromium, titanium, tin and lead are added to some aluminum casting alloys. These alloys are widely used in the construction of internal-combustion engine cylinder blocks and crankcases in the United Kingdom. Columbium acts as a grain refiner, reducing any tendency towards hot tearing and improving pressure-tightness. The amount of columbium contained is from 0.05 to 0.30 per cent.

British Standard LM 7 casting alloy, having the names Alminal C7, Ceralumin B and Hiduminium RR 50, specify 0.05 to 0.30 per cent of columbium and titanium. Manufacturers of castings from these alloys are: Renfrew Foundries Limited, Glasgow; J. Stone & Company Limited, London; and High Duty Alloys Limited, Slough Bucks.

Nuclear Power

In the production of power by means of fissile materials, the fuel must be protected from the cooling media by means of a protective sheath called a can which must be able to withstand the action of the fuel and the coolant and also not absorb the emitted neutrons. This can is subjected to corrosion at high temperatures and it or its oxide must be stable and resistant to further corrosion. Metals which have these characteristics are tantalum, tungsten, titanium, zirconium, columbium and molybdenum.

Possible columbium and tantalum consumption in this field will depend upon their nuclear properties, strength, fabrication properties, price and supply compared with other suitable metals. During 1956, the United States Atomic Energy Commission requested bids on 15,000 pounds of high-purity columbium metal.

TABLE IX

Composition of Some High Temperature Alloys Containing Columbium

Alloy	C	Cr	Ni	Co	Mo	W	Cb	Ti	Fe	Other
EME	.1	19	12			3.2	1.2		63	Nz - .15
Inconel X	.05	15	73				1	2.5	7	
1-1360	.10	10	70		5		2		4.5	Al 6 Ba5
N-155	.3	20	20	20	3	2	1		.32	Na .11
S-590	.4	20	20	20	4	4	4		25	
S-816	.4	20	20	44	4	4	4		3	
19-9-DL	.3	19	9		1.2	1.2	.3	.3	67	
MIT-NZ	1.0	20	30	20	3	2.2			21	Ta 2
C.S.A.	.25	19	5.5		1	1	.4		BAL.	Mn 5, Si .5
E.M.E.	.15	19	12			3	.15		BAL.	Mn .5,Si .5
F.C.B.(T)	.13	18	11				1.2		BAL.	Mn 1.3,Si .7
Gamma Columbium	.4	15	24		4		4		BAL.	Mn .7,Si 1.8
G.T.A.	.19	20	32	30	5		4		BAL.	Mn .65,Si .64,N_2 .1
G.T. 45	.08	17.3	13.8		2.9		.45	.3	BAL.	Mn 1.25,Cu 3.1,Si .5
G. 2 A.	0.4	13	13			3	.2			Mn 5,Si 1.2
G. 18 B.	.4	13	13	10	2	2.5	3			Mn .8,Si 1
G. 32	.3	19	12	45	2		1.2			Mn .8,Si .3,V 2.8
Red Fox 36	.07	18	13				1			Mn 1.3,Si .37
R. 20	.14	19	14				1.7			Mn .8,Si .3

Acid-proof Equipment and Surgical Implants

The chemical inertness of tantalum has created great interest and has led to the most important commercial use of the metal. It is particularly useful for handling chlorine and hydrochloric acid and is widely used in hydrochloric acid absorption plants as the heat transfer surface, piping and bayonet heaters. It is widely used in equipment needed for the manufacture of pharmaceutical products, aluminum anodizing racks, spinnerets for extruding rayon fibres and laboratory equipment such as spatulas and crucibles.

Surgeons have used tantalum metal for human implants because of its immunity to corrosion by body acids and its neutral effect with regard to living tissues. Injured or severed nerves are sutured with fine tantalum wire, tantalum plates are used to repair skull injuries and tantalum wire and plates are used in the repair of broken bones and in plastic surgery. Zirconium has also been successfully used in this field during the past year.

Gettering in Electron Tubes

Certain materials are used in the manufacture of vacuum tubes to absorb or combine with residual gases in the tube. Among the metals used are zirconium, tantalum, mischmetal and columbium. Flash getters are used in the form of pellets, strips or wires and are usually evaporated on the tube walls during tube manufacture to form a mirror. Columbium pellets and tantalum sheet and powder are used for this purpose.

Bulk getters are mounted on a hot electrode in the tube and absorb the gases during tube operation. Columbium and tantalum sheet and wire are used as are also zirconium and titanium. Tantalum is used to a greater extent because of its high melting point and ease of fabrication in addition to its gettering properties.

Radioisotopes

Columbium 95 and Tantalum 182 with half-lives of 35 days and 115 days respectively are produced commercially. The half-life of a radioisotope is the time required for one-half the atoms to decay.

These isotopes are produced by Union Carbide Nuclear Company who operate the Oak Ridge National Laboratory for the United States Atomic Energy Commission at Oak Ridge, Tennessee. They are produced in a nuclear reactor. They could also be produced at the Atomic Energy Project, Chalk River, Ontario.

Rectifiers and High-voltage Surge Arresters

When tantalum is immersed in an electrolyte and current is applied, an anodic film is formed which permits the flow of electrical current from the electrolyte to the tantalum but not in the opposite direction. In lightning and high-voltage surge arresters designed to protect railway signal equipment, three porous tantalum pellets are immersed in a jar of electrolyte, two being connected to each leg of the signal circuit and the third to the ground. At normal signal voltages, no current flows through the arrester, but a flash of lightning passes harmlessly through the arrester and is discharged to ground.

The same principal is used in the production of electrolytic condensers of high capacity used to minimize undesirable noises in telephone circuits.

The Balkite rectifier consists of a positive tantalum electrode and a negative antimonial-lead electrode in a sulphuric acid electrolyte. When an alternating current is applied, current flows from the lead to the tantalum but very high resistance is offered to current in the reverse direction. These rectifiers are suitable for low voltage applications such as for railway signal service and for charging storage batteries.

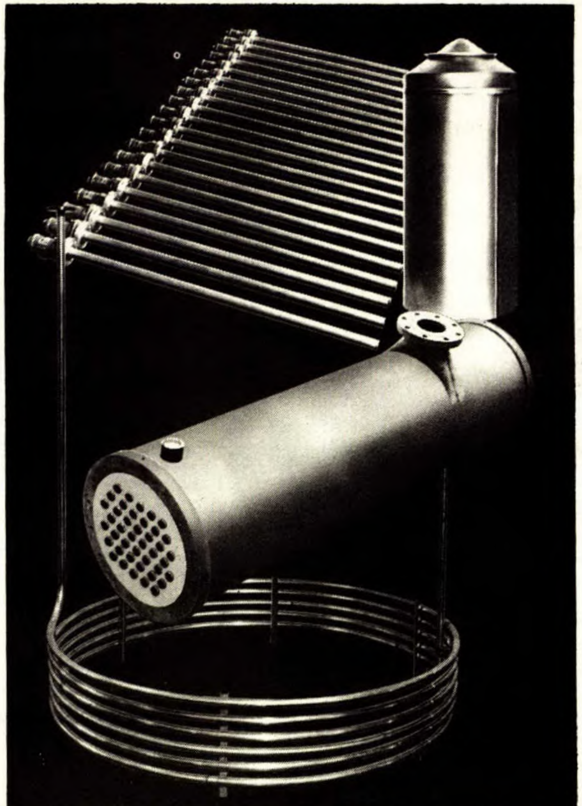
Cemented Carbides

Columbium and tantalum like tungsten, titanium, molybdenum, chromium, vanadium, hafnium and zirconium form carbides which have the required properties for use in cemented carbides. Tungsten carbide with cobalt was the first used, it was first produced by Moissan and first marketed by Krupps of Essen, Germany.

Columbium carbide was probably first produced at Metallwerke Plansee, Reutte, Austria. Columbium and tantalum carbides are prepared by heating the pure pentoxides or metal with lamp black or by dissolving tantalum in molten aluminum in the presence of carbon. After heating, the aluminum and aluminum carbide are removed by acid treatment.

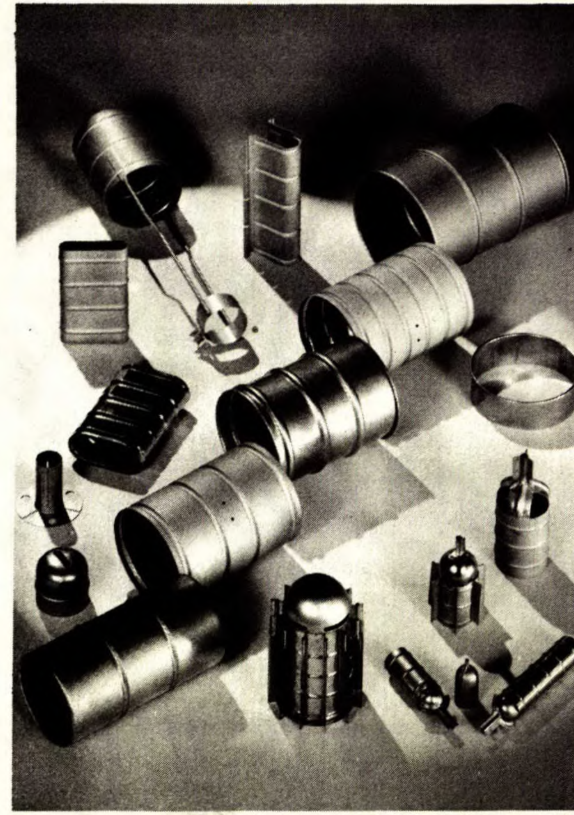
Tantalum and columbium carbides blended with tungsten carbide and cobalt metal are pressed into shape, pre-sintered in an electric furnace, machined, and given a final sinter in a hydrogen atmosphere to produce the finished carbide. Tantalum in these carbides, when used in hot forging dies, improves the anti-welding and crater-resistant properties. When used in cutting tools, the tantalum increases the strength, crater resistance and resistance to edge wear. For extremely heavy turning operation, tantalum carbide when added to tungsten carbide produces a good wear-resistant and shock-resistant tool.

The carbides possess the highest melting points of all substances and are used in blades for jet-engines. They are used also for valves, valve seats



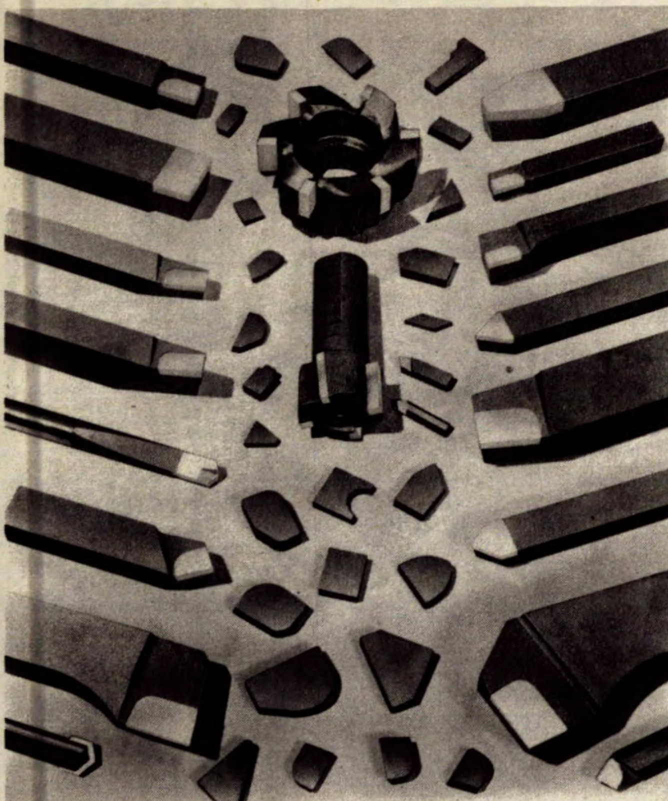
Courtesy Fansteel Metallurgical Corporation

Figure 15 - Acid-proof tantalum equipment.



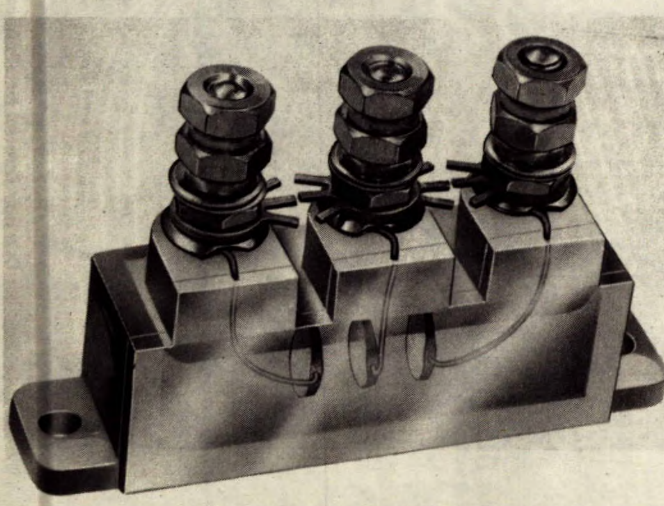
Courtesy Fansteel Metallurgical Corporation

Figure 16 - Anodes for electronic power tubes.



Courtesy Fansteel Metallurgical Corporation

Figure 17 - Tools and cutter blanks containing combination of carbides of tungsten, tantalum, columbium and titanium.



Courtesy Fansteel Metallurgical Corporation

Figure 18 - Lightning and high voltage surge arrester.

and guides for high-power reciprocating engines. Producers are: Kennametal Inc., Latrobe, Pennsylvania; Metallwerke Plansee, Reutte, Austria; and Vascoloy-Ramet, Waukegan, Illinois.

Tantalum and columbium carbides are also added to alloys composed principally of cobalt, chromium and tungsten to form a series of wear-, heat- and corrosion-resistant alloys. Columbium and tantalum carbides perform a self-lubricating action in such alloys when used as a cutting tool material.

Tantalum as a Catalyst in the Synthesis of Butadiene from Ethyl Alcohol

During World War II, a wide range of tantalum pentoxide and silica catalysts were used in the production of synthetic rubber. The catalyst consisted of silica gel impregnated with alcohol plus tantalum pentoxide. Columbium pentoxide and zirconia catalysts were also used.

Butadiene plants in the United States using these catalysts were operated by Carbide and Carbon Chemicals Corporation and by Koppers Company, Inc.

Optical Glass

Tantalum pentoxide is used with potassium oxide and silica in the production of optical glass that has a high refractive index. The refractive index increases with an increase in tantalum pentoxide content. The high refractive index and low dispersion makes this glass particularly useful in aerial camera lenses.

Tantalum-Tungsten Alloy

This alloy which contains 7.5 per cent tungsten with the balance being tantalum maintains its elasticity when heated to a high temperature and is used extensively in electronic tubes where this characteristic is required.

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