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COLUMBIUM AND TANTALUM

A Materials Survey

By William R. Barton

information circular 8120

* * * * *



UNITED STATES DEPARTMENT OF THE INTERIOR

Stewart L. Udall, Secretary

BUREAU OF MINES

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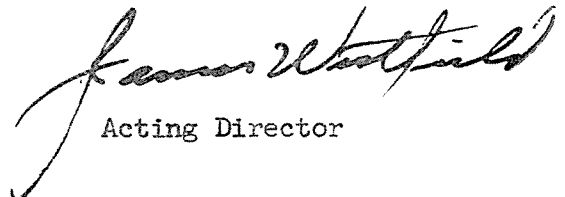
September 24, 1962

Honorable Edward A. McDermott
Director
Office of Emergency Planning
Executive Office of the President
Washington 25, D.C.

Dear Mr. McDermott:

The Materials Survey on Columbium and Tantalum has been prepared by the Bureau of Mines under the terms of the April 15, 1955 agreement between the Department of the Interior and the Office of Defense Mobilization. This agreement assigned responsibility to Interior for the preparation and revision of Surveys covering 45 mineral commodities. Copies are being forwarded to you.

Sincerely yours,


Acting Director

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Foreword

Materials Surveys are designed to bring into a single publication all the fundamental data needed by war or defense personnel with major responsibilities with respect to the survey subjects. The surveys dealing with metals and minerals summarize the demand-supply position in the United States and include information on production, imports, consumption, exports, capacity, interchangeability, substitutes, possibilities for expansion, and pertinent history, usually in some detail back to 1925. The properties of the commodity and its principal compounds and alloys are described. Exploration, mining, and metallurgical methods are discussed. Domestic and foreign resources and reserves are covered. An extended presentation of the structure of the industry, including major corporations, transportation service, processing facilities, interrelationships with other industries, pertinent laws and taxation policies, tariffs, Government controls, special labor problems, and history of wartime control experiences, are included. Other special data are presented for particular commodities. A general cutoff date for inclusion of information in this report was its availability on or before June 30, 1960.

In undertaking Materials Surveys, precedence is given to commodities of highest priority in defense urgency. Columbium is an important high temperature and ferroalloying metal, and tantalum is important for both its high temperature and electronic uses. Both metals are essential to the Nation's military and civilian economies.

The Columbium and Tantalum Materials Survey was prepared in the Division of Minerals first under the supervision of Charles T. Baroch, Chief, Branch of Rare and Precious Metals, and later under the supervision of Paul Yopes, Chief, Branch of Nonferrous Metals. The manuscript was reviewed, in whole or in part, by specialists in the Bureau of Mines, Geological Survey, other Government agencies, and industry.

CHARLES W. MERRILL
Chief, Division of Minerals

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COLUMBIUM AND TANTALUM

A MATERIALS SURVEY¹

By

William R. Barton²

Introduction and Summary

COLUMBIUM (niobium) and tantalum are similar refractory metals with closely related properties and uses and close association in ore deposits. Until recently both metals, particularly columbium, were essentially laboratory curiosities. However, since the beginning of World War II, applied technology, under the impetus of national security requirements, has realized many chemical, electronic, nucleonic, and missile and rocketry applications, where high temperature or corrosive environments require the use of columbium or tantalum. At the same time, advances in these fields have resulted in intensive research on the geological distribution, resources, metallurgy, and properties of these two metals; their alloys and compounds; and the development of a full-fledged columbium-tantalum industry. The future appears to promise continued acceleration in such studies and ever-increasing demand for these elements.

The raw material supplies of columbium are abundant, and the extractive metallurgy of columbium is well enough advanced to assure adequate supplies of the element at reasonable prices to meet any projected demands. Although the extractive metallurgy of tantalum is similarly well developed, raw material supplies are so limited that, lacking new ore discoveries, an increase in demand to five times the 1959 level would severely tax ore-production facilities. Approximately 120 tons of tantalum and about one-half as much columbium was produced in 1959. Additional quantities are consumed as compounds or additive alloys such as ferrocolumbium and ferrotantalum columbium (55).³

¹ Work on manuscript completed December 1961.

² Geologist (mineral deposits), Bureau of Mines.

³ Italicized figures in parentheses refer to items in the selected bibliography at the end of this report.

CHAPTER 1.—PROPERTIES, FORMS, SPECIFICATIONS, AND USES

PROPERTIES

Tantalum possesses certain remarkable properties that have several important applications in the nucleonic, electronic, and extraterrestrial age (51, 58). It exhibits a rectifying or electrolytic-valve action in an electrolyte because it can form anodic oxide films of unusual stability. Almost completely resistant to corrosion, it has one of the highest melting points of all the elements. At elevated temperatures it will absorb and retain common gases, acting as a getter (sequesterer) to maintain high vacuum in electronic tubes. It also has low vapor pressure, low work function, low thermal expansion, excellent ductility, and excellent weldability to recommend it. At very high temperatures it oxidizes rapidly.

Columbium resembles its sister metal tantalum in most respects, differing more in degree of auspicious properties than in its basic nature. It is a less favorable film former than tantalum and somewhat less resistant to corrosion and heat, but it has more pronounced sequestering properties than tantalum. For the same reason, it oxidizes even more rapidly at elevated temperatures, although far less rapidly than its competitor refractory metal, molybdenum. It has an even lower work function than tantalum. Important in this age of space travel and nucleonics is its lighter weight and a thermal neutron capture cross section only about one-twentieth as great as its sister metal tantalum (tables 1 and 2).

The only metal that is superior to tantalum in resistance to chemical corrosion is platinum. The halogens, organic compounds, most salts, gases at normal temperatures, and water with a pH less than 9 will not affect tantalum, which is particularly recommended for use in processes involving chlorine and its compounds, including hydrochloric acid. Alkalis and salts that contain or hydrolyze to strong alkalis will attack the metal at normal temperatures. Hydrofluoric acid will attack tantalum rapidly, as will fuming sul-

furic acid (oleum). Phosphoric and sulfuric acids will corrode the metal above 145° and 170° C., respectively (table 3). Tantalum is also one of the materials most resistant to attack by molten metals.

Columbium has less corrosion resistance to acids than tantalum; principally, it is more readily attacked by sulfuric acid. Like tantalum, it is seriously corroded by alkalis, but compared with most other metals, columbium has excellent resistance to chemical corrosion (tables 3-6).

COMMERCIAL FORMS AND SPECIFICATIONS

Columbium Ore or Concentrate

Columbite ore or concentrate is marketed on the basis of a minimum of 65 percent combined columbium and tantalum pentoxides in specified ratios, such as 10 to 1, 8½ to 1, or 4 to 1 for Cb_2O_5 to Ta_2O_5 , respectively. National stockpile purchase specifications for columbite (P-15-R2) are based on three grades. Grade A requires 65 percent combined pentoxides and a minimum ratio of 10 parts Cb_2O_5 to 1 part Ta_2O_5 . Grade I requires 60 percent combined pentoxides with a minimum ratio of 8.5 parts Cb_2O_5 to 1 part Ta_2O_5 . Grade II requires 55 percent contained combined pentoxides with a minimum ratio of 1 part Cb_2O_5 to 1 part Ta_2O_5 . For all grades the following maximum permissible impurities were specified: FeO, 25 percent; MnO, 8 percent; TiO_2 , 8 percent; SnO_2 , 8 percent; TiO_2 plus SnO_2 , 12 percent. Phosphorus could not exceed 0.20 percent in the first two grades or 0.30 percent in the last.

General Services Administration (GSA) specifications for domestic columbite ore purchased under Public Law 733, 84th Congress (expired Dec. 31, 1958), were established as follows:

For lots of more than 2,000 pounds, a minimum of 35 percent combined pentoxides and a Cb_2O_5 - Ta_2O_5 ratio of not less than 1:1 were

COLUMBIUM AND TANTALUM—A MATERIALS SURVEY

TABLE 1.—Properties of columbium and tantalum

Item	Columbium	Tantalum
Atomic number.....	41	73
Atomic volume.....cc./g.-atom..	10.83	10.89
Atomic weight.....	92.91	180.95
Boiling point.....°C.	4,930	5,430
Coefficient of linear thermal expansion at room temperature.....	7.1×10^{-6}	$6.5, 6.6 \times 10^{-6}$
Crystal structure.....	Body-centered cube	Body-centered cube
Density at 20° C.....g./cc..	8.57	16.6
Elongation at 20° C., annealed sheet.....	49	40
Hardness (Rockwell-E), annealed sheet.....	50 ±	60
Heat of combustion.....cal./g..	2,379	1,379
Ionization potential.....volts..	6.77	7.3 ± 0.3
Lattice constant at 20° C.....Angstroms..	3.3004	3.3026
Magnetic mass susceptibility.....egs. units..	1.5×10^{-6}	0.93×10^{-6}
Melting point.....°C.	2,415	2,997
Positive-ion emission.....ev.	5.52	10.0
Specific heat at 10° C.....cal./cm. ² /cm./sec./°C.	0.065	0.034
Specific resistance at 20° C.....microhm/cm..	14.1	21.3
Thermal neutron cross section.....barns..	1.1	0.130
Ultimate tensile strength at 20° C., annealed.....tons/in. ² .	17.7	25
Young's modulus of elasticity at room temperature.....lb/in. ² .	15.08×10^6	27.0×10^6
Work function.....ev.	4.01	4.10
Vapor pressure.....mm. Hg..	1.0×10^{-11}	9.525×10^{-11}
Electrical conductivity at 18° C.....IACS, percent..	0.00395	0.00382
Temperature coefficient of resistance at 20° C.....	0.1926	0.3749
Electrochemical equivalent.....mg./coulomb..	1.80	2.05
Refractive index.....	8.3	4.38
Superconductivity, upper limit.....°K.	170.9	180
Heat of sublimation.....kcal./g.-atom..	6.087	6.101
Heat capacity at 100° C.....cal./g.-atom/°C.	10.49	11.71
Entropy at 400° K.....ST cal./g.-atom/°K.	0.96	1.12
Standard electrode potential.....volts..	1.47	1.46
Atomic radius.....Angstroms..	2.8	3.0-4.0
Creep strength, tons/in. ² to cause 1 percent in 24 hrs.....	75,000-150,000	100,000-150,000
Tensile strength, at room temperature.....p.s.i..	13,000-17,000	15,000-20,000
Tensile strength, at 1,000° C.....p.s.i..		

¹ At 1,680° C.
² At 1,727° C.

TABLE 2.—Comparison of columbium and tantalum with other materials

Material	Density, g./cc.	Melting point, °C.	Boiling point, °C.	Thermal neutron capture cross section, barns	Material	Density, g./cc.	Melting point, °C.	Boiling point, °C.	Thermal neutron capture cross section, barns
Columbium.....	8.57	2,487	4,929	1.1	Nickel.....	8.9	1,453	2,899	4.5
Tantalum.....	16.6	3,013	5,300	21.0	Cobalt.....	8.9	1,492	3,100	34.0
Titanium.....	4.507	1,670	3,260	5.8	Iron.....	7.83	1,535	3,033	2.39
Zirconium.....	6.4	1,852	2,900	0.22	Rhenium.....	21.40	3,180	5,903	84.0
Molybdenum.....	10.2	2,625	4,890	2.4	Carbon.....	2.26	3,500	4,200	.0032
Tungsten.....	19.3	3,410	5,930	17.7					

required; lots of less than 2,000 pounds were required to contain a minimum of 50 percent combined pentoxides in any ratio. In all instances maximum content of certain impurities also was specified. TiO₂ had to be less than 8 percent; FeO, less than 25 percent; MnO, under 13 percent; and SnO₂, not to exceed 8 percent.

Pyrochlore concentrate from Norway contains a minimum of 50 percent Cb₂O₅. Euxenite concentrate from Idaho contains more than 26 percent Cb₂O₅ plus Ta₂O₅.

Tantalum Ore or Concentrate

Specifications for tantalum ore are not regularly quoted but are a matter of negotiation between purchaser and vendor. Premium ore contains 60 percent or more Ta₂O₅ but ore containing as little as 20 percent Ta₂O₅ is purchased. In such low-grade tantalite, the Cb₂O₅ to Ta₂O₅ ratio may be 1:1, or Cb₂O₅ may even exceed Ta₂O₅.

National Stockpile Purchase Specification P-54-R2 required tantalum minerals to cor

TABLE 3.—Corrosion behavior of tantalum (58)¹

Corrosive media	Temperature, °C.	Corrosion resistance of tantalum ²	Corrosive media	Temperature, °C.	Corrosion resistance of tantalum ²
Acetic acid:			Iodine:		
Liquid.....	20-390	Excellent.	Wet.....	20-150	Excellent.
Vapor.....	20-150	Do.	Dry.....	20-150	Do.
Acetic anhydride.....	20-150	Do.	Magnesium chloride.....	20-150	Do.
Alcohol:			Mercuric chloride ⁷	100	Do.
Ethyl.....	20-150	Do.	Methyl chloride:		
Methyl.....	20-150	Do.	Wet.....	20-150	Do.
Aluminum chloride ³	20-150	Do.	Dry.....	20-150	Do.
Aluminum potassium sulfate (alum).....	20-150	Do.	Methyl-sulfuric acid.....	20-150	Do.
Ammonium chloride.....	20-150	Do.	Monochlorobenzene.....	20-150	Do.
Ammonium hydroxide.....		Do.	Nickel chloride.....	20-150	Do.
Ammonium nitrate.....	20-150	Do.	Nickel nitrate.....	20-150	Do.
Ammonium sulfate.....	20-150	Do.	Nickel sulfate.....	20-150	Do.
Amyl acetate.....	20-150	Do.	Nitric acid.....	20-150	Do.
Amyl chloride.....	20-150	Do.	Nitric-sulfuric acid.....	20-150	Do.
Antimony trichloride.....	20-150	Do.	Nitrobenzene.....	20-150	Do.
Aqua regia.....	20-60	Do.	Nitrous acid.....	20-150	Do.
Arsenic acid.....	20-150	Do.	Nitrosyl chloride:		
Benzoic acid.....	20-150	Do.	Wet.....	20-150	Do.
Bromine:			Dry.....	20-150	Do.
Liquid.....	20-150	Do.	Organic chlorides.....	20-150	Do.
Dry.....	20-150	Do.	Oxalic acid ⁷	20-95	Do.
Carbon tetrachloride.....		Do.	Perchloric acid.....	20-150	Do.
Chlorine:			Phenol ⁷	20	Do.
Saturated water.....	20-150	Do.	Phosphoric acid.....	20-120	Do.
Gas.....	20-150	Do.	Phthalic anhydride.....	20-150	Do.
Chlorobenzene.....	20-150	Do.	Potassium chloride.....	20-150	Do.
Chloroform.....	20-150	Do.	Potassium dichromate ⁸	20-150	Do.
Chromic acid ⁴	20-150	Do.	Potassium hydroxide:		
Citric acid.....	20-150	Do.	Aqueous solution ⁹	110	Do.
Cupric chloride.....		Do.	Do. ¹⁰	110	Poor.
Ferric chloride ³	20-150	Do.	Silver nitrate ¹¹	20-150	Excellent.
Ferric nitrate.....	20-150	Do.	Sodium bromide ⁵	20-150	Do.
Ferric sulfate.....	20-150	Do.	Sodium chlorate.....	20-150	Do.
Ferrous chloride.....	20-150	Do.	Sodium chloride ⁶	20-150	Do.
Ferrous sulfate.....	20-150	Do.	Sodium hydroxide:		
Fluorine.....	20-150	Poor.	Aqueous solution ⁹	100-110	Do.
Fluosilicic acid.....	20-150	Do.	Do. ¹⁰	100-110	Poor.
Formic acid.....	20-150	Excellent	Sodium nitrate ¹¹	20-150	Excellent
Hydrobromic acid ⁵	20-150	Do.	Stannic chloride ³	20-150	Do.
Hydrochloric acid.....	20-150	Do.	Stannous chloride.....	20-150	Do.
Hydrocyanic acid.....	20-150	Do.	Sulfuric acid:		
Hydrofluoric acid.....	20-150	Poor.	Air free, no velocity....	20-120	Do.
Hydrogen bromide.....	20-150	Excellent.	Air saturated, no velocity.....	20-120	Do.
Hydrogen chloride ⁶	20-150	Do.	Fuming.....	20-150	Poor.
Hydrogen peroxide.....	20-150	Do.	Zinc chloride ³	20-150	Excellent
Hypochlorous acid.....	20-150	Do.	Zinc sulfate.....	20-150	Do.

¹ Concentration is 0-100 percent except as indicated in footnotes 7, 9, and 10.

² Excellent—less than 0.5-mil penetration per year; poor—more than 10-mil penetration per year.

³ With or without HCl.

⁴ Very slight attack at high temperatures and concentration.

⁵ With or without free Br.

⁶ With or without free Cl.

⁷ Saturated.

⁸ With or without H₂SO₄.

⁹ 5 percent concentration.

¹⁰ 40 percent concentration.

¹¹ With or without HNO₃.

tain a minimum of 40 percent Ta₂O₅, which equals or exceeds the Cb₂O₅ content. Individually, TiO₂ or SnO₂ could not exceed 4 percent or, combined, exceed 6 percent.

Under Public Law 733, purchases of lots of more than 2,000 pounds required a minimum of 25 percent Ta₂O₅ plus 20 percent Cb₂O₅. For lots less than 2,000 pounds, 70 percent combined pentoxides and at least 40

percent Ta₂O₅ was the minimum requirement; the TiO₂ or SnO₂ contents were the same as for the National Stockpile Purchase Specification P-54-R2.

Ferrocolumbium

Specifications for ferrocolumbium, a silvery metal, vary, depending on the manufacturer and process used. Alloy made in

TABLE 4.—Corrosion of tantalum by liquid metals (58)

Liquid metal	Corrosion behavior of tantalum
Bismuth.....	Slight attack after 160 hours at 980° C. Resistant up to 1,000° C. Pronounced intergranular attack after 227 hours at 1,000° C. Tantalum carbide showed no attack after 167 hours at 1,000° C.
Lead.....	Excellent resistance to attack at 1,000° C; rate of attack appears to be less than 0.1 mil per year.
Bismuth-lead (eutectic alloy).....	Poor resistance to pitting after 1 hour at 1,093° C; estimated disintegration rate 2,007 mils per year.
Lithium.....	Good resistance at 1,000° C.
Mercury.....	Probably would resist attack at elevated temperatures.
Potassium.....	Similar to sodium.
Potassium-sodium.....	Do.
Sodium.....	Satisfactory corrosion resistance up to 1,010° C, provided that the oxygen content of the liquid metal is low.
Thallium.....	If oxide present in the liquid metal, attack can start as low as 250° C.
Tin.....	Tantalum has been suggested for possible use in liquid thallium.
Tin.....	After 1 hour at 1,740° C, tin dissolved 0.33 weight-percent tantalum.
Uranium.....	Dissolves in liquid uranium at all temperatures; upper limit as a container material about 1,450° C.
Uranium.....	Becomes permeable to the liquid metal as a result of intergranular diffusion between 1,200° and 1,250° C, resulting in the loss of mechanical strength.
Zinc.....	Foil completely dissolves in less than 50 hours at 440° C.

an electric furnace contains 50 to 65 percent columbium and 5 or 6 percent tantalum (ranging from 10:1 to 13:1 in columbium-tantalum ratio). Silicon does not exceed 8.0 percent, and carbon is less than 0.4 percent.

Aluminothermic alloy contains 58 to 63 percent columbium and 3 to 6 percent tantalum. Silicon does not exceed 1.5 percent, and carbon is less than 0.15 percent. Aluminum may be as high as 1.5 percent.

Special-grade ferrocolumbium, which is especially low in high neutron capture cross-section elements such as tantalum and boron, is made for steels to be used in reactors.

Commercial stock sizes of ferrocolumbium are minus 2-inch, minus 1/2-inch, minus 1/4-inch, minus 8-mesh, and minus 20-mesh. Finer sizes down to minus 150-mesh can be obtained by special order.

Eutectic alloys of iron and columbium (about 73 percent columbium) were produced experimentally in 1960 and were expected to increase in favor as a steel additive in place of regular ferrocolumbium. The special grade ferrocolumbium shown in table 7 approaches the eutectic composition.

TABLE 5.—Oxidation of tantalum and tantalum alloys in air at 1,200° C. (58)

Alloy composition, wt.-pct.	Weight gain, mg./cm. ² /hr.	Appearance of scale	Alloy composition, wt.-pct.	Weight gain, mg./cm. ² /hr.	Appearance of scale
100Ta.....	105.5	Voluminous, porous, white.	Ta-20V.....	159.9	Voluminous, porous, brown.
Ta-2.9B.....	138.9	Do.	Ta-30V.....	96.2	Porous, gray-white.
Ta-5.6B.....	94.3	Do.	Ta-5W.....	57.4	Porous, white.
Ta-7.4B.....	59.4	Porous white.	Ta-10W.....	61.4	Do.
Ta-5Cb.....	50.6	Voluminous, porous, white.	Ta-30W.....	45.7	Do.
Ta-10Cb.....	55.8	Voluminous, porous, white-brown.	Ta-32W.....	3 59.0	Thick, adherent.
Ta-30Cb.....	(1)	Voluminous, porous, white.	Ta-50W.....	12.0	Thin, white.
Ta-7Co.....	114.5	Pink, porous.	Ta-68W.....	3 21.0	Partly granular, nonadherent; thin layer, very adherent, yellowish.
Ta-4.6Cr.....	49.3	Tan, porous.			Yellow, porous.
Ta-11.3Cr.....	33.5	Porous, brown.	Ta-0.1Y.....	90.1	Do.
Ta-6.2Fe.....	64.4	Do.	Ta-1Y.....	82.8	Do.
Ta-8Fe.....	50.1	Thin, adherent, gray.	Ta-1Zr.....	90.6	Voluminous, porous, white.
Ta-1Hf.....	154.3	Voluminous, porous, white.	Ta-5Zr.....	35.0	Gray, porous.
Ta-5Hf.....	69.9	Adherent, tan.	Ta-10Zr.....	32.4	Do.
Ta-10Hf.....	47.7	Do.	Ta-20Zr.....	61.0	Do.
Ta-30Hf.....	28.0	Do.	Ta-30Zr.....	59.8	Do.
Ta-32Hf.....	39.0	Dark, adherent.	Ta-10Ti-5Cb.....	74.7	Tan, thick, adherent.
Ta-50Hf.....	18.0	Thin, adherent, tan.	Ta-10Ti-5W.....	76.0	Do.
Ta-68Hf.....	10.0	Dark, thin, adherent.	Ta-20Ti-5Al.....	16.7	Thin, yellow-white, adherent.
Ta-5Mo.....	69.9	Porous, gray.	Ta-20Ti-5Cb.....	28.9	Thin, adherent, light-yellow.
Ta-10Mo.....	51.5	Do.	Ta-20Ti-5Cr.....	15.5	Thin, adherent, beige.
Ta-20Mo.....	2 28.8	Do.	Ta-20Ti-5Mo.....	43.1	Thin, adherent, tan.
Ta-30Mo.....	(1)	Do.	Ta-20Ti-5V.....	19.3	Thin, adherent, dark-gray.
Ta-10Ni.....	57.0	Adherent, black.	Ta-20Ti-5W.....	39.4	Thin, adherent, light-yellow.
Ta-49.3Ni.....	18.0	Small amount of black.	Ta-20Ti-10W.....	42.1	Thin, adherent, yellow.
Ta-5Re.....	71.3	Thin, adherent, yellow-white.	Ta-5W-10V.....	122.0	Voluminous, porous, brown.
Ta-10Re.....	(1)	Granular, yellow.	Ta-4W-4Hf.....	101.0	Voluminous, porous, white.
Ta-32Re.....	3 25.0	Nonadherent, granular, yellow.	Ta-5W-10Hf.....	72.2	Thin, adherent, white.
Ta-3.3Si.....	40.7	Porous, white.	Ta-8W-8Hf.....	24.0	Very adherent; whitish part spalled.
Ta-0.5Th.....	101.8	Voluminous, porous, yellow.			Partly spalled; whitish very adherent.
Ta-8Th.....	168.8	Do.	Ta-8W-16Hf.....	32.0	Thick, nonadherent, light-yellow.
Ta-1Ti.....	104.2	Voluminous, porous, white.			Thin, very adherent, whitish.
Ta-5Ti.....	39.1	Yellow, porous.	Ta-26W-5Hf.....	64.0	Thin, spalled, gray yellow.
Ta-10Ti.....	21.5	Small amount of black.	Ta-33W-33Hf.....	18.0	Thin, adherent, whitish.
Ta-27Ti.....	21.5	Thin, adherent, white.	Ta-48W-4Hf.....	3 26.0	Thin, very adherent, yellow-black.
Ta-5V.....	55.5	Porous, gray-white.	Ta-60W-4Hf.....	4 7.5	Granular, nonadherent, with thin white adherent subscale.
Ta-10V.....	68.4	Do.	Ta-33W-33Re.....	3 24.0	

1 Rate data not available; part of scale lost at conclusion of test because of crucible fracture.

2 Sample smoked during test, indicating MoO₃.

3 Calculated from weight loss after scale was removed.

4 Probably low value because of loss of tungsten oxide by volatilization. Scale could not be removed mechanically, so value could not be calculated from weight loss after scale removal.

TABLE 6.—Corrosion of columbium in aqueous media¹

Medium	Temperature, °C	Duration of test, days	Loss in weight, g./dm. ² /day
Hydrochloric acid:			
20 percent.....	21	82	0.00025
Concentrated.....	21	82	.0006
Do.....	100	67	² .0234
Nitric acid: Concentrated....	100	67	0
Aqua regia.....	22	6	0
Sulfuric acid:			
20 percent by volume.....	21	3,650	.00002
25 percent by volume.....	21	3,650	.00003
Concentrated, 98 percent..	21	3,650	.00056
Concentrated.....	50	67	² .0048
Do.....	100	32	² .1131
Do.....	150	2	² 1.247
Do.....	175	1	8.32+
Concentrated + CrO ₃	100	42	² .0464
Phosphoric acid:			
85 percent.....	21	82	.00007
85 percent.....	100	31	² .0193
Tartaric acid: 20 percent....	22	82	0
Oxalic acid: 10 percent.....	21	82	.0033
Ammonium hydroxide.....	21	82	0
Sodium carbonate: 20 percent.	100	50	² .0074
Caustic soda:			
5 percent.....	21	31	.0066
5 percent.....	100	5	² 13.0
Potassium hydroxide:			
5 percent.....	21	31	.0442
5 percent.....	100	5	² .2744
Hydrogen peroxide: 30 percent.	21	61	.0011

¹ Figures from Fansteel Metallurgical Corp.
² Sample became brittle.

National Stockpile Purchase Specification P-104 for ferrocolumbium requires a minimum columbium content of 60 percent (Cb-Ta ratio not less than 8:1). Maximum permissible impurities are: aluminum, 1.5 percent; tin, 0.15 percent; manganese, 2 percent; silicon, 4 percent; carbon, 0.15 percent; sulfur, 0.03 percent; phosphorus, 0.03 percent.

Ferrotantalum-Columbium

Ferrotantalum-columbium, a silvery metal, usually contains 40 to 45 percent columbium and 15 to 20 percent tantalum. It contains

about 25 percent iron and not more than 0.3 percent carbon. It is available in the same stock sizes as ferrocolumbium.

National Stockpile Purchase Specification P-88-R1 requires a minimum of tantalum plus columbium of 45 percent; the columbium-tantalum ratio must be less than 8:1 but at least 3:1 by weight. Aluminum must not exceed 3.0 percent; silicon, 3.0 percent; tin, 0.15 percent; titanium, 4.0 percent; sulfur, 0.03 percent; phosphorus, 0.03 percent; and carbon, 0.2 percent. Two sizes are accepted: Size I (minus 2-inch) and size II (minus 1/2-inch).

Tantalum-Columbium-Aluminum

This new additive will produce a martensitic titanium-base alloy. It contains 12 to 24 percent tantalum; 24 to 28 percent, columbium; and 48 to 52 percent, aluminum. Iron is about 1.5 percent and silicon about 0.5 percent.

Chrome-Columbium and Nickel-Columbium

These additives for high temperature alloys contain about 60 percent columbium plus tantalum (minimum columbium-tantalum ratio, 10:1), less than 7.5 percent silicon, and a maximum of 0.1 percent carbon. Some experimental forms have contained 80 percent columbium and 20 percent chromium or nickel.

Columbium Oxide

Several grades of white columbium pentoxide powder are available commercially. These are variously designated by the individual companies, which call their products

TABLE 7.—Typical analyses of some columbium-tantalum master alloys, percent

Alloy	Cb+Ta	Cb	Ta	C	Si	Al	Sn	Ti	Mn	Cr	Fe
Ferrocolumbium:											
Electric furnace.....	65.00	60.00	5.00	0.40	8.00	0.03	0.75	2.50	25.00
Aluminothermic.....	68.50	63.00	5.50	.10	.16	1.55	.16	.42	2.00	25.75
Special grade.....	70.60	65.60	5.00	.02	.98	0.29	.02	.20	2.18	28.00
Ferrotantalum-columbium:											
Electric furnace.....	60.00	45.00	15.00	.30	8.00	25.00
Aluminothermic.....	65.00	45.00	20.00	.40	.02	1.50	.60	.10	3.0	29.0
Chromocolumbium.....	60.00	55.00	5.00	.10	7.5010	.90	29.0	2.0
Ferrocromocolumbium.....	20-50	1.0	3-8	20-25	¹
Tantalum-columbium-aluminum.....	40.59	27.76	12.8354	49.85	1.45

¹ Balance is Fe.

high-purity and technical grade, or simply grades 1, 2, and 3, or 98, 99, and 99.5 percent. One company's grades do not necessarily correspond with those sold by another.

High-purity oxide usually is that containing 99.5 percent or more Cb_2O_5 , but some marketed under that designation has contained as low as 99.0 percent Cb_2O_5 . A maximum of 0.2 percent Ta_2O_5 is tolerated. Commercial grade may be a useful term to apply to 99.0 percent Cb_2O_5 . Generally, Ta_2O_5 content is about 0.25 percent but sometimes is as high as 0.5 percent. Technical grade, which usually contains no more than 1.0 percent Ta_2O_5 , may be used to refer to 98.0 percent Cb_2O_5 . Other grades of oxide are marketed containing about 97.0 and 89.0 percent Cb_2O_5 and 2.0 percent and 10.0 percent Ta_2O_5 , respectively. Powders are ground to a variety of particle-size specifications, usually minus-200, or minus 325-mesh, or 0.2 to 5.0 microns.

Tantalum Oxide

Many grades of white tantalum oxide and mixed tantalum-columbium oxides are marketed. For convenience they may be separated into three major classes: High purity (99.5+ percent Ta_2O_5), commercial grade (99.0+ percent Ta_2O_5), and technical grade (98.0+ percent Ta_2O_5). It should be stressed that individual company product nomenclature varies greatly and may not conform to this system. High purity Ta_2O_5 should not contain more than 0.05 percent Cb_2O_5 ; commercial grade, 0.5 percent; and technical grade, 1.0 percent.

Lower grade mixtures of the two oxides are marketed, containing various ratios between the oxides. Examples are H. C. Starck's mixed oxide grades such as No. 2, 88 Ta_2O_5 -11 Cb_2O_5 ; No. 3, 80 Ta_2O_5 -18 Cb_2O_5 ; No. 4a, 72.5 Ta_2O_5 -25 Cb_2O_5 ; and No. 5, 46.5 Ta_2O_5 -46.5 Cb_2O_5 . Particle sizes are the same as for columbium oxide.

Potassium Tantalum Fluoride

Dry potassium tantalum fluoride, the principal soluble salt of tantalum, has a theoretical tantalum content of 46.1 percent. Commercial material, however, contains minor quantities of Cb_2O_5 , SiO_2 , TiO_2 and Fe, which somewhat reduce the Ta content. The usual grain size is 50-mesh and finer.

Columbium Carbide

Several grades of brown columbium carbide are marketed. These may contain from

78 to 88 percent columbium and from 0.1 to 10 percent tantalum. Particle-size ranges mostly from 0.2 to 10 microns.

Tantalum Carbide

Brown tantalum carbide usually contains from 92.3 to 93.5 percent tantalum and not more than 1.0 percent columbium. Other grades marketed include several ranging from 84 tantalum-9 columbium through 45 tantalum-45 columbium. Standard size is minus 200-mesh.

Columbium Boride

Columbium boride, containing 81 to 95 percent columbium depending on grade and formula, may be CbB , CbB_2 , or Cb_2B .

Tantalum Boride

Tantalum boride contains 89 to 94 percent tantalum depending on grade and whether it is TaB or TaB_2 .

Columbium Metal

Grades of columbium vary from company to company. Those grades above 99.0 percent columbium can be classed as high purity (some firms advertise 99.8 or 99.9 percent material). Technical grade has a columbium content ranging from 97.0 to 99.0 percent. High-purity metal should contain not more than 0.1 percent tantalum, standard grade metal less than 0.5 percent tantalum, and technical grade a maximum of 2.0 percent tantalum. Ultrapure columbium for nuclear and other applications is obtainable on special order. For such grades impurities may be specified to 0.000× percent for certain deleterious elements such as boron. Columbium is available as powder, pellets, roundels, rods, sponge, sheet, electrode segments, and ingot.

Tantalum Metal

Tantalum grades have been established longer and are more easily correlated from company to company; 99.9, 99.5, and 98.5 percent tantalum are the most frequent purities offered. Capacitor-grade tantalum is 99.9-percent pure but also must have the necessary electrical properties for capacitor service. Each lot of high-purity tantalum must be tested for capacitance to determine its suitability.

Tantalum sheet is supplied in thicknesses from 0.001 to 0.060 inch. Plate is available in gages up to one-fourth inch thick. Standard foil gages are 0.00025, 0.0005, and

0.00075 inch. Rod is produced in diameters from 0.110 to 1 inch. Wire is sold in diameters from 0.002 to 0.1 inch. Ribbon and welded or seamless tubing are produced in a variety of sizes. In tubing, a great variety of combinations of wall thickness and diameter can be obtained. All the above forms are available annealed or unannealed.

Powder can be obtained in various sizes. The most common are 80- and 325-mesh. Briquets of pressed powder also are available. Roundels, pellets, dendrites, electrode segments, and various sizes of ingots also are sold. Some producers fabricate and market their own capacitor anodes, whole capacitors, or chemical apparatus.

Alloys

Columbium- and tantalum-base alloys are sold as ingot, bar, sheet, and tubing; other forms may be obtained on special order.

USES

Capacitors

Tantalum capacitors are characterized by high capacity in a small volume, stability of the oxide film, long shelf life, tolerance to a wide range of operating temperatures, and low reverse current (back leak). They represent a rapidly expanding use of tantalum (7).

Tantalum capacitor assemblies may be asymmetrical, that is, consisting of one tantalum element, an electrolyte, and an element of a second metal such as silver; or symmetrical, consisting of two tantalum elements in an electrolyte. The electrolyte may be either wet or dry. The tantalum component may consist of foil, one-half mil (0.0005 inch) or less thick; wire; or a porous pellet made by pressing and sintering tantalum powder. The tantalum anode acquires its dielectric property from a thin tenacious oxide film formed by anodic oxidation before or after assembly.

A tantalum capacitor occupies about one-half to one-fifth the space of an aluminum one of equal rating, and certain other advantages also are apparent. The operating range for a tantalum capacitor is from minus 60° to plus 200° C.; for aluminum minus 20° to plus 85° C. Because of its inertness to corrosion by the electrolyte, tantalum has an extremely long shelf life, compared with 2 years for aluminum components. Guided missiles, which may be built many years before being fired, must contain capacitors that will not deteriorate while awaiting service.

Dry-electrolyte capacitors, gaining rapidly in application, use a sintered porous tantalum slug for an anode and manganese dioxide for an electrolyte. This slug permits maximum exposure of the dielectric surface film to the electrolyte, thus providing maximum capacity in minimum volume. The manganese dioxide eliminates corrosive acids from the system and will yield oxygen to repair failures that may occur in the dielectric film.

Vacuum Tubes

Tantalum is preferred as a getter and electron emitter in electronic tubes. When heated to the normal tube operating range of 650° to 1,000° C., tantalum can absorb and hold residual gases contained in the tube, thus enhancing and preserving the vacuum. In addition, it is an excellent thermal emitter of electrons, has a high melting point, and is easy to fabricate.

Columbium, with even more attractive sequestering properties and a lower work function (emissivity), has been relatively little used for these purposes but offers excellent prospects.

Rectifiers

The Balkite rectifier consists of a tantalum strip and an antimonial-lead bar in a sulfuric acid electrolyte. When an alternating current is applied on a cell of this type, current will flow from the lead to the tantalum, but not in the opposite direction, thus converting the alternating current into direct current. These rectifiers are used for low-voltage applications such as railway signals, highway-crossing devices, and battery chargers.

Surge Arresters

In lightning and high-voltage surge arresters, three porous tantalum pellets are immersed in electrolyte. At normal voltage no current passes out of the circuit through the arrester, but a flash of lightning or a sudden surge of extra voltage will pass through the tantalum and be discharged to the ground.

Cryogenics

A new type of miniature electronic valve, the cryotron, which does the work of vacuum tubes or transistors, consists of a tiny strip on tantalum around which a fine columbium wire is wrapped. The combination becomes superconductive when refrigerated in a bath of liquid helium in a unit called a cryostat. Columbium becomes superconductive near 8°

TABLE 8.—*Temperatures at which certain elements become superconductive*

Element:	Transition temperature, ° K
Techneium	11.0
Columbium	8.8
Vanadium	4.89
Tantalum	4.4
Lanthanum	5.3
Rhenium	1.7
Thorium	1.39
Uranium	1.1
Osmium71
Zirconium70
Titanium53
Ruthenium47
Hafnium35

K.; tantalum, at 4.4° K. (table 8). However, should current be introduced into the tiny coil, creation of a magnetic field destroys superconductivity in the straight wire, and resistance is returned. Such a component has a considerably lower power consumption than a thermionic valve, permits the handling of extremely minute currents without power loss, and a hundred such devices will fit into a thimble. Cryotrons offer possibilities for revolutionary computer design and for great improvement in radar devices. Cryotrons offer several advantages: The possibility of designing of 1-cubic-foot computers that do the job of present large-sized ones, greater efficiency in speed and measurement, inexpensive mass-produced computer components, and low power consumption (47).

Steel

Columbium and tantalum have been used as alloying elements in steel primarily to solve the problem of intergranular corrosion. Usually columbium is added to steel in a ratio of 10 times the carbon content. Tantalum, because of its high atomic weight, is only one-half as effective in stainless steel as columbium, on a weight basis. Because the cost of pure columbium and tantalum is high, the elements usually are added to the molten steel as ferrocolumbium (50 to 60 percent columbium) and ferrotantalum-columbium (40 to 45 percent columbium, 15 to 20 percent tantalum). Other steel additive forms suggested as alternatives to ferrocolumbium have been ferrochrome-columbium (20 to 50 percent columbium), and iron-columbium eutectic alloys (21 percent or 73 percent columbium (24)). A series of exothermic mixtures of high-purity columbium oxide with reducing agents has been developed.

These mixtures offer a cheap and effective means of adding columbium to melts, particularly when only part of the melt requires columbium stabilization or when the usual impurities in ferrocolumbium would be undesirable.

About 1 percent columbium is added to some 18-8 type stainless steel to eliminate carbide precipitation in the temperature range 400° to 900° C. Stabilization prevents intergranular embrittlement after exposure to such temperatures and prevents lessening of corrosion resistance on either side of weld seams. High-temperature strength and thermal-fatigue resistance are usually enhanced; subzero temperature impact properties are excellent.

Columbium increases the strength and impact properties and lowers the temperature, at which loss of toughness occurs in low-carbon and low-alloy steel. Columbium, combining readily with nitrogen above 400° C., is a useful constituent with aluminum in nitriding steels. In wrought 2 to 16 percent chromium steels, columbium reduces air-hardening characteristics, prevents brittleness, and improves creep strength (table 9).

Uses of columbium-bearing steel include aircraft engine exhaust systems; welded equipment for the chemical, oil, and food processing industries; structural and corrosion-resistant members in nuclear reactors; piping in reactor heat exchangers; construction equipment; aircraft landing gear; and rocket launching stands (23).

High-Temperature Alloys (49,74)

Columbium is used as an additive to certain steels and complex nonferrous superalloys in quantities up to 5 percent for use at elevated temperatures. Tantalum appears to be an effective substitute for columbium on a weight-for-weight basis in high-temperature alloys. Columbium improves hot strength and hot ductility and imparts resistance to thermal shock at such temperatures. Serviceable columbium-base alloys have not yet been applied widely at high temperatures because of the problem of oxidation and resultant failure of the part. New alloys and cladding techniques appear to have about solved this difficulty. The present low-columbium alloys can be used in jet engines, automotive gas turbines, nuclear-powered aircraft, and rockets for service up to about 1,700° F. The new columbium-base alloys marketed and being developed by Fansteel, DuPont, Union Carbide, Wah Chang, and

TABLE 9.—Typical compositions of some columbium- and tantalum-bearing alloys, percent

Type	Cb (+Ta)	Mo	W	Ti	Mn	C	Cr	Ni	Co	Fe	Si	Other
H. 46	0.15	0.60			0.35	0.15	11.00			86.60	0.40	V 0.75
F. C. B. (T)	1.00					.12	17.50	12.00		69.38		S .03
347	.80				2.00	.08	18.00	10.00		68.05	1.00	P .04
19-9WX	1.30	.50	1.50	.20		.11	20.50	8.50		67.39		
19-9DL	.40	1.25	1.20	.30	1.20	.30	19.00	9.00		66.75	.60	
Croloy 15-15N	1.00	1.50	1.50		2.00	.15	16.00	15.00		61.88	.75	N .15 S .04 P .03
318	.80	2.50			2.50	.08	18.00	14.00		61.09	1.00	S .03 P .04
316Cb	.80	3.00			2.00	.10	18.00	14.00		61.03	1.00	S .03
FBD	2.15	1.75			1.08	.15	17.30	16.00		60.22	1.35	
G. T.-45	.45	2.00		.30	1.25	.08	17.30	14.10		60.12	.50	Cu 3.00 S .03
309S Cb	.80				2.00	.08	23.00	13.50		59.55	1.00	P .04
-Cb	4.00	4.10			.70	.40	15.20	24.60		49.20	1.80	
G18B	3.00	2.00	2.50		.90	.40	13.00	13.00	10.00	53.80	1.40	
S-590	4.43	3.68	4.06		1.31	.44	20.32	19.65	20.20	25.51	.40	
G32	1.40	2.20			.80	.27	19.00	10.50	46.60	15.73	.50	V 3.00
DVL 52	4.88	4.90	4.54		.53	.04	14.90	34.30	25.10	10.32	.40	
G. T. A	4.09	5.09			.74	.19	20.00	32.00	30.00	7.43	.64	
S-816	4.00	4.00	4.00		1.50	.40	20.00	20.00	42.40	3.00	.70	Al .90
Inconel 550	.90			2.40	.70	.04	15.00	72.16		7.00	.40	B .50 Al .45
Inconel-X	1.00			2.50	.70	.05	15.00	73.00		7.00	.30	Al 6.00
Inconel 713C	2.00	4.50		.75	1.00	.20	12.50	63.05		5.00		Al 5.00
I-1360	2.00	5.00				.10	10.00	70.00		4.50		Ba 3.40 Al 6.00 B .30
GE-B-129	2.00	15.00			.40	.06	5.00	32.56		4.00	.40	Zr 8.00
DuPont D-31	180.00	10.00		10.00								Al 8.00
MST 881	1.00			83.00								Al 8.00
MST 821	3.00			89.00								Al 8.00
Cb-65	191.75			7.5								Zr 0.75
Fansteel 80	199.0											Zr 1.00
Fansteel 82	160.0											Zr 1.00
Fansteel 85	233.0 161.0		11.0									Zr 1.00
Union Carbide Cb-7	227.00		23.0	7.0								
Union Carbide Cb-84	165.0	3.0	20.0	7.0								V 5.0
Union Carbide Cb-16	170.0		20.0	10.0								V 3.0
Union Carbide	165.0											Al 3.0 V 3.0
Do	194.0											Al 3.0
Do	187.0			7.0								Al 3.0

¹ Columbium.
² Tantalum.

others may be able to serve up to 2,500° F. The Fansteel series of 80, 82, and 85 alloys exhibit very good high-temperature strength and markedly improved oxidation resistance, may be readily formed, and have excellent weldability.

The columbium may be added as ferro-columbium, or ferrotantalum-columbium, or if it is a low-iron alloy, as chrome-columbium or nickel-columbium (both 60 percent Cb). When low impurity content is essential, columbium metal may be used. An alloy of 90 percent tantalum and 10 percent tungsten has recently been used for rocket nozzles in solid-fuel rocket engines (fig. 1).

Aluminum Casting Alloys

British Standard aluminum casting alloy LM-7 specifies 0.05 to 0.30 percent of columbium plus titanium and LM-8 specifies 0.2 percent columbium plus titanium. Columbium acts as a grain refiner in these alloys for use in cylinder blocks and crank cases.

Tantalum-Tungsten Alloy

An alloy of 92.5 percent tantalum-7.5 percent tungsten maintains its elasticity at high temperatures and is used to manufacture springs for use at elevated temperatures, such as springs or clips used in plating or electro-polishing baths. It also is used in electronic-

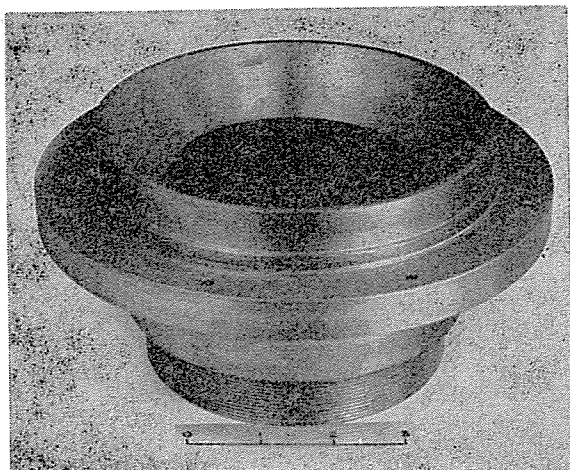


FIGURE 1.—Rocket Nozzle Throat Insert and Retaining Ring Made of 90 percent Tantalum—10 percent Tungsten Alloy.

(Courtesy of Stauffer Metals Co. and Temescal Metallurgical Corp.)

tube components such as filament and grid coil springs where elasticity is required. Tantung-G (28 percent chromium—16 percent tungsten—46 percent cobalt—2 percent carbon—0.2 percent boron—2 percent iron—5 percent tantalum) is used for cutting tool tips and wear-resistant surfaces. (See also High-Temperature Alloys, p. 10.)

Titanium-Aluminum-Columbium-Tantalum Alloy

A new weldable high-temperature titanium alloy contains 8 percent aluminum, 2 percent columbium, and 1 percent tantalum. The addition of columbium and tantalum permits higher tolerance for aluminum in the alloy and results in a medium-strength alloy with good tensile strength and bend ductility up to 1,000° F. (1).

The alloying elements are usually added in the form of a master alloy which contains: tantalum, 12 percent; columbium, 28 percent; and aluminum, 60 percent.

Chemical and Metallurgical Processing Equipment

Because tantalum is inert, weldable, ductile, tolerant to temperature changes, and strong, the metal is especially suitable for fabricating equipment used for handling corrosive chemicals (such as acids, chlorine, bromine, hydrogen peroxide, and petroleum derivatives) and for catalytic cracking equipment employed in the petroleum industry. Because it is resistant to corrosion and is strong, thinner gage metal can be used, per-

mitting higher heat-transfer efficiency. Sheet 0.013 to 0.02 inch thick is used to make piping; reactors; absorbers; vapor condensers; and bayonet, coil, and U-type multitube heat exchangers of all types and sizes. A tantalum condenser may dissipate 120,000 B.t.u. per hour through each square foot of water-cooled surface. Columbium, since its corrosion resistance although good is lower than that of tantalum, has not been used to any great extent for such equipment except when its nuclear properties are important. It may be used as a substitute for tantalum in some of the less corrosive environments. Tantalum metal, tantalum carbide, and tantalum monoboride are used as containers during some metallurgical procedures involving highly corrosive materials such as molten uranium or rare-earth metals. The particular choice depends upon the temperature and corrosive nature of the environment.

Surgical and Dental Uses

Tantalum, because it is resistant to attack by body acids and is compatible with body tissue, is used as wire and foil for suturing nerves and mending broken bones; as sheet and plate for repairing the cranium; and in surgical and dental prosthetics. Surgical and dental instruments made of tantalum, although not in use for many years, have given satisfactory service.

Laboratory Ware

Spatulas, crucibles, crucible liners, and other corrosion- and heat-resistant laboratory equipment are fabricated from tantalum.

Nuclear Applications (14)

Columbium, with a thermal neutron capture cross section of only 1.1 barns, withstands the corrosive action of liquid alkali metal coolants, enhances the corrosion resistance of uranium in aqueous systems at high temperatures, stabilizes the physical characteristics of fuel elements, and causes γ -phase uranium to be retained even after quenching.

Columbium is used in the Experimental Boiling Water Reactor, Argonne, Ill., as a fuel alloying element with zirconium, where it prevents violent reaction between the fuel and boiling water if cladding failure occurs. In the fast reactor constructed at Dounreay, Scotland, columbium is used as a fuel cladding with vanadium. Here, it stabilizes the shape of the elements, resists corrosion and high operating temperature, and prevents escape

of radioactive elements into the reactor system.

As higher operating temperatures become more commonplace in reactor systems, the importance of columbium is expected to increase.

Tantalum, with a neutron capture cross section almost 20 times as high, is less suitable for reactor purposes but might be used for its outstanding corrosion resistance in reactor heat-exchanger systems. Small amounts have been used as a shielding constituent and as the fuel-container lining in the Los Alamos Molten Plutonium Reactor Experiment. Tantalum is being considered for use as the fuel container in a reactor to be fueled with a uranium-bismuth slurry, where resistance to corrosive action of the fuel is of extreme importance. It is also a suitable structural material for the new thermionic cells used experimentally to convert nuclear heat directly to electricity.

Cemented Carbides

Tantalum and columbium carbides can be used separately or blended with tungsten carbide or titanium carbide and cemented with cobalt, nickel, or iron. These carbides are used in hot-forging dies, cutting tools, jet-engine turbine blades, valves, valve seals, and valve guides and also are added to cobalt, chromium and tungsten alloys. Columbium and tantalum carbides increase resistance to wear, shock, and corrosion; reduce the coefficient of friction; and improve the resistance to cratering. The tendency for steel chips to weld to the tool face is also reduced. Carbides possess extremely high melting points (CbC, at 3,900° C.; TaC, at 3,880° C.) and are suited for turbine blades and reciprocating-engine valve parts.

Welding Rods

Columbium-bearing electrodes are used for welding stabilized stainless steels to preclude precipitation of chromium carbide along the joint. Columbium is added to the rods either as a columbium-containing core wire or as a coating in the form of a powdered alloy. A typical weld-metal alloy contains 17.5 to 19.5 percent chromium, 8 to 10 percent nickel, 0.1 percent carbon, 1.0 to 1.3 percent columbium, 0.8 percent silicon, 1.5 percent manganese, 0.04 percent sulfur, 0.04 percent phosphorus and the remainder iron. The weld-metal deposit usually contains less than 1.5 percent

and preferably less than 1.0 percent columbium to minimize crater cracking. A type of rod used in inert-gas welding is formulated of 100 parts tungsten, 15 parts thoria, and 5 parts tantalum.

Optical Glass

Tantalum pentoxide is added to potassium oxide and silica to produce a special optical glass for aerial camera lenses having a high index of refraction and low dispersion. The index of refraction increases directly as tantalum pentoxide content increases.

Synthetic Rubber Catalyst

Butadiene of excellent quality can be made by using a tantalum oxide on silica gel catalyst with ethanol-acetaldehyde feedstock. From 1943 to 1946, this process was used on a large scale in three U.S. plants. The cost of butadiene from this process was about three times that of butadiene obtained by another process using petroleum feedstocks and not requiring a tantalum catalyst. After World War II, because natural rubber was available and the capacity of petroleum butadiene plants had increased, alcohol plants were closed. From time to time, very small special orders of butadiene for export are still filled using the old method.

Radioisotopes

Columbium 95 and tantalum 182 radioisotopes, with half-lives of 35 and 115 days, respectively, are produced at Oak Ridge National Laboratory for use as radioactive tracer elements. They are both important gamma emitters in the radioisotope family.

Miscellaneous Uses

Tantalum also is used in spinnerets for synthetic-fiber production, anode baskets for electroplating devices, and extrusion rings for manufacturing synthetic sausage casings. Certain permanent magnet alloys contain 1 or 2 percent columbium. Tantalum has been used as a resistor material in vacuum furnaces operating above 2,000° C. Tantalum and columbium hydrides are sometimes used as components in metal-to-ceramic sealing compounds. Columbium or tantalum hydride is used for soldering contacts on silicon semiconductors. Iron trioxide plus tantalum pentoxide is a blood coagulant.

CHAPTER 2.—HISTORY

DISCOVERY AND EARLY RESEARCH

In 1784 John Winthrop presented an unidentified heavy black mineral from Connecticut to the Royal Society of London. The specimen passed to the British Museum, where it eventually attracted the attention of Charles Hatchett, an English chemist. In 1801 he discovered a new metallic oxide in the mineral. Hatchett named the metal columbium and the mineral that contained it columbite, in honor of the land where it had been found. A year later, Anders Ekeberg, a Swedish chemist, isolated what he thought was a new element when examining similar specimens from Sweden and Finland. He named the element tantalum after Tantalus of Greek mythology, because it had tantalizingly kept its identity from his reach and because the oxide of the new metal was difficult to dissolve in acids. A period of confusion as to the relationship of the two new elements (some chemists thought they were the same) existed until 1844 when the German chemist, Heinrich Rose, proved the existence of two similar metal oxides in columbite from Bodenmais, Bavaria. One metal he identified as Ekeberg's tantalum, the other, apparently a tantalum-columbium mixture, he called niobium after Niobe, the daughter of Tantalus.

It was not until 1866 that Jean Charles Marignac, the Swiss chemist, definitely separated pure compounds of the two metals and characterized columbium and tantalum as related but distinct elements. To this day, the dual terminology for columbium exists. In the United States, where the largest commercial use exists, metallurgists and industry employ the term "columbium;" chemists of all nations prefer "niobium."

The work of Marignac also resulted in a method of commercial separation of columbium from tantalum, which is still used. This classical method depends upon the difference in solubilities of K_2TaF_7 and K_2CbOF_5 .

Berzelius, Blomstrand, Moissan, and Goldschmidt made impure columbium or tantalum metal in the 19th century. However, the first successful attempt to produce pure tan-

talum was made in Germany by a Polish scientist, Werner von Bolton, in 1903, who reduced K_2TaF_7 by thermal reduction with sodium to produce tantalum powder and buttons and opened the way for its commercial application. In 1907 he produced the first pure columbium by reduction of Cb_2O_5 with aluminum.

EARLY PRODUCTION AND USE

Commercial use of tantalum began in 1903 when the German firm, Siemens-Halske A.G., began production of tantalum wire to replace carbon in incandescent-light filaments. In 1904, the United States began producing tantalite for export to Germany in the Black Hills of South Dakota. In 1906, Siemens-Halske introduced tantalum-tipped penpoints and a short time later, tantalum-containing surgical and dental instruments. However, by 1909 tungsten began to replace tantalum in filaments and by 1912 this substitution was complete. The new industry had died practically at birth, but laboratory interest in the attractive properties of tantalum continued. As early as 1918, experimental quantities of ferrocolumbium and ferrotantalum were produced. In 1921, P. M. McKenna patented a high-speed cutting steel containing 1 to 3 percent tantalum and columbium.

DEVELOPMENT OF PERMANENT USES

Dr. Clarence Balke of Fansteel Metallurgical Corp. placed tantalum on a commercial basis in 1922 by perfecting a practical process to produce ingot from powder. The next year the firm marketed the Balkite charger, which used a tantalum rectifier to convert alternating to direct current for charging batteries that powered radio receiving sets in use at that time. Subsequently, several important patents were issued for using columbium or tantalum in alloys and electrical devices. In 1927, technological advances in radio design largely eliminated the need for Balkite rectifiers, but research and development had begun to find new uses for tantalum.

In the same year Siemens-Halske patented a corrosion-resistant tantalum-nickel alloy, and Bishop and Co., Malvern, Pa., began to manufacture tantalum laboratory ware. By 1929 Fansteel had succeeded in producing columbium rod and sheet. Both elements were beginning to find use in ferroalloys, and tantalum by then was commercially established for use in chemical-laboratory equipment, spinnerets for artificial silk manufacture, jewelry, and radio tubes. The same year the Bureau of Mines recognized tantalum's worth; it found it possible to construct a tantalum-bakelite precision calorimeter for \$50 where a platinum instrument would have cost \$1,500. The use of both elements slowly increased during the 1930's; Fansteel, and later Kennametal, Inc., began producing tantalum carbides for use in cutting-tool bits. In 1933, Electro-Metallurgical Co. (now Union Carbide Metals Co.), studying the effect of columbium in stainless steels, began the first major production of ferrocolumbium.

WORLD WAR II

The beginning of World War II in Europe gave the first great impetus to columbium and tantalum consumption. Coincidentally, Fansteel began making sintered tantalum capacitors in 1940. The advent of radar and the need for military communications greatly expanded the demand for tantalum electronic-tube parts. Columbium, and to a lesser extent tantalum, played a prominent role on both sides in the rapid development of alloys for gas turbines and jet engines. Ore and processing-facility shortages resulted in temporary import and allocation controls and ore stockpiling by the U.S. government. To alleviate the short supply of tantalum metal in 1942 and 1943, the Defense Plant Corporation provided \$5,350,000 to build new tantalum-production facilities next to the Fansteel Metallurgical Corp. in North Chicago, Ill. Tantalite ore shortages were alleviated by flying ore from Africa and South America in military transports. In 1943, tantalum oxide came into use as a catalyst for producing butadiene from alcohol. By the end of the war all controls on tantalum and columbium were revoked as ores and products came into ready supply.

POSTWAR DEVELOPMENTS

After World War II the urgent demand for ore slackened owing to reduced military needs; by 1949 world production of concentrates had been greatly reduced, even though use of columbium and tantalum in civilian applications had remained strong. However, in 1950 beginning hostilities in Korea drastically altered the situation. Industry was not able fully to supply the ferrocolumbium needed for the swiftly expanding jet-engine program. Both ferrocolumbium and ferrotantalum-columbium were placed under controls by the National Production Authority. Uses were restricted until June 30, 1953, and substitution was ordered when possible. To encourage increased production of columbium-tantalum concentrates, the Defense Materials Procurement Agency entered into several ore procurement contracts and on May 29, 1952, announced a Government purchase program for ore containing 15 million pounds of Cb_2O_5 plus Ta_2O_5 . The prices paid under this program were much higher than the previous world prices. This incentive to ore producers had three results: It stimulated ore production, encouraged prospectors who found vast new reserves of columbium (but not tantalum) ore, and also caused consumers to seek substitutes for ferrocolumbium as the price skyrocketed. At the same time military designers attempted to eliminate columbium from their designs because supplies had been so undependable in the past. In May 1955, the U.S. Government announced that deliveries plus forward commitments to buy ore greatly exceeded the objectives and announced cessation of foreign ore purchases. The immediate effect was a confused market in which production and prices of ore began to drift lower. Designers had to be reeducated to the fact that columbium and tantalum ores were readily available, and that the necessity to substitute for them had been removed. Since 1956 industrial demand has improved and military requirements for tantalum capacitors have expanded rapidly. Several new firms entered the field, encouraged by the burgeoning demand for capacitors, by the Atomic Energy Commission (AEC) orders for columbium for use in nuclear reactor research, and by the promise of columbium- or tantalum-bearing high-temperature alloys for jet engines, gas turbines, and missiles.

CHAPTER 3.—RESOURCES

MINERALOGY

Most columbium- and tantalum-bearing minerals are classed as multiple oxides; that is, they are a mixture of oxides of more than one metallic element. The presence of radioactive elements results in a degree of radioactivity in many ores.

The chemical constitution of those oxide minerals containing columbium or tantalum as a major constituent can be expressed by the general chemical formula $A_mB_nO_{2(m+n)}$, where the ratio of m to n is between 1:1 and 1:2, and A = rare earth elements, U, Ca, Th, Fe^2 , Na, Mn, Zr, and B = Cb, Ta, Ti, Sn, W, Zr, Fe^3 . Palache, Berman, and Frondel of Harvard University classified primarily on the AB-O ratio and secondarily according to the A-B ratio (59). Their classification is given in table 10.

One important columbium silicate ore mineral, niocalite $4[(Ca,Cb)_4Si_2(O,OH,F)_2]$ exists.

The titanium or tin minerals in table 11 have been reported to contain columbium as a substantial minor constituent, replacing tin or titanium in the crystal lattice.

Other ore minerals that may contain columbium are albramite (tungsten ore) and lepidolite (an ore of lithium). The minerals, mined for their columbium-tantalum content or considered to be possible future sources, are pyrochlore-microlite, fergusonite, yttrotantalite, columbite-tantalite, euxenite-polycrase, samarskite, betafite, ilmenite, rutile, cassiterite, anatase, brookite, perovskite, fersmite, and niocalite.

Pyrochlore-Microlite Series

The mineral is generally brown or black, has a hardness of 5 to $5\frac{1}{2}$, and a specific gravity of 4.2 to 6.4, increasing with tantalum content. Pyrochlore is the columbium-rich end member of the series; microlite is the tantalum-rich end member. Columbium and tantalum vary continuously throughout the series, but most analyses are close to one end or the other. The rare-earth elements are

most common at the pyrochlore end; (Cb, Ta) $_2O_5$ ranges from 38.54 percent to 82.14 percent. Pyrochlore typically occurs associated with alkalic rocks in pegmatites, nepheline syenite, various alkalic dike rocks, carbonatites associated with alkalic intrusives, extrusive alkalic rocks, greisen, and in decomposition products of these rocks. Microlite typically occurs in the albitized parts of granite pegmatites, frequently with associated columbite or tantalite (table 12).

Intermediate members of the series and special varieties are pyrrhite, koppite, hatchettolite, chalcolamprite, endeiolite, margnacite, ellsworthite, neotantalite, and metasimpsonite.

Scheteligite

This black mineral, with a hardness of 5.5 and a specific gravity of 4.74, occurs in a pegmatite at Torvelona, Norway, associated with plagioclase, tourmaline, native bismuth, euxenite, thortveitite, monazite, alvite, beryl, garnet, and magnetite. An analysis gave 20.00 percent Ta $_2O_5$ and 8.65 percent Cb $_2O_5$.

Fergusonite Series

The mineral may be black, brown, gray, or yellow and has a hardness of 5.5 to 6.5 and a specific gravity of 5.6 to 5.8, increasing with tantalum content. A complete isomorphous series exists between fergusonite, the predominantly columbium member, and formanite, the tantalum-rich end member. The reported content of (Cb,Ta) $_2O_5$ ranges from 40.21 to 66.17 percent. Fergusonite minerals occur in granite pegmatites, particularly in those rich in rare-earth elements, columbium, tantalum, and beryllium, and in placers derived from such rock. Special varieties are rutherfordite, sipylite, and risorite (table 13).

Yttrotantalite

Black or brown in color, yttrotantalite has a hardness of 5 to 5.5 and a specific gravity of 5.7 ± 0.2 . Analyses indicate a (Cb,Ta) $_2O_5$ content ranging from 55 to 60 percent. The

TABLE 10.—Classification of columbium-tantalum minerals (59)

81—ABX₄TYPE

Classification No.	Minerals	A	B
811.....	Pyrochlore-microlite, series A ₂ B ₂ O ₆ (O, OH, F):		
8111.....	Pyrochlore.....	Na, Ca, K, Mg, Fe, Mn, Ce, La, Di, Er, Y, Th, Zr, U	{Cb, Ta, Ti, Sn, Fe ³ , W
8112.....	Microlite.....		{Ta, Cb, Ti, Sn, Fe ³ , W
812.....	Scheteligite ?A ₂ B ₂ (O, OH) ₇	Ca, Y, Sb, Mn.....	Ti, Ta, Cb
813.....	Fergusonite series ABO ₄ :		
8131.....	Fergusonite.....	Y, Er, (Ce, La, Di), Fe ² , U, Zr, Th, Ca	{Cb, Ta, Ti, Sn, W
8132.....	Formanite.....		{Ta, Cb, Ti, Sn, W
814.....	Yttrotantalite ABO ₄	Fe ² , Y, U, Ca, Mn, Ce, Th	Cb, Ta, Ti, Zr, Sn
815.....	Polymignite ABO ₄	Ca, Fe ² , (Y, Er, Ce), Zr, Th	Cb, Ti, Ta, Fe ³
816.....	Ishikawaite ABO ₄	U, Fe ² , (Y, Er, Ce).....	Cb, Ta
817.....	Loranskite.....	Y, Ce, Ca, Zr.....	Ta, Zr
818.....	Stibiotantalite series ABO ₄ :		
8181.....	Stibiotantalite.....	Sb, Bi.....	Ta, Cb
8182.....	Stibiocolumbite.....	Sb, Bi.....	Cb, Ta
819.....	Bismutotantalite.....	Bi.....	Ta, Cb
81.10.....	Simpsonite ABO ₄	Al.....	Ta

83—AB₂X₆TYPE

831.....	Tapiolite series AB ₂ O ₆ :		
8311.....	Tapiolite.....	Fe, Mn.....	Ta, Cb
8312.....	Mossite.....	Fe, Mn.....	Cb, Ta
832.....	Columbite-Tantalite series:		
8321.....	Columbite.....	Fe, Mn, Sn.....	Cb, Ta, W
8322.....	Tantalite.....	Fe, Mn.....	Ta, Cb
833.....	Euxenite-Polycrase series:		
8331.....	Euxenite.....	Y, Ca, Ce, U, Th.....	Cb, Ta, Ti
8332.....	Polycrase.....	Y, Ca, Ce, U, Th.....	Ti, Cb, Ta, Fe ³
	Eschwegeite.....	(Y, Er), U, Th.....	Cb, Ta, Ti, Fe ³
	Fersmite.....	Ca, (Ce, La), Na.....	Cb, Ti, Fe, Al
835.....	Eschynite-priorite series:		
8351.....	Eschymite.....	Ce, Ca, Fe ² , Th.....	Ti, Cb, Ta
8352.....	Priorite.....	Y, Er, Ca, Fe, Th.....	Ti, Cb, Ta
8361.....	Samarskite.....	Y, Er, Ce, La, U, Ca, Fe, Pb, Th	Cb, Ta, Ti, Sn, W, Zr
8362.....	"Samarskite" ¹	Ca, Pb, Y, U.....	Cb, Ta, Ti, Fe ³
837.....	Thoreaulite AB ₂ O ₇	Sn.....	Ta

84—AmBnX_pTYPE²

841.....	Betafite.....	U, Ca, Th, Pb, Ce, Y....	Ti, Cb, Ta, Fe, Al
842.....	Djalmaite.....	U, Ca, Pb, Bi, Fe.....	Ta, Cb, Ti, Zr
843.....	Ampangabeite.....	Y, Er, U, Ca, Th.....	Cb, Ta, Fe ³ , Ti

¹ A different species similar to true samarskite.

² Where m:n = <1:3.

TABLE 11.—Titanium or tin minerals containing columbium (59)

44—A ₂ X ₃ TYPE		
Classification No.	Mineral	Formula
4413.....	Ilmenite.....	FeTiO ₃
45—AX ₂ TYPE		
4511.....	Rutile (including strueverite).....	TiO ₂
4515.....	Cassiterite.....	SnO ₂
452.....	Anatase.....	TiO ₂
453.....	Brookite.....	TiO ₂
74—ABX ₃ TYPE		
7421.....	Perovskite.....	CaTiO ₃
	Dysanalite ¹	Ca(Ti,Cb)O ₃
	Loparite ¹	Ca(Ti,Cb)O ₃
	Knopite ¹	(Ce,Ca)(Ti,Cb)O ₃

SILICATES

Sphene.....	CaO•(Ti,Cb)O ₂ •SiO ₂
Fersmannite.....	2Na ₂ (O,F) ₂ •4CaO•4(Ti,Cb)O ₂ •3SiO ₂

¹ Variety of perovskite.

TABLE 12.—Pyrochlore-microlite analyses, percent (59)

	Pyrochlore ¹	Koppite ²	Hatchettolite ³	Microlite ⁴	Microlite ⁵
Na ₂ O.....	4.99	2.89	2.86	1.18
K ₂ O.....	.60	1.6429	.15
CaO.....	18.13	15.88	13.25	11.80	12.78
MnO.....01	.5111
FeO.....	1.1447
MgO.....27	.36	1.01
Y ₂ O ₃62	.23
Er ₂ O ₃17
Ce ₂ O ₃	4.36	8.15
Di ₂ O ₃	1.68	.12
La ₂ O ₃
UO ₂	11.40
UO ₃	4.41	1.59
ZrO ₂	4.90	.61	4.12	1.05
SnO ₂	1.4437
ThO ₂	1.52
SiO ₂	1.5740
Fe ₂ O ₃	9.73	3.46	.29	.72
TiO ₂75	11.3751
Cb ₂ O ₅	63.64	56.43	31.33	7.74	3.64
Ta ₂ O ₅15	10.29	68.43	77.00
F.....	4.31	1.53	2.85	1.09
H ₂ O.....	.47	1.09	4.29	1.17	2.00
Remainder.....54	.77	.55
Total.....	102.54	100.81	99.60	100.25	100.97

¹ Alno, Sweden.² Kaiserstuhl, Germany.³ Hybla, Ontario.⁴ Amelia Court House, Va.⁵ Wodgina, Australia.

TABLE 13.—Fergusonite-formanite analyses, percent (59)

	Fergusonite ¹	Siplyite ²	Risorite ³	Formanite ⁴
CaO.....	1.40	2.61	1.93	2.18
MnO.....	(⁵)87
MgO.....	.58	.05
FeO.....	2.04	2.61	(⁵)
Y ₂ O ₃	23.00
Er ₂ O ₃	40.39	31.36	36.28	8.38
Ce ₂ O ₃	1.37
La ₂ O ₃89	3.92	2.88	.94
Di ₂ O ₃	4.06
UO ₂	3.1810
UO ₃	1.18
ThO ₂	(⁵)	1.02
ZrO ₂	2.09
SnO ₂35	.08	.01
Al ₂ O ₃	1.3581
Fe ₂ O ₃66	1.20
TiO ₂	1.15	6.00	2.20
Cb ₂ O ₅	44.97	46.66	36.21	2.15
Ta ₂ O ₅	2.00	4.00	55.51
H ₂ O.....	3.92	3.19	7.11
Remainder.....	1.14	4.47	1.33	3.36
Total.....	99.98	100.48	100.47	100.79

¹ Hakatamura, Japan.² Amherst Co., Va.³ Risor, Norway.⁴ Cooglegong, Western Australia.⁵ Trace.

ratios of the principal constituents are Fe:Y:U=9:8:1 and Cb=Ta ratio is approximately 1:1. The mineral is found in pegmatites.

Polymignite

Usually black, this mineral has a hardness of 6.5 and a specific gravity of 4.77 to 4.85. Columbium and tantalum pentoxide content ranges from 13.34 to 48.54 percent in alkalic pegmatites, granite pegmatites, and syenite.

Ishikawaite

Ishikawaite is black and has a hardness of 5 to 6 and a specific gravity of 6.2 to 6.4. An analysis gave Cb₂O₅ as 36.80 percent and Ta₂O₅ as 15.00 percent. It was found with samarskite in a pegmatite in Iwaki Province, Japan.

Loranskite

The black mineral has a hardness of 5 and a specific gravity of 4.16 to 4.6 and reportedly contains 47.00 percent Ta₂O₅; it was found in a pegmatite near Lake Ladoga, Finland.

Stibiotantalite Series

The series varies in color through different shades of brown, red and yellow and has a hardness of 5.5 and a specific gravity of 5.98 to 7.34, increasing with tantalum content. A complete isomorphous series from stibio-columbite to stibiotantalite exists. The

(Cb,Ta)₂O₅ content may range from 47.69 to 60.24 percent. It occurs typically with columbite, tantalite, or microlite in pegmatites, and also is found in cassiterite placers.

Bismutotantalite

The mineral is pitch black when unaltered and has a hardness of 5 and a specific gravity of 8.28. (Cb,Ta)₂O₅ content may range from 45.90 to 48.67 percent. It occurs in pegmatites.

Simpsonite

This colorless mineral, with hardness not reported and specific gravity of 5.92 to 6.27, ranges in Cb₂O₅ plus Ta₂O₅ content from 71.80 to 81.25 percent. The mineral occurs in a pegmatite at Tabba Tabba, Western Australia.

Tapiolite Series

The members of this series are black; hardness is 6 to 6.5 and specific gravity is 7.90 ±0.05, increasing with tantalum content. Tapiolite is the tantalum end of the series; mossite, the probable columbium end. The (Cb,Ta)₂O₅ content ranges from 79.86 to 86.01 percent. Tapiolite minerals occur in granite pegmatites and as a detrital mineral in areas of granite pegmatites. Special varieties include ixiolite.

Columbite-Tantalite Series

These most important ore minerals are black to brownish black with a hardness of 6 to 6.5 and a specific gravity ranging from 5.19 to about 8.00, increasing almost in direct proportion to increased tantalum content. A complete gradational series exists between columbite and tantalite. (Cb,Ta)₂O₅ ranges from 75.10 to 86.12 percent (table 14).

Minerals of the series are abundant and widespread. They occur as accessory minerals in granite; in granite pegmatites, particularly those with albite or lithium minerals; and in derived detrital deposits. Marked variation in the ratios of columbium to tantalum and iron to manganese is often found in material from a single locality and even in a single specimen or crystal.

Special varieties are ferrocolumbite, mangancolumbite, ferrotantalite, and manganotantalite.

Euxenite-Polycrase Series

The black minerals forming this series have a hardness of 5.5 to 6.5 with a specific gravity of 5.0 to 5.9. The series grades from a high

TABLE 14.—Columbite-tantalite analyses, percent (59)

	Columbite 1	Columbite 2	Tantalite 3	Manganon tantalite 4	Ferro tantalite 5
MnO.....	5.97	14.79	5.66	14.15	1.20
FeO.....	15.04	5.45	11.91	1.63	14.30
SnO ₂67	.88	.44	.48	.82
WO ₃	72.37	68.00	27.22	15.11	13.14
Cb ₂ O ₅	5.26	9.88	53.47	68.65	70.53
Ta ₂ O ₅58	.53	1.30	.55
Remainder.....
Total.....	99.89	99.53	100.00	100.57	99.99

- 1 Annerod, Norway.
- 2 Old Mike Mine, Custer Co., S. Dak.
- 3 Tin Mountain, Custer Co., S. Dak.
- 4 Wodgina district, Western Australia.
- 5 Rosendal, Kimito Parish, Finland.

columbium plus tantalum member, euxenite, to a high titanium member, polycrase; (Cb, Ta)₂O₅ contents ranging from 19.48 to 51.14 percent (table 15) have been reported. Minerals of this series are found in granite pegmatites and as a detrital mineral in areas of granitic rocks. They sometimes occur in close association with columbite and monazite. Special varieties are tanteuxenite (tantalum euxenite), tantalian polycrase, and lyndochite (euxenite).

TABLE 15.—Euxenite-polycrase analyses, percent (59)

	Tanteuxenite 1	Euxenite 2	Polycrase 3	Tantalian polycrase 4	Euxenite 5
CaO.....	2.22	1.92	1.02
MgO.....0335
MnO.....	.35	.2834
PbO.....	(6)	1.35
FeO.....	2.87	(6)
Al ₂ O ₃2676
(Ce, etc.) ₂ O ₃	7.22	.44	3.55	27
(Y, Er) ₂ O ₃	17.48	24.31	27.55	25.03
UO ₂	8.61
UO ₃	3.35	.20	13.77	6.69
U ₃ O ₈	10
ThO ₂	(6)	3.94	1.76	3
SnO ₂14	.07
SiO ₂90	.09
Fe ₂ O ₃	1.18	2.07	7
TiO ₂	14.17	22.96	29.31	30.43	20
Cb ₂ O ₅	3.83	28.62	19.48	4.35	27
Ta ₂ O ₅	47.31	2.65	23.10	3
H ₂ O.....	2.23	5.18
Remainder.....	2.40	.26	2.82	3
Total.....	100.55	100.29	98.16	100.20	100

- 1 Cooglegong, Western Australia.
- 2 Sabine township, Nipissing district, Ontario.
- 3 Henderson County, N.C.
- 4 Cooglegong, Western Australia.
- 5 Bear Valley, Idaho: Average of several analyses by Bureau of Mines, Albany, Oreg.
- 6 Trace.

Fersmite

Fersmite is a black to dark brown mineral with a hardness of 4 to 4.5 and a specific gravity of 4.69 to 4.79. Its Cb_2O_5 content ranges from 70.12 to 74.44 percent, and Ta_2O_5 is either absent or present only in trace amounts. Fersmite has been found in pegmatites in the Ural Mountains and as inclusions and intergrowths with a tantalum-free columbite in carbonate rocks in Ravalli County, Mont. (28).

Eschynite-Priorite Series

Members are various shades of black, brown, or yellow and have a hardness of 5 to 6 and a specific gravity of 4.95 to 5.19, increasing from priorite to eschynite. Priorite is the cerium-rich end member; eschynite is higher in yttrium and erbium. The content of columbium and tantalum pentoxides ranges from 16.38 to 36.68 percent. Eschynite is found most often in nepheline syenite with zircon and samarskite. Priorite occurs in granite pegmatites with euxenite, zircon, monazite, and other rare-earth minerals. Both minerals occur in placers.

Samarskite

Samarskite is black or brownish black and has a hardness of 5 to 6 and a specific gravity of about 5.69. However, depending upon the degree of alteration, the specific gravity can be as low as 4.1 or, in the presence of titanium, as high as 6.2. The content of Cb_2O_5 ranges from 42.30 to 60.68 percent (table 16). Samarskite is found in granite pegmatites often in close association with columbite, and in derived detrital deposits.

Related or altered varieties are vietinghofite, rogersite, plumboniobite, hydrosamar-skite, wiikite, nuolaite, and "samarskite?" (from Petaca, N.M.).

Thoreaulite

Brown in color, thoreaulite has a hardness of 6 and a specific gravity of 7.6 to 7.9. Cb_2O_5 is present only in trace amounts. Ta_2O_5 has been reported from 72.83 to 77.59 percent. The mineral occurs with cassiterite in a pegmatite at Manono, Katanga Province, Congo.

Betafite Series

The members of the betafite series are shades of black, brown, or yellow, with a hardness of 4.0 to 5.5 and a specific gravity of 3.7 to 5.0. Combined columbium and tan-

TABLE 16.—*Samarskite analyses, percent (59)*

	Samarskite ¹	Samarskite ²	Samarskite ³
CaO.....	0.55	2.43	0.37
MgO.....			.09
FeO.....	10.75	5.40	
MnO.....	.78		.79
PbO.....			.51
Y ₂ O ₅	14.49	9.50	
Er ₂ O ₃			14.10
Ce ₂ O ₃		4.05	
La ₂ O ₃			
Di ₂ O ₃	4.17	8.70	
UO ₂			17.20
UO ₃	12.55		
SnO ₂08		
ThO ₂		1.05	3.03
TiO ₂		1.42	
Fe ₂ O ₃			10.18
Al ₂ O ₃80	
Cb ₂ O ₅	37.51	43.60	51.38
Ta ₂ O ₅	18.20	11.15	
H ₂ O.....	1.12	11.14	1.55
Remainder.....			.09
Total.....	100.20	99.24	99.29

¹ Mitchell County, N.C.

² Antanamalaya, Madagascar.

³ Topsham, Me.

talium pentoxide content ranges from 17.72 to 51.80 percent (table 17). Minerals of the series are found typically with euxenite, fergusonite, allanite, metamict zircon, or beryl in granite pegmatites and in detrital deposits. Special varieties are titanium betafite, samiresite, blomstrandite, and mendeleevite.

Djalmaite

This yellow-brown to black mineral has a hardness of 5.5 and a specific gravity of 5.75 to 5.88. An analysis gave Cb_2O_5 , 1.41 percent, and Ta_2O_5 , 72.27 percent. Djalmaite was found in a granite pegmatite on Posse farm, Conceicao County, Minas Gerais, Bra-

TABLE 17.—*Betafite analyses, percent (59)*

	Samiresite ¹	Titanian betafite ²	Titanian betafite ²
CaO.....		8.96	7.02
PbO.....	7.35	1.70	1.42
FeO.....	1.06		
(Y, Er) ₂ O ₃			3.30
(Ce, La) ₂ O ₃20		
UO ₂	21.20		
UO ₃		15.52	20.20
SnO ₂10		
ThO ₂04
SiO ₂62	.64
Al ₂ O ₃74	.98	.36
Fe ₂ O ₃		5.52	6.96
TiO ₂	6.70	35.05	34.22
Cb ₂ O ₅	45.80	8.51	10.11
Ta ₂ O ₅	3.70	12.85	7.61
H ₂ O.....	12.45	9.63	7.45
Total.....	99.60	99.34	99.33

¹ Samiresy, Madagascar.

² Both samples from Tangen, Norway.

zil. It may possibly represent the tantalum equivalent of betafite.

Ampangabeite

Ampangabeite is various shades of brown, and has a hardness of 4.0 and a specific gravity of 3.36 to 4.64. The Cb_2O_5 plus Ta_2O_5 content ranges from 43.70 to 52.58 percent, with Cb_2O_5 predominant, in potash-rich pegmatites.

Niocalite

Niocalite occurs in carbonatite at Oka, Quebec, Canada, as prismatic crystals, comprising up to 10 percent of the rock. Chemical analyses show 16 to 19 percent Cb_2O_5 . The mineral is chemically and crystallographically similar to the woehlerite group.

Other Minerals

Many titanium minerals contain appreciable columbium. Fleischer, Murata, Fletcher and Narten (19) listed the columbium content for many titanium-bearing minerals as follows:

Mineral:	Columbium, Percent
Ilmenite	0.00002- 0.9
Rutile004 -23.3
Anatase032 - 1.8
Brookite01 - 2.4
Perovskite (includes dysanalite, loparite, and knopite)006 -18.2
Sphene (includes keilhauite).....	.0001 - 2.3

These minerals are of domestic importance in Arkansas where high columbium values are reported in titanium deposits and titanium-bearing bauxites.

Columbium also has been reported in a number of other minerals. Wolframite has been reported to contain up to 1.2 percent Cb. Columbium or tantalum also have been reported to occur in cassiterite, fersmannite, uranoniobite (uraninite with Cb_2O_5), epistolite, eucolite, woehlerite, lavenite, guarinite, rosenbuschite, graphite, sphalerite, fluorite, titaniferous magnetite, chromite, vredenburghite, martite, "ainalite," pyrolusite, "ixionolite," nitratite, calcite, anhydrite, alunite, huebnerite, monazite, triplite, amblygonite, pyromorphite, topaz, zircon, thorite, chevkinite, verdelite, schorlrite, astrophyllite, eudialite, catapleite, diopside, aegerite, hyperssthene, lamprobolite, muscovite, phlogopite, biotite, and nepheline.

GEOCHEMISTRY (66,67)

Columbium and tantalum are a coherent pair of elements, similar in this respect to

zirconium-hafnium and the elements of the lanthanide series. Because of their similarity they commonly, but not always, occur together in nature, and as might be expected they are difficult to separate and analyse. In the earth's crust they are midway in abundance between zirconium and gold. Their ratio has been estimated at 11.4 parts columbium to 1 part tantalum, but in a given occurrence either metal may be predominant. Because large columbium deposits have recently been discovered, a recalculation of this ratio probably would prove the preponderance of columbium over tantalum much greater. These strongly oxyphile elements (their behavior as metal is analogous in the way they readily absorb oxygen when heated) occur only rarely as sulfides or silicates. The occurrence of tantalum and columbium, lithophile and apparently strongly enriched in the upper lithosphere, as native metals, while reported, has never been verified.

TABLE 18.—Content of columbium and tantalum and columbium-tantalum ratio in igneous rocks (67)

Rock type	Columbium, weight-percent	Tantalum, weight-percent	Columbium-tantalum ratio
Monomineralic.....	0.00003	0.00008	0.4:1
Ultrabasic.....	.0016	.0012	16.0:1
Ecolgites.....	.0003	.0007	4.3:1
Gabbros.....	.0019	.0012	17.3:1
Diorites.....	.00036	.0007	5.1:1
Granite.....	.002	.0042	4.8:1
Syenites.....	.0035	.002	15.0:1
Nepheline syenite...	.31	.0008	357.5:1
Basic alkalie.....	.0010	.0012	8.3:1

In igneous rocks (table 18) where the average content has been estimated to be 0.0024 percent columbium and 0.00021 percent tantalum (generally accepted as reflecting their content in the entire crust), their occurrence has been studied considerably. Both elements have been concentrated in late crystallates during magmatic differentiation. Both are accessory constituents in granites where the maximum tantalum concentration occurs. Columbium reaches its peak concentration in syenites and nepheline syenites. Some columbium has been noted, however, in more basic rocks, where NaCb, removed from the magma in a comparatively early stage, has replaced CaTi.

Columbium and tantalum substitute in crystal lattices for, and are true geochemical satellites of, titanium or zirconium because of their similar ionic size and valence. Less important replacements occur with tin, tungsten, chromium, and manganese. Replacement

is theoretically plausible with molybdenum. Atomic radii of iron and manganese are similar, but because of marked valence differences, columbium and tantalum do not compound with iron or manganese during early magmatic crystallization (table 19).

TABLE 19.—*Ionic radii of certain ions, in Angstrom units*

Element	Ion	Ion radius
Columbium.....	5+	0.69
Tantalum.....	5+	.68
Titanium.....	3+	.69
Do.....	4+	.64
Zirconium.....	4+	.79
Tin.....	4+	.71
Tungsten.....	6+	.62
Do.....	4+	.68
Chromium.....	3+	.64
Manganese.....	3+	.70
Molybdenum.....	4+	.66
Do.....	6+	.62
Iron.....	2+	.75
Magnesium.....	2+	.65

In nature columbium and tantalum occur often in the same minerals with zirconium, titanium, tin or tungsten or in close association with minerals containing them. Columbium-tantalum minerals form isomorphous series with minerals in which zirconium, titanium, tin, or tungsten are primary constituents. Both elements are characteristic of granite pegmatites (columbium is characteristic of nepheline syenite pegmatites) and in these occurrences are often in close association with rare-earth elements. Many of the most characteristic rare-earth minerals, structurally, are mixed oxides containing columbium and tantalum and usually titanium or zirconium as well. Antimony and bismuth also are fairly frequent associates. Silicates containing columbium or tantalum as major constituents are rare because Cb^{5+} and Ta^{5+} ions do not readily form electrically neutralized structures of sufficient stability for development of silicates.

Some enrichment of columbium and tantalum appears in pneumatolytic formations. The elements occur in greisens with tin, tungsten, and sometimes uranium. Geochemical conclusions have not been drawn for columbium and tantalum in metamorphic rocks, but a typical mica gneiss is reported to average 0.001 percent Cb_2O_5 .

During exogenic differentiation most columbium and tantalum, being contained in

heavy minerals, will concentrate in residual or placer deposits. Bauxites derived from residual weathering of syenites contain columbium, some of which is in solid solution in titanium and zirconium minerals and some reprecipitated from alkali columbates dissolved from the primary host rock. Most of the dissolved and transported columbium and tantalum is precipitated in hydrolysates (clay minerals at an early stage). Minor quantities remain at a very low concentration in sea water or are deposited in marine carbonate beds, evaporates, or deep sea manganese nodules (table 20).

TABLE 20.—*Content of columbium and tantalum and columbium-tantalum ratio in sediments (67)*

Rock group, sediments	Average Cb_2O_5 , percent	Average Ta_2O_5 , percent	Columbium-tantalum ratio
Minerogenic.....	0.0018	0.00017	9.1:1
Chemical.....	.00006	.00002	2.6:1
Organogenic.....	.00009	.00002	3.8:1
Deep sea.....	.0059	.00012	49.2:1

ECONOMIC GEOLOGY

The general mineralogical associations and types of occurrence are controlled by factors discussed in the preceding section on geochemistry. As noted, columbium has an affinity for granitic and alkalic rocks; tantalum, an affinity for granitic rocks. Even within these environments large deposits generally depend on special conditions. Undecomposed granites or syenites rarely contain potentially economic quantities of columbium or tantalum. These concentrations occur only where weathering has led to residual or placer concentrations of the ore minerals or where the rocks have associated pegmatites or carbonatites (carbonate-silicate rocks), in which minerals containing rare elements have crystallized in greater abundance. Even in these concentrations, the columbium and tantalum content alone usually is too small for economic recovery, and their geochemically related elements must be recovered at the same time. The principal geologic types of occurrence discussed below are

1. Carbonatites and associated rocks.
2. Granites and granite pegmatites.
3. Syenites and syenite pegmatites.
4. Deposits in bauxite, alluvium, and eluvium.

Carbonatites and Associated Rocks (60)

Carbonatites, in 1960, were of great interest because of their tremendous potential economic importance. They commonly occur in alkaline rock complexes characterized by the presence of silica-deficient sodium-rich rocks such as syenite and nepheline syenite. In some complexes the carbonatites are the most abundant rocks exposed. Usually, surrounding country rocks such as gneiss have been fenitized; that is, metasomatically altered to alkaline rocks. All these rocks may contain large potential sources of columbium included in complex oxide minerals with rare earths, uranium, thorium, and titanium. The principal minerals are pyrochlore, columbian perovskite, betafite, and niocalite. Apatite, an ore of phosphate, is a common associate.

Ring structures are common in carbonatite areas. Carbonatite dikes and veins also occur, and groups of deposits often are aligned in belts. Examples are the deposits associated with the east-west trending Monterigian area of Quebec and its westward extension into Ontario and the deposits associated with East African rift valleys. The ring structure may vary greatly in detail but the general rock succession can be typified by the deposit at Nemegosenda Lake,

Ontario. The core is syenite surrounded by concentric rings of syenitic contact rocks and breccias, carbonatite (calcite-feldspar) breccias, pyroxenitic fenites with or without magnetite, garnet, and wollastonite, alkalic fenitized gneiss, and gneiss. Considerable controversy exists among geologists concerning the origin of carbonatites (60), some geologists consider them to be metasomatized limestones; others believe they are of true magmatic origin. Other geologists are less dogmatic and refer their origin to carbonate-rich solutions rather than carbonate magma. Probably all three processes have played a role in different deposits or in parts of the same deposit. In fact, rock formed from a sedimentary limestone, which had been melted or dissolved and had become mobile, might be virtually impossible to differentiate from an abyssal carbonate magma or from carbonate-rich end liquors from magmatic differentiation.

Important carbonatite bodies reported to date are listed in table 21.

The unique mineral assemblages and structure in carbonatite-bearing alkalic areas permit relatively easy prospecting. Most of the known bodies are abnormally radioactive and were discovered by using beta-gamma (Geiger-Mueller) counters or scintillation

TABLE 21.—Location of carbonatite rocks of the world

Location	Deposit Name	Columbium minerals	X=interest for columbium, 1960
North America:			
Canada:			
British Columbia	Granite Creek	Pyrochlore, microlite, columbite, columbian rutile.	X
Do.	Lemprière	Pyrochlore	X
Ontario:			
Baneroft.	Basin deposit	Betafite	X
Chapleau	Nemegosenda Lake	Pyrochlore	X
Do.	Nemegos.	Do	X
Hastings County	Faraday	Betafite	X
Lake Nipissing	Newman deposit	Pyrochlore	X
Quebec: Oka	Oka Hills	Pyrochlore, columbian perovskite, niocalite, and betafite.	X
United States:			
Arkansas: Hot Springs County	Magnet Cove	Columbian perovskite, columbian rutile.	X
California: San Bernardino County	Mountain Pass	Not identified (columbium reported in specific analysis).	
Colorado: Gunnison County	Iron Hill (Powderhorn)	Columbian perovskite and pyrochlore.	X
Idaho: Lemhi County	Mineral Hill District	Columbian rutile.	
Montana:			
Ravalli County	Dark Star	Columbite and fersmite.	X
Hill County	Big Sandy Creek	Pyrochlore	X
South America:			
Brazil:			
Goiás	Catalao	Pyrochlore	X
Minas Gerais	Araxá	Do	X
Do.	Tapira	Do	X
Do.	Serra Negra—Patrocínio	Do	X
Do.	Salitre		
Do.	Pocos de Caldas		
Santa Catarina	Anitapolis		
São Paulo	Jacupiranga	Pyrochlore	
Do.	Ipanema		
Do.	Juquia-Registro		

TABLE 21.—*Location of carbonatite rock of the world—Continued*

Location	Deposit Name	Columbium minerals	X=interest for columbium, 1960
Europe:			
Finland: East-central.....	Kuusamo.....		
Germany: Rhine Valley.....	Kaiserstuhl.....	Pyrochlore, columbian perovskite.....	X
Norway: Ulefoss, Telemark.....	Sove.....	Do.....	X
Sweden: Klingarfjarden Bay.....	Alno.....	Do.....	
U.S.S.R.:			
Kola Peninsula.....	Khibine and Lovozero.....	Pyrochlore, and columbian perovskite (var. loparite).....	X
Ukraine.....	Zhdanov.....		
Asia:			
U.S.S.R.:			
North-west Siberia.....			
South-central Siberia.....	Sayan Mountains.....		
Yakut Republic.....	Aldan River.....		
Africa:			
Congo, Republic of the: Kivu.....	Lueshe Valley.....	Pyrochlore.....	X
Kenya:			
Homa Mountain.....	Homa.....		
Kisingiri Mountain.....	Rangwe.....		
Mrima Hill.....	Mrima.....	Pyrochlore.....	X
Ruri Mountain.....	Ruri.....		
Mozambique: Lupata Gorge.....	Muambe.....		
Rhodesia and Nyasaland, Federation of:			
Nyasaland:			
Lake Chilwa.....	Chilwa Island.....	Do.....	X
Do.....	Tundulu.....	Do.....	
Do.....	Nkalonje Hill.....	Do.....	
Do.....	Songwe.....	Do.....	
Do.....	Ilonga Hill.....	Do.....	X
Shire Valley.....	Kangankude.....		
Northern Rhodesia:			
Feira District.....	Feira.....	Pyrochlore.....	
Nkumbwa Hill.....	Nkumbwa.....	Do.....	X
Southern Rhodesia:			
Sabi Valley.....	Shawa.....		
Do.....	Doriowa.....		
Do.....	Chishanya.....		
Tanganyika:			
Kisaki.....	Wigu Hill.....		
Lake Rukwa.....	Oldonyo Dili.....		
Panda Hill.....	Sengeri Hill.....		
Do.....	Mbeya.....	Pyrochlore.....	X
Uluguru Mountain.....	Uluguru Mountains.....		
Uganda:			
Karamoja.....	Lukupoi.....		
Do.....	Toror.....		
Mount Elgon.....	Mount Elgon.....		
Tororo.....	Tororo Hills.....	Pyrochlore.....	X
Do.....	Bukusu.....	Do.....	X
Do.....	Sukulu.....	Do.....	X
Do.....	Budeda.....		
Do.....	Sekululo.....		
Union of South Africa:			
Transvaal.....	Magnet Heights.....		
Do.....	Premier.....		
Do.....	Loolekop.....		
Do.....	Spitzkop.....		
Do.....	Glenover.....	Radioactive columbite.....	
Do.....	Goudini.....		
Do.....	Tweerivier.....		
Do.....	Kruidfontein.....		

counters. Many other columbium-bearing complexes, discovered through pronounced magnetic anomalies, contain disseminated magnetite. Ring structures are common, and some of the African complexes were identified by studying aerial photographs.

Granites and Granite Pegmatites

Columbite or tantalite can occur as a primary accessory mineral in granite or in pegmatites associated with granite bodies. The latter type of occurrence has been the historical source of most columbite, tantalite,

and microlite. In Nigeria, the Jos-Bukuru granites have been worked for their accessory columbite, and granite in Kaffo Valley is a potential source of pyrochlore, which is present as an abundant accessory constituent. Outside Nigeria, large granite bodies that are worth working for their columbium-tantalum values, have not been found. Elsewhere the source has been pegmatites of a great variety of size, shape, and grade. Granite pegmatites are simply described as coarse to very coarse-grained granites. Besides covering a great range in gross size of

the pegmatite and grain size of the minerals, the columbium content may range from abundance to total absence. Probably only about one pegmatite in one hundred has any record of being of economic value. No two pegmatites are exactly alike, and even a single deposit may have marked differences in texture, composition, size, and attitude in various sections. Because within a given pegmatite the minerals often occur in definite zones, some fairly plausible assumptions can be made as to zonal extent, mineral assemblages, tonnage, grade, and the mining methods of probable optimum efficiency. Most pegmatites are operated for more than one valuable mineral, beryl, cassiterite, columbite, feldspar, lepidolite, mica, pollucite, quartz, spodumene, and tantalite being most often sought. Pegmatites are widely distributed in regions of crystalline rock, and pegmatites rich in rare elements have been worked in many countries, including the United States, Canada, Spain, Portugal, U.S.S.R., Brazil, Argentina, India, Nigeria, Republic of the Congo, Malagasy Republic (formerly Madagascar), Australia, South-West Africa, Bolivia, Korea, and Mozambique. Divergent theories of origin have been presented for pegmatites; each theory is probably applicable to different pegmatites. According to the "magmatists" the solutions that formed pegmatites were derived as end products of the differentiation of deep-seated magmas; the "granitizationists" hold that not only pegmatites, but even some granite bodies themselves did not originate from an acid magma but resulted from heating, recrystallization, and the action of hot solutions (heated connate water) on old, deeply buried rocks.

Prospecting for pegmatites is to some extent predictable, since in certain instances their distribution is controlled by the structure of the rocks that enclose them; well-bedded, foliated, or fractured rocks may contain roughly aligned pegmatites. Often the pegmatites are found to have formed in so-called pressure shadows in crests and troughs of folds, which tend to form a non-random pattern in regularly folded areas. In addition, weathering may result in distinctive topographic expression such as pegmatites standing in relief with respect to the surrounding rocks. Light-colored pegmatites sometimes are prominent features in aerial photographs.

In weathered or deeply covered terrain, diagnostic minerals in the regolith are often an aid to prospecting. Some quartz-rich

pegmatites or pegmatite-core zones may be distinguished by low magnetic intensities or by resistivity.

Syenite and Syenite Pegmatites

Columbium minerals may also occur in syenite and nepheline syenite and in the corresponding pegmatites where carbonatites are not associated. Syenite consists mostly of alkalic feldspar and a mafic mineral such as hornblende or biotite; quartz is present only in small amounts. In nepheline syenite the primary constituents are alkalic feldspar, an alkalic mafic mineral, and nepheline. These rocks resemble granite and granite pegmatites in occurrence. Tantalum content is low. Most of the possible economic concentrations of columbium occur in titanium minerals where columbium represents a possible byproduct of titanium production or in pyrochlore. Some process, such as bacterial leaching, may eventually be developed enough to permit recovery of columbium from nepheline syenites, not of economic interest in 1960.

Deposits in Bauxite, Alluvium, and Eluvium

Many economic concentrations of columbium and tantalum result from weathering of the rocks discussed above. The weathering processes result in chemical decomposition and mechanical disintegration of some of the rock; concentration of columbium-tantalum minerals is greater in the residual fraction. These concentrations are variously referred to as residual deposits, eluvial deposits, and saprolite. A special type of residual product is bauxite, an ore of aluminum. Some bauxite derived from syenite may contain columbium, usually in accessory titanium minerals. When the mineral grains freed by weathering have been transported, sorted, and concentrated by running water, the resultant sands and gravels are called alluvial, and may include many valuable concentrations of heavy minerals such as those which contain columbium and tantalum.

For the formation of economic residual deposits several environmental conditions are required. First, the valuable minerals must be resistant to chemical and mechanical weathering and must be present in a rock in which the undesired minerals are soluble and less resistant to weathering under surficial weathering conditions. Second, the climate must be suitable for chemical decay. Third, the topography must be relatively flat to prevent washing away of the residue. Finally,

the deposit must be either not subjected to or protected from erosion by running water or glacial ice. Residual weathering has caused the feldspar of a syenite to weather to bauxite, which persists in situ while other constituents are removed.

Alluvial concentrations or placers (in the restricted geological sense) are formed when minerals released from their matrix by weathering are slowly washed downslope and at the same time comminuted by attrition. Minerals eventually reaching a stream or the seashore are sorted by moving water that sweeps away the lighter substances while the heavy minerals that have moved relatively short distances are gradually concentrated in stream or beach deposits. To form placers, water velocity must be sufficient to remove lighter minerals but not enough to sweep away and dissipate the valuable grains. Concentrations commonly occur where a decrease in stream gradient, meandering, spreading, or obstructions (natural riffles) produce a slackening of velocity that permits heavy minerals to drop and accumulate. Wind and water currents along shorelines also perform similar concentration tasks, which can result in placers. In addition to modern placers,

fossil placers also are found in solidified gravels (conglomerates and sandstones), where water-concentrated deposits have been buried, preserved, lithified, and subsequently reexposed by a later erosion cycle.

DESCRIPTION OF DEPOSITS AND RESOURCES (46,89)

Until after World War II, known columbium and tantalum deposits were almost exclusively in pegmatites or their derived weathering products. Ore shortages during World War II and the Korean war resulted in ore prices (including U.S. Government bonuses) that greatly encouraged exploration. Beginning in the mid-1950's this exploration resulted in the discovery of unexpectedly large resources of columbium, mostly in carbonatites. By 1960, total cataloged resources were more than 9 million tons of Cb_2O_5 . New tantalum resources discovered still total only about 100,000 tons of recoverable Ta_2O_5 , although the actual but untabulated resources in pegmatites may be several times as much. Known resources of the two elements are tabulated in table 22, and the location of the deposits are shown in figure 2. Although the

TABLE 22.—Columbium-tantalum resources of the world¹

Location and company	Ore, tons	Grade (contained oxides, percent)	Cb_2O_5 , tons	Ta_2O_5 , tons	Remarks
North America:					
Canada:					
Lake Nipissing, Ontario (Nova Beaucage Mines, Ltd.)	5,400,000	0.7	37,800	Pyrochlore.
Lackner Lake, Ontario (Multi-Minerals, Ltd.)	50,000,000	.26	130,000	Do.
Chapleau Ontario (Dominion Gulf).	{ 20,000,000 15,000,000	.5	100,000	}.....	Do.
		.3	45,000		
Oka, Quebec:					
Quebec Columbium, Ltd. (Kennecott & Molybdenum Corp.)	30,000,000	.60	180,000	}.....	Pyrochlore. Inferred as much as 100 million tons of ore.
	25,000,000	.35	85,000		
Columbium Mining Products, Ltd. (Coulee-Red Lake).	106,000,000	.25	266,000	Pyrochlore.
Oka Rare Metals Mining Co., Ltd.	200,000	.29	580	Do.
St. Lawrence Columbium and Metals Corp., Ltd.	⁴ 17,600,000	.36	63,360	}.....	Do.
	⁵ 45,000,000	.36	162,000		

See footnotes at end of table.

TABLE 22.—*Columbium-tantalum resources of the world—Continued*

Location and company	Ore, tons	Grade (contained oxides, percent)	Cb ₂ O ₅ , tons	Ta ₂ O ₅ , tons	Remarks
North America—Continued					
Canada—Continued					
Bugaboo Creek, British Columbia: Quebec Metallurgical Industries (Ventures, Ltd.)	(⁶)	(⁷)	5,100	Placer.
Total oxides, Canada.....			1,074,840	
United States:					
Colorado: Powderhorn (DuPont).	40,000,000	0.25	100,000	Pyrochlore carbonate.
Idaho:					
Bear Valley (Porter Brothers).	(²)	(³)	8,000	2,000	Black sand placer with euxenite, columbite, and samarskite.
Dismal Swamp.....			1,000	100	Black sand placers.
Arizona: Yavapai County.....			1,000	Pegmatites.
New Mexico: Rio Arriba County.....			1,000	Placers and pegmatites.
Montana.....					Placers, syenite and carbonate veins.
Arkansas:					
Bauxite.....	44,000,000	.10	44,000
Titanium deposits.....	8,000,000	0.05-.15	8,500	Inferred resources exceed 100,000 tons of Cb ₂ O ₅ .
Alumina plant wastes...	2,000,000	.13	2,600	
Oklahoma: Otter Creek Valley.	374,400,000	.004	14,300	Placer.
Total oxides, United States.....			180,400	2,100	
Total oxides, North America.....			1,255,240	2,100	
South America:					
Brazil:					
Araxá (D. E. M. A.).....	{ 200,000,000	3.0	6,000,000	}.....	Pyrochlore.
	{ 7,000,000	5.0	350,000		
Tapira (H. M. Borges).....					Some reports indicate 8 million tons of Cb ₂ O ₅ .
Tantalite.....			4,000	10,000	Pegmatites.
French Guiana: Tantalite.....	- 300,000	.4	1,200	Placers.
Total oxides South America.....			6,355,200	10,000	
Europe:					
Germany: Kaiserstuhl (Niob-Bergbau G.m.b.H.).	750,000	.5	3,750	Pyrochlore.

See footnotes at end of table.

TABLE 22.—Columbium-tantalum resources of the world—Continued

Location and company	Ore, tons	Grade (contained oxides, percent)	Cb ₂ O ₅ , tons	Ta ₂ O ₅ , tons	Remarks
Europe—Continued					
Norway: Sjøve (A/S Norsk Bergverk).	11,000,000	0.2-0.5	38,500	Pyrochlore. Additional reserves of at least 50 million tons of ore probably are available.
Total oxides, Europe.....			42,250	
Africa:					
Congo, Republic of the (formerly Belgian Congo) (columbite-tantalite).			50,000	50,000	Additional reserves exist but are not tabulated.
Kenya: Mrima Hill (Anglo-American, Ltd.)	26,000,000	.78	202,800	Pyrochlore.
Nigeria:					
Kaffo Valley.....	140,000,000	.26	364,000	Do.
Columbite.....			100,000	10,000	Several hundred million more tons inferred.
Tanganyika: Mbeya (N. V. Billiton and Government).	{ 3,800,000 14,000,000 63,000,000	{ .79 .34 .30	{ 30,000 50,000 190,000	}.....	Pyrochlore.
Uganda: Tororo (Sukulu Mines, Ltd.) (Frobisher, Olin-Mathieson, and Uganda Government).	202,000,000	.20	404,000	Do.
Total oxides, Africa.....			1,390,800	60,000	
Total oxides, Asia.....					
Total oxides, Oceania: Australia.			1,000	Alluvial and pegmatite.
Total Cb+Ta oxides, world.			9,044,490	72,100	

¹ Indicated and measured. Inferred resources are additional and mentioned in remarks column. No attempt has been made to differentiate reserves from resources since in most cases this varies annually depending upon supply-demand situation and concomitant price adjustments.
² 200 million cubic yards.

³ 1.0 pound per cubic yard.
⁴ Proved.
⁵ Indicated.
⁶ 65 million cubic yards.
⁷ 0.11 pound of columbium per cubic yard.

map shows a broad worldwide distribution for columbium and tantalum, the accompanying table shows that the large resources are concentrated in a very few countries, and that the United States is almost completely dependent upon overseas supplies for tantalum.

North America

Canada (37,71)

Very small quantities of columbium-tantalum minerals have been produced in

Canada, mostly from the Yellowknife area, Northwest Territories. Canada could be a large supplier of columbium ore from vast pyrochlore deposits.

The total Canadian reserve, almost all in pyrochlore-type deposits, is estimated to be 1 million tons of contained Cb₂O₅.

British Columbia

Pyrochlore, euxenite, and polycrase occur about 25 miles from Spillimacheen in gravels derived from erosion of the Bugaboo granite stock. In 1956 small quantities were mined

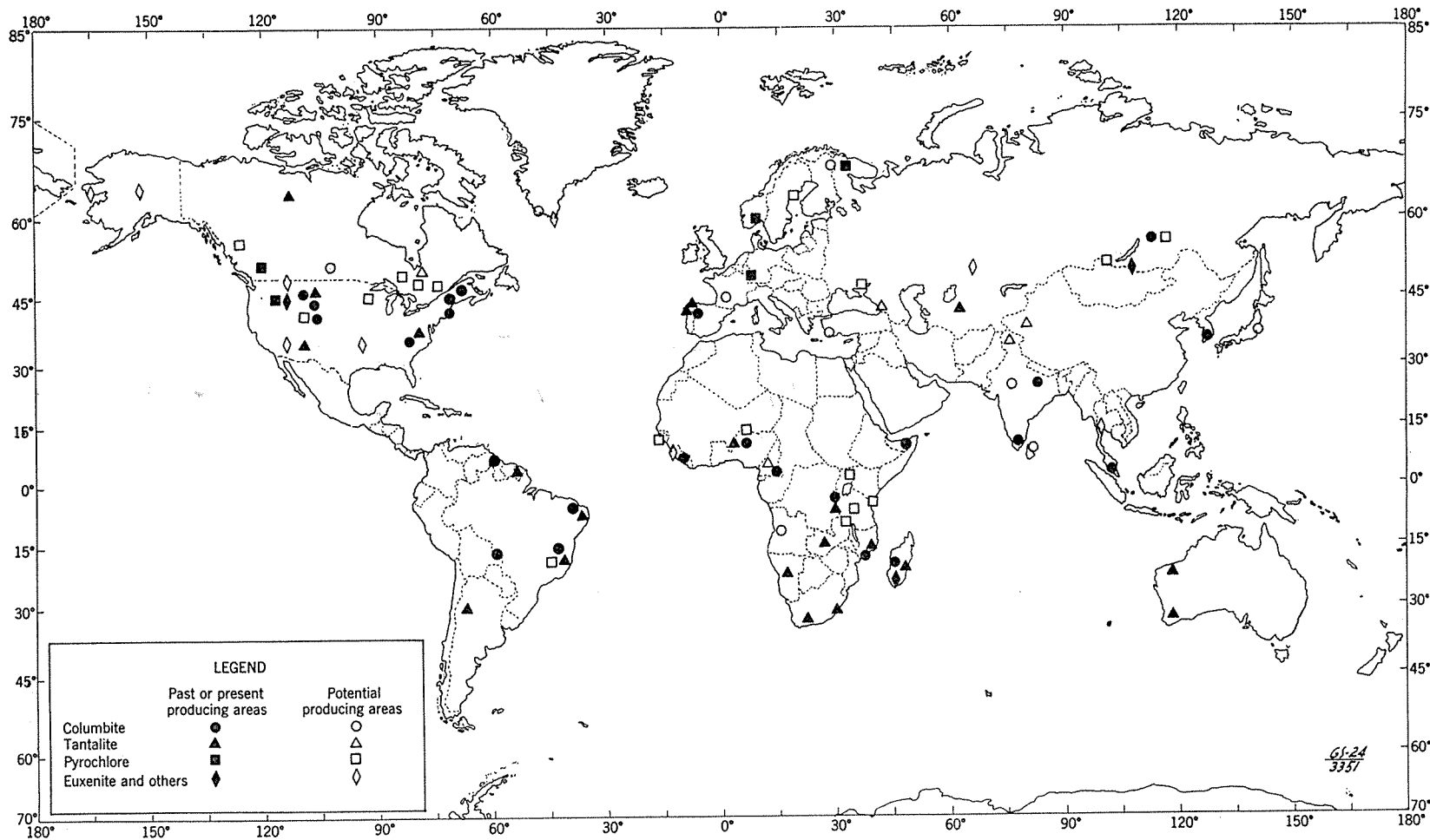


FIGURE 2.—Location of Columbian-Tantalum Resources of the World.

and concentrated and used in experimental production of columbium metal, columbium pentoxide and ferrocolumbium by Quebec Metallurgical Industries, Ltd., Ottawa. The deposit contains 65 million cubic yards of gravel, from which 0.11 pounds per yard of columbium could be recovered. This represents a potential reserve of 5,114 tons of Cb_2O_5 .

Columbium is present in pyrochlore, columbite, and ilmenorutile disseminated through alkaline and carbonatite rocks on the Lonnie deposit of Northwest Exploration, Ltd., along Granite Creek, a tributary of Manson Creek. The complex is exposed over an area 200 feet by 2,400 feet.

Radioactive minerals occur in pegmatite near the head of Moose Creek, a tributary of Beaverfoot River. Samples taken from different sections of the pegmatite assayed 0.016 to 0.08 percent columbium.

In 1950, pyrochlore was discovered in a carbonate rock interbedded in gneiss, 4 miles south of Lemprière Station. No ore of importance was found.

Manitoba

Red feldspar dikes in the Oiseau River area about 9 miles from Pointe du Bois contain columbite, tantalite, beryl, and uraninite. Columbite-tantalite occurs near Bernic Lake in pegmatite that is rich in lithium and cesium.

A pegmatite in the Rush Lake area on the Odd Claim, Lac du Bonnet Mining Division, contains 0.23 percent $(Cb, Ta)_2O_5$. The granitic pegmatites in several localities of the Winnipeg River area contain columbite-tantalite in subcommercial quantities.

Ontario

Pyrochlore occurs with magnetite and apatite in alkaline rocks and carbonatite bodies on the property of Multi-Minerals, Ltd., 7 miles northeast of Nemegos Station in the Sudbury mining district. Diamond drilling outlined 50 million tons of ore, averaging 0.26 percent Cb_2O_5 in two deposits (130,000 tons of contained Cb_2O_5). The property was discovered by aeromagnetic surveying in 1951.

Two pyrochlore-type ore bodies were outlined in an alkaline complex at the Dominion Gulf Co. property at Nemegosenda Lake, 17 miles northeast of Chapleau in the Sudbury mining district. Diamond drilling proved 20 million tons of 0.5 percent Cb_2O_5 ore in one body and indicated at least 15 million tons of 0.3 percent Cb_2O_5 ore in the other (145,000 tons of contained Cb_2O_5). The mineraliza-

tion was first indicated by aeromagnetic surveys in 1954.

Pyrochlore occurs in gneisses and carbonate rocks underlying the Manitou Islands and surrounding parts of Lake Nipissing. Drilling and underground development in the area east of Newman Island proved 617,000 tons of ore containing 1.06 percent Cb_2O_5 and 0.075 percent U_3O_8 ; 1,824,000 tons of ore containing 0.88 percent Cb_2O_5 and 0.05 percent U_3O_8 ; and 2,695,500 tons of ore containing 0.6 percent Cb_2O_5 and 0.042 percent U_3O_8 . The total Cb_2O_5 content in this zone would be 41,190 tons. A 50-ton-per-day test mill was erected at Yellek, 5 miles west of the town of North Bay. The ore at Newman Island is reported to contain acmite, 20 to 60 percent; calcite, 10 to 40 percent; apatite, 2 to 30 percent; biotite, 10 to 30 percent; and pyrochlore, magnetite, pyrite, and pyrrhotite, up to 20 percent.

Samarskite and uraninite occur in a feldspar dike near Blackstone Lake in Conger Township. Toddite, a uranium-rich columbite, occurs in a feldspar dike in Dill Township.

Uraniferous betafite crystals 1 to 3 inches long occur in a sill-like calcite body near alkaline rock on the property of Silver Crater Mines, Ltd., near Bancroft, Faraday Township. The betafite contains 41.5 percent Cb_2O_5 , 1.4 percent Ta_2O_5 , and 21.4 percent U_3O_8 and is associated with mica, apatite, and scapolite. Columbium minerals also are reported in small quantities from Monteagle, Lyndoch, Brudenell, Raglan, and Cardiff Townships.

Quebec

Columbium, tantalum, and rare-earth minerals were first identified in the Oka region in 1953 by the Molybdenum Corp. of America. Since then, large areas have been outlined, containing columbium, tantalum, rare-earths, uranium, thorium, iron, and ilmenite in pyrochlore, perovskite, magnetite, betafite, and niocalite. Seventeen companies reportedly have holdings in a roughly circular area more than 5 miles in diameter. The ore occurs in both carbonatite and alkaline rocks in contact zones of the Monteregean intrusives. Since pyrite and calcite are common in such ore, flotation cells would be required in any beneficiation plant to remove these deleterious minerals.

Quebec Columbium, Ltd., reports 30 million tons of 0.6 percent Cb_2O_5 and 25 million tons of 0.35 percent Cb_2O_5 ore. Columbium Mining Products, Ltd., reports 106 million tons of 0.25 percent Cb_2O_5 ore. St. Lawrence Colum-

bium and Metals Corp. reports 17.6 million tons of 0.36 percent Cb_2O_5 proved; total reserves of proved and indicated ore were 62.6 million tons.

The reserves for the entire Oka district are believed to contain more than 600,000 tons of Cb_2O_5 .

Other occurrences of columbium are reported from Preissac, Figuery, Lacorne, and Maissoneuve Townships.

Northwest Territories

Pegmatites in the Yellowknife-Beaulieu area contain columbite-tantalite along with beryl, spodumene, and amblygonite. The Moose, Tan, and Best Bet groups on Hearne Channel, Great Slave Lake, have been worked at various times for tantalite. They were last worked by Boreal Rare Metals, Ltd., from 1953 to 1956. The mineral was used at the company's refinery at Cap de la Madeleine, Quebec, to produce tantalum and columbium pentoxides.

The Lita group on the northwest shore of Buckham Lake contains columbite, tantalite, spodumene, and beryl. The Peg Group between Ross and Rideout Lakes, containing columbite-tantalite with beryl, was worked in 1946 and again in 1951. In 1946 the concentrate ran 70 percent Ta_2O_5 and 10 percent Cb_2O_5 .

In all, at least 60 pegmatites in the Yellowknife-Beaulieu area contain columbium-tantalum minerals. These pegmatites occur in diorite and granodiorite and are as much as 500 feet long by 40 feet wide.

Nova Scotia

Columbite is reported in a coarse pegmatite, at the Lavers Mine near New Ross, Lunenburg County.

GREENLAND

Accessory columbite occurs in pegmatite closely associated with cryolite deposits at Ivigtut in the Arsur fjord region. Fergusonite is reported in the Cape Farewell region, southern Greenland.

UNITED STATES

Until the early 1950's U.S. resources of both columbium and tantalum were believed to be extremely small. Between about 1955 and 1960, however, potential resources of contained columbium pentoxides were found amounting to more than 200,000 tons of Cb_2O_5 . Although the ores are mostly low-grade and presently not of commercial quality, they represent an ample supply for emergency use. Tantalum resources remain virtually unknown. The proven re-

serve is only about 2,000 tons on Ta_2O_5 , and the inferred reserve probably does not exceed 10,000 tons.

Alaska

Some of the Alaskan tin placers contain columbium. Spectrographic analyses of churn-drill concentrates from the Cape Creek placer at the western tip of Seward Peninsula indicated 0.01 to 0.1 percent columbium. Chemical analyses of churn drill concentrates from the Boulder Creek placer, also on the Seward Peninsula, indicated small quantities of columbium and tantalum in all samples (54). Near Tofty (65° 05' N. 151° W), in the Manley Hot Springs area, Hot Springs district, Central Alaska, spectrographic analyses of samples from the Deep Creek area indicated 0.1 to 5.0 percent columbium and chemical analyses of concentrate from Miller Gulch ranged from 0.2 to 7.0 percent Cb_2O_5 (83).

Pegmatites in many parts of Alaska are unevaluated but may be potential sources of columbite-tantalite.

Arizona

Euxenite, microlite, and pyrochlore occur in pegmatites of the White Picacho district, Yavapai and Maricopa Counties. The minerals comprise small isolated masses and thin coatings along fractures in small dikes up to 100 feet long by 20 feet wide. The ore is low grade and erratically distributed within the pegmatites.

Arkansas

Columbium is associated with Arkansas titanium and aluminum ores (20, 56, 57). In the Magnet Cove area, Hot Springs County, titanium minerals occur in association with a carbonatite-bearing alkaline complex. In places the rock averages as high as 0.04 percent rutile or 0.07 percent brookite. Single brookite crystals may contain 0.8 to 9.6 percent columbium and rutile crystals may contain up to 1.7 percent. Titanium concentrate made by the Bureau of Mines from the rutile contained 1.2 percent columbium, and brookite concentrate contained 2.0 percent. The principal columbium-bearing alkaline rock, a perovskite-bearing magnetite-pyroxenite, contains 0.03 to 0.04 percent columbium in places. A carbonatite exposed in one quarry averaged 0.01 percent columbium. In Garland County, at Potash Sulfur Springs, 6 miles west of Magnet Cove, a heavily weathered complex of syenite and other alkalic rocks contains 0.1 to 0.9 percent columbium. In Pulaski and Saline Counties a titanium-bearing bauxite derived from

nepheline syenite contains 0.05 percent columbium. Total tabulated resources in the bauxite, titanium deposits, and alumina plant wastes from treating Arkansas bauxite exceed 50,000 tons of Cb_2O_5 , and the inferred resources in the titanium deposits alone exceed 100,000 tons.

California

Traces of columbium have been reported in analyses of rare-earth minerals from the carbonatite at Mountain Pass, San Bernadino County. Pyrochlore is reported to have been found in San Diego County; columbite, at Rincon, Santa Cruz County, and Ramona, San Diego County; euxenite, at Palmdale, Los Angeles County; betafite and brannerite, near Lucerne Valley, San Bernardino County; and fergusonite, near Ramona, San Diego County.

Colorado

The largest known columbium reserve in the United States occurs in an alkalic complex at Powderhorn, Gunnison County, Colo., principally as pyrochlore, in a large carbonatite intrusive plug composed mainly of dolomitic marble (25). Magnetite, vermiculite, and perovskite have attracted the attention of prospectors and developers to the area at various times, and a group of claims in the area even was patented in 1897.

The carbonatite plug has a basal area of about $1\frac{1}{2}$ square miles and forms the two prominent Big and Little Iron Hills, the highest of which rises 900 feet above the nearby valley of Cebolla Creek. Surrounding the plug on three sides is a horseshoe-shaped area of lower elevation that is underlain by a variety of less resistant rocks, the principal one of which is pyroxenite. The flat, debris-filled valley of the Cebolla Creek is on the fourth side. High hills surround the pyroxenite area and are composed of fenites grading outward into granite.

The pyrochlore was overlooked until October 1956, when the similarity between the Powderhorn rocks and those recognized as carbonatites in Africa was observed, and the pyrochlore that should be there was found. Because this deposit was potentially important as a raw material source, the duPont Company subsequently acquired, by staking and by purchasing existing claims, the mineral rights to almost 4,000 acres of land covering the carbonatite and the surrounding pyroxenite areas.

Intensive exploration of the carbonatite in 1957 and 1958 can by no means be considered complete, but enough of its surficial area has

been sampled by drilling to indicate an ore reserve of considerably more than 100,000 tons of Cb_2O_5 in rock averaging at least 0.25 percent Cb_2O_5 . An impressive quantity of ore averaging 0.35 percent Cb_2O_5 has been indicated in areas large enough to be mined by open cutting, and higher grade ore occurs in significant quantities. The minute pyrochlore crystals generally occur in narrow shear zones characterized by elongate dolomite crystals and accessory elongate apatite and blue amphibole crystals. The pyrochlore contains a little thorium but not enough that radioactivity can be used as a guide to pyrochlore. Other thorium-bearing minerals are believed to occur in the rocks but have not been specifically identified. The apatite contains rare earths of the cerium group. Pyrite is widespread in the carbonatite and has been altered to iron oxide to a notable extent. Magnetite, mostly altered to brown and red hydrated iron oxides, is abundant locally. Jasperoid silica is prominent in places.

In Park County, west of Antero Junction, samarskite is reported with magnetite disseminated in granite.

Production of columbite-tantalite and some microlite from many pegmatites has been small and sporadic in Boulder, Chaffee, Clear Creek, Douglas, Fremont, Gilpin, Gunnison, Jefferson, Larimer, and Park Counties. The mineral occurs, often with beryl, in the intermediate zones of the pegmatites, but the total reserves have not been estimated.

Connecticut

Columbium was first identified in a mineral specimen collected near New London in the 18th century. Since then small quantities of columbite have been produced sporadically as a byproduct from the Portland beryl-mica pegmatite district in Hartford and Middlesex Counties and the Branchville pegmatite area in Fairfield County.

Idaho

In Elmore County, the Dismal Swamp placer contains columbite-tantalite, samarskite, monazite, and zircon derived from weathering of pegmatites associated with the Idaho batholith. The gravel contains 1.40 to 1.87 pounds per yard of weakly magnetic material containing 14 to 20 percent $(Cb, Ta)_2O_5$ and 0.15 to 0.19 percent U_3O_8 . The gravel was worked for a few years during the 1950's by J. R. Simplot and Co., but reserves are relatively small (3).

In Idaho County a gold placer on the Red River, 10 miles south of Elk City, contains columbium in radioactive black minerals. The

nonmagnetic fraction from jig-bed concentrate contains 0.2 percent columbium (4).

Columbium occurs in Lemhi County, 30 miles northwest of Salmon in the Mineral Hill district. A belt 1.5 miles wide and 18 miles long contains monazite, columbium-bearing rutile, ilmenite, magnetite, thorite, and allanite in carbonate veins, replacing amphibolites, gneisses, and limestone. The rutile contains 4 to 10 percent columbium, small quantities of which also occur in the ilmenite. Individual deposits averaging 1 to 2 feet wide are traceable for only a few dozen feet due to heavy soil cover. The maximum known dimensions are 1,000 feet by 8 feet for an individual deposit. No large deposits have been found that are rich in rutile, but some may exist (2).

Bear Valley is in the southeastern part of Valley County, Idaho, about 23 miles northeast of Lowman. The deposits are in the center of the area underlain by rocks of the Idaho batholith. The country rock, a quartz monzonite near the deposit, is cut by numer-

ous aplite and pegmatite dikes. Though columbium mineralization occurs in both dikes and country rock at many localities, conditions were amenable for concentrating heavy minerals in placers in only a few places, such as Bear Valley (fig. 3). Localization of deposits was controlled by (1) favorable distribution of heavy minerals in slopes above the valley, (2) sufficient weathering to free large quantities of the minerals, (3) damming of normal drainage by late Pleistocene glaciation leading to accumulation of valley fill, and (4) quickened down-slope movement of enriched mantle during glaciation. A typical analysis of the placer deposit would show the following quantities of heavy minerals, in pounds per yard of gravel: Euxenite, 1.0 ; monazite, 0.5; garnet, 13.0; columbite, 0.2; zircon, 0.05; ilmenite, 28.0; and magnetite, 7.0; for a total of 49.75 pounds per yard (40). Similar placers of unevaluated extent are known at Cascade, Deadwood, and Gold Fork. The Gold Fork placer was drilled and sampled by the Bureau of Mines in August

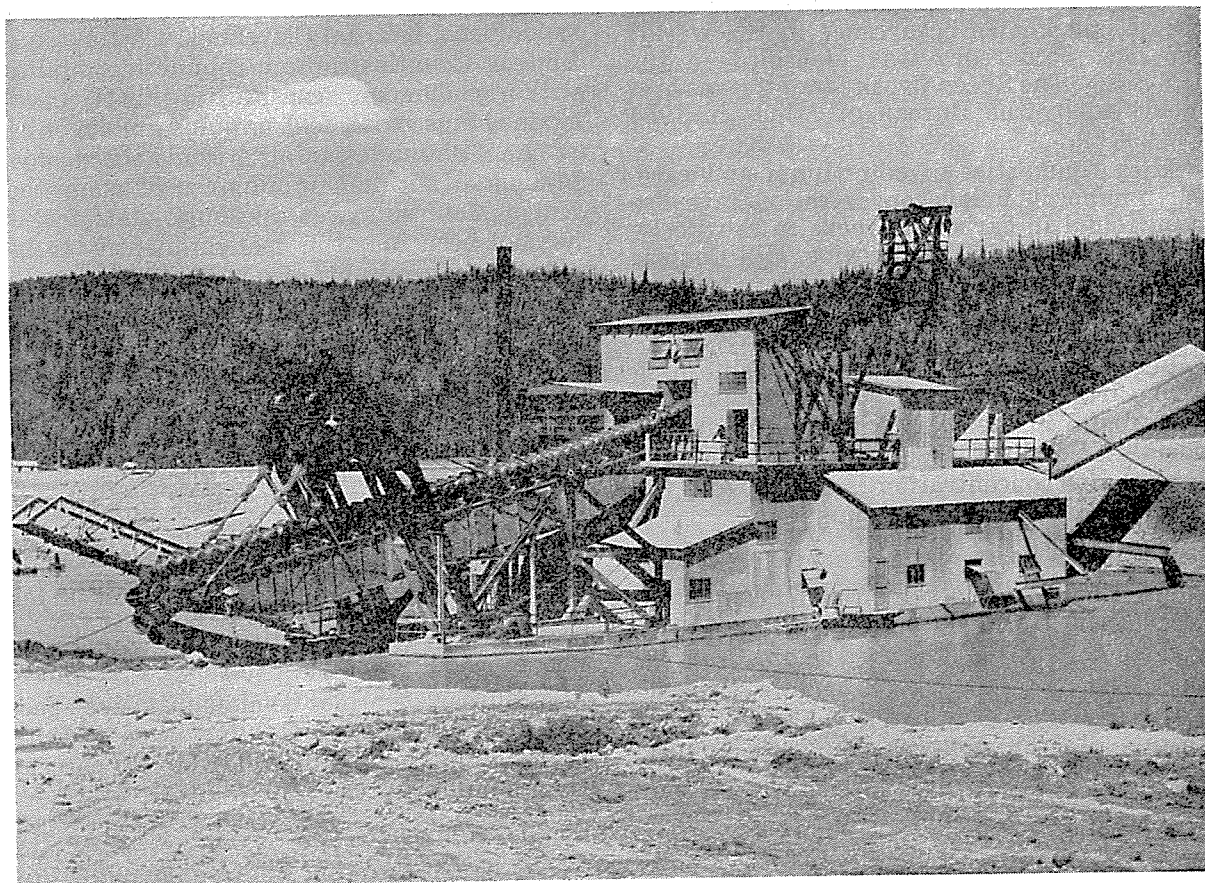


FIGURE 3.—Six-Cubic-Foot Bucket Dredge at Bear Valley, Idaho, Columbium Placer Mine.
(Courtesy of Porter Brothers Corp.)

1956; the black sand concentrate that was prepared from the samples contained 0.20 to 1.10 percent (Cb, Ta)₂O₅ (80). Ilmenite concentrate from Cascade yielded 0.42 pounds of columbium per ton of mill feed.

Maine

Pegmatites in Oxford and Sagadahoc Counties occasionally have yielded small quantities of byproduct columbite-tantalite. The Black Mountain quarry in Oxford County is estimated to contain 10 tons of columbite-tantalite.

Montana

Columbium-bearing minerals are found in several places in Montana. On Sheep Creek in southern Ravalli County, columbite, euxenite, and fersmite, along with the rare-earth minerals, ancylite, allanite, and monazite, occur in concordant carbonate zones in amphibolite, gneiss, and schist. The columbium and rare-earth minerals are erratically distributed in the irregular carbonate bodies. The bodies average 4 to 5 feet thick and 0.10 to 0.30 percent columbium. The fersmite is the second known occurrence in the world; the first occurrence was identified in the U.S.S.R. The Sheep Creek fersmite was first identified by the Bureau of Mines in 1954 (28).

Large quantities of columbium and rare-earth minerals in low concentration have also been found in carbonate rocks in the Rocky Boy stock, Bearpaw Mountains, Hill County. Columbium minerals also occur in placers such as at Lake Delmo, Sand Basin, and the head of California Gulch near Laurin.

Fergusonite, a complex mineral containing rare earths, columbium, and uranium, has been found in the Sappington pegmatites in northern Madison County. However, the fergusonite concentration is spotty, and the deposits have not been commercial.

New Hampshire

Intermittent, small, byproduct columbite-tantalite output has been reported from pegmatites in Grafton County; leading sources have been the Pattuck, E. Smith, Palermo No. 1, Ruggles, and Keyes mines. Conway granite at Iron Mountain, Carroll County, is reported to contain 0.01 to 0.02 percent columbium, and one Bureau of Mines semiquantitative spectrographic analysis indicated 0.01 to 0.1 percent columbium. At Red Hill, wohlerite, NaCa₂ (Zr,Cb) FSi₂O₈, is reported as sparingly present.

New Mexico

Pegmatites and derived placers in Rio Arriba, San Miguel, and Taos Counties, contain euxenite and columbite-tantalite. One pegmatite, the Harding, has been the most successful U.S. tantalum mine. Tantalite and microlite occur with spodumene, lepidolite, and beryl in a well-zoned quartz-microcline-muscovite pegmatite at least 800 feet long and up to 200 feet wide. The pegmatite was drilled by the Bureau of Mines in the 1940's, and although the drilling was not entirely conclusive, the tantalum-bearing zone in the mine appeared to be almost worked out. There are outcrops of other unexplored pegmatites in the area, which may contain similar mineralogical suites (8). In Otero County, in nepheline syenite, eudialite contains 0.53 percent columbium; the quantity of ore is unknown, but the rock is said to resemble material from the Kola peninsula, U.S.S.R.

North Carolina

The pegmatites of the North Carolina tin-spodumene belt in Cleveland, Gaston, and Lincoln Counties contain columbite-tantalite with spodumene, cassiterite, beryl, fine-grained muscovite, feldspar, and amblygonite (39). Columbite-tantalite could be a byproduct of lithium mining, such as in the Kings Mountain area, although thus far it has not been found in enough quantity to be extracted profitably as a byproduct. In Burke County, fergusonite has been reported in old gold placers.

Oklahoma

In Otter Creek Valley, Kiowa County, columbium is a minor constituent of ilmenite-bearing placer sands. Ilmenite concentrate produced in tests contained 0.08 to 0.18 percent columbium; representing a total of 14,300 tons of Cb₂O₅ in the deposit. This columbium is not of economic interest under present conditions.

Rhode Island

A Bureau of Mines semiquantitative spectrographic analysis of Quincy-type granite from Cumberland indicated 0.3 to 3.0 percent columbium.

South Carolina

The North Carolina tin-spodumene belt of columbite-tantalite bearing pegmatites extends into Cherokee County.

South Dakota

Columbium-tantalum minerals have been a byproduct, usually of beryl or lithium minerals, from numerous pegmatites.

The principal columbium-tantalum pegmatites occur in Custer, Lawrence, and Pennington Counties. In Custer County the pegmatites are Beecher, Helen Beryl, High Climb, Highland, Hot Shot, Lucky Strike, Old Mike, Snowflake, and Tin Mountain. At the Tin Mountain pegmatite, columbite-tantalite occurs in the wall zone, the second intermediate zone, and the core. In the first two zones the grains are rare, platy, and small. In the core the columbite-tantalite is more abundant, and grains as much as 0.04 square foot in the exposed area were found. This unit was estimated to contain up to 4 pounds of columbite-tantalite per ton of rock. Microlite was found only in the core of the pegmatite as grains up to 1 inch in diameter. The microlite grains are usually in narrow bands of pale-purple or greenish lepidolite and albite.

Most of the tantalite-columbite in the Helen Beryl pegmatite is in a band as much as a foot thick at the outside edge of the quartz-spondumene-perthite-albite core. Specific gravities of the mineral grains indicate a range in composition of from 66 percent Ta_2O_5 and 19 percent Cb_2O_5 to 73 percent Ta_2O_5 and 12 percent Cb_2O_5 ; the average is 70.5 percent Ta_2O_5 and 14 percent Cb_2O_5 .

In Lawrence County the principal pegmatites are the Tinton, Giant Volney, Rough and Ready, and Tantalum Hill. The Tantalum Hill and Rough and Ready deposits were explored by the Bureau of Mines by diamond drilling and drifting (32). Composite samples averaged 0.4 percent tantalum. From 1936 to 1938 the Tantalum Hill mine was worked by Fansteel to recover 21,884 pounds of tantalite containing 45 percent Ta_2O_5 .

In Pennington County the principal pegmatites are the Bob Ingersoll, Peerless, and Whitecap. Columbite-tantalite averages 1 pound in 10 tons of rock at the Peerless pegmatite. Although single masses of columbite up to 200 pounds have been found, the crystals average 0.2 inch long by 0.01 inch thick, and hand-cobbing is difficult, owing to intergrowth with muscovite and cleavelandite.

In addition to the pegmatites mentioned, about 20 others have produced tantalum minerals.

Texas

In Llano County at Barringer Hill, 12 miles north of Kingsland, euxenite, fergusonite, and other rare-earth minerals occur in pegmatite.

Utah

Beryl-bearing pegmatites with sparse accessory columbite-tantalite occur in Beaver, Tooele, and Juab Counties.

Virginia

In Amelia County, pegmatites in the Amelia Courthouse area contain manganotantalite, columbite, pyrochlore, fergusonite, and microlite. Microlite, the most abundant, occurs in masses up to 18 inches in diameter. Some of the pegmatites contain up to 2 pounds of columbium-tantalum minerals per ton of rock. Small quantities of columbite-tantalite are found in pegmatites in Amherst, Bedford, and Powhatan Counties (61).

Veins in granodiorite have been worked for tin near Irish Creek, Rockbridge County. Among the associated minerals, wolframite was analysed, which contained 0.96 percent $(Cb, Ta)_2O_5$.

Wisconsin

In Marathon County near Wausau, pyrochlore occurs with zircon in quartz-feldspar pegmatites associated with nepheline syenite. In addition to the columbium in the pyrochlore, the zircon contains 0.9 percent $(Cb, Ta)_2O_5$.

Wyoming

Columbite-tantalite has been produced in very minor amounts as a byproduct of mica and beryl mining. It has been reported from Albany, Converse, Crook, Fremont, and Goshen Counties.

South America

ARGENTINA

A small quantity of columbite and tantalite has been produced from pegmatites in San Luis and Cordoba Provinces since 1941. Best known is the Angel mine between Marlo and San Javier in Cordoba, where columbite occurs in masses up to a few hundred pounds. Other columbite- or tantalite-bearing pegmatites are reported in La Rioja, Catamarca, and western Salta Provinces.

BOLIVIA

Several small columbite mines are known in the Department of Santa Cruz. The columbite occurs with sheet mica and monazite close to the quartz core in pegmatites. The largest producer has been the La Verde Mine;

others are the San Jorge Mine and the Re-compensa Mine. A pegmatite of undetermined extent near Cochabamba between La Paz and Sucre contains an average of 4.0 percent (Cb, Ta)₂O₅ over an exposed face of 9 by 20 feet.

Bolivian reserves are nominally estimated to be less than 100 tons of Cb₂O₅.

BRAZIL

Brazil ranks second as a source of tantalum ore and is the leading source of high-grade high-ratio (tantalum-columbium) tantalite (table 23). Literally thousands of pegmatites occur in eastern Brazil extending from the State of Rio Grande do Sul in the south to the territory of Amapa in the north. Hundreds of these deposits have produced at one time or another (35). In addition alkalic rock-carbonatite complexes are known at 10 places in São Paulo, Santa Catarina, Minas Gerais, and Goiás (44).

In order of importance, producing pegmatite districts have been

1. Northeastern States: Ceará, Paraíba, Rio Grande do Norte, Bahia, Goiás.
2. Central States: Minas Gerais, Espírito Santo.
3. Southern States: São Paulo, Paraná, Rio Grande do Sul, Rio de Janeiro.
4. Amapa Territory.

The columbite-tantalite occurs in well-zoned pegmatites, which are associated with granites and quartz veins cutting Precambrian schists, gneisses, quartzites, and marbles. The pegmatites consist principally of muscovite, quartz, and albitized microcline. The

pegmatites are resistant to weathering and stand in relief above the landscape as conspicuous topographic features locally called altos. The lens-shaped bodies average about 500 by 100 meters in surface exposure and are well zoned. Other pegmatites in the area form tabular dikes, are homogeneous in mineralogy, and do not constitute sources of columbite-tantalite. The sequence in the zoned pegmatites typically is schist wall rock; muscovite zone; quartz, feldspar, mica zone with some cassiterite; giant feldspar (sometimes albitized) zone with beryl, tantalite, and spodumene; and quartz core frequently with beryl and, rarely, tantalite and spodumene.

The tantalite usually is a byproduct of beryl or cassiterite mining, and simultaneously favorable markets for all three stimulate production. The mineral also occurs in placers derived from weathering of the pegmatites, but these alluvial deposits have been of relatively small importance except for the alluvium immediately upon the slopes of the altos.

Deposits have been reported in the following areas:

Northeastern area:	
Amapa Territory	Numerous placers.
Rio Grande do Norte	Parelhas, Acari, Barra, Lages, Currais Novos, Jardim do Serido, and São Thome.
Paraíba	Picui, Santa Luzia, Joazeiro, Coite, Alagoa Grande, and Soledade districts.
Ceará	Cachoeira, Quixeramobim, Cascavel, Aracoiaba, Guarani, Quixada, and Pernambuco.
Bahia	
Goiás	

TABLE 23.—Analyses of Brazilian columbium-tantalum minerals, percent (43)

Mineral and location	Cb ₂ O ₅	Ta ₂ O ₅	MnO	Fe ₂ O ₃	TiO ₂	RE ₂ O ₃	ZrO ₂	ThO ₂	U ₃ O ₈										
										PbO	SnO ₂	SiO ₂	WO ₃	MgO	CaO	H ₂ O	Total		
Columbite, Salgado, Picui	66.65	13.50	6.71	13.59	23.70	23.12	4.23		7.50										
Blomstrandite-priorite, Espírito Santo	28.70	3.20	5.50	3.12				0.57	1.96										
Columbite, João Baptista Palermo, Minas Gerais	77.85	21.58		14.20	18.75	27.28		2.28	8.86										
Columbite, São Jose da Lagoa, Minas Gerais	25.17			2.05	2.20	37.25													
Eschwegeite, São Jose da Lagoa, Minas Gerais	46.44			.56															
Euxenite, Santo Antonio da Penitencia, Minas Gerais	(1)	82.48	17.60																
Manganotantalite, Salinas, Minas Gerais	59.04	22.80	3.36	15.08															
Columbite, Pinhao, Minas Gerais	51.64	28.48	3.86	13.92					8.20										
Do	31.80				38.20	17.00													
Polyrase, Santa Clara, Minas Gerais																			
Columbite, Salgado, Picui	0.14	(1)			0.28	1.03	(1)	0.11	99.56										
Blomstrandite-priorite, Espírito Santo								6.41	100.12										
Columbite, João Baptista Palermo, Minas Gerais								3.09	99.36										
Columbite, São Jose da Lagoa, Minas Gerais								2.14	100.45										
Eschwegeite, São Jose da Lagoa, Minas Gerais									99.73										
Euxenite, Santo Antonio da Penitencia, Minas Gerais						(1)	(1)		100.08										
Manganotantalite, Salinas, Minas Gerais		0.24				.41	0.89		100.28										
Columbite, Pinhao, Minas Gerais			6.00					4.00	99.44										
Do									99.20										
Polyrase, Santa Clara, Minas Gerais																			

1 Trace.

2 FeO.

Central area:

Minas Gerais.....Pecauha, Antonio Dias, Rio Piraciçaba, São Domingos do Prata, Conceição, Sabinópolis, Fortaleza, Theophilo Ottoni, Jequitinhonha, Malacaiheta, Itambacury, Minas Novas, Arassuaby, Serro, Diamantina, Salinas, Itabirito, Ouro Preto, Caratinga, Vicosá, Uba, Pomba, Muriahe, Carangola, Alem Parahyba, Bicas, Juiz de Fora, Andrelandia, São João del Rei (6), Governador Valadares, Aracuai, Itinga, and Santa Maria do Suacuí.

Espírito Santa.....Cachoeiro, Castello, and Itagussu.

Southern area:

São Paulo.....São Paulo, Mogi das Cruzes, Itapeperica, Iguape, Xiririca.

Paraná.....

Rio Grande do Sul.....

Rio de Janeiro.....Valença.

Alkalic complexes, some with associated pyrochlore or one of its alteration products, are known at several localities in Brazil. Some of the pyrochlore occurs in the alkalic intrusive rocks and some in associated carbonatites.

Reported localities are

São Paulo.....Jacupiranga, Juquia, Ipanema.

Minas Gerais.....Araxá (26), Tapira, Serra Negra, Salitre, Poços de Caldas.

Goias.....Catalao.

Santa Catarina.....Anitapolis.

Araxá, Tapira, and Serra Negra are potential sources of columbium. The Araxá deposit has been proved to contain 6 million tons of Cb_2O_5 in ore, averaging 3.0 to 5.0 percent Cb_2O_5 . This reserve is in a relatively small area and only to a depth of 45 meters, the thickness of the weathered mantle. The entire deposit contains many times as much recoverable columbium pentoxide. In January 1959 construction of a 200-ton-per-day concentrator was begun on the Araxá property. Some reports indicate that Tapira may contain similar tonnages.

Total proved columbium reserves in Brazilian pyrochlore are estimated to be at least 6,350,000 tons of Cb_2O_5 . Reserves of columbium-tantalum minerals in pegmatites and placers are not known but are estimated at 4,000 tons of Cb_2O_5 and 10,000 tons of Ta_2O_5 .

BRITISH GUIANA

Columbite-tantalite occurs in alluvial and eluvial deposits in a wide belt extending northeastward from the lower Puruni River to Rock Point in the lower Essequibo River district. The placers are underlain by granites and granite gneiss cut by pegmatite veins, from which the columbite-tantalite was derived. In addition to the Puruni and Essequibo Rivers, the Rumong-Rumong and Morabisi areas on the Mazaruni River are important.

Reserves have been estimated for only a few localities. Nominally they are estimated to total 500 tons of Cb_2O_5 and 250 tons of Ta_2O_5 . The deposits are 2 to 24 feet thick on an average and contain 1 to 4.5 pounds of columbite per cubic yard.

FRENCH GUIANA

Tantalite alluvial deposits have been mined along the Sinnamary River and its tributaries from 18 to 25 kilometers south of Sinnamary. The tantalite averages about 50 percent Ta_2O_5 and 30 percent Cb_2O_5 . The deposits are up to 4 feet thick, and the gravels are reported to contain up to 10 pounds of tantalite per ton.

Reserves, incompletely reported, are nominally placed at 500 tons of Ta_2O_5 and 300 tons of Cb_2O_5 .

SURINAM

Tantalite-columbite deposits have been found in the lower Marowijne River area and also on the east bank of the Surinam River, 35 miles south of Paramaribo. Reserve figures have not been revealed, and mining has not been undertaken.

Europe

FINLAND

Numerous columbium-tantalum bearing pegmatites are reported in northern Finland.

A carbonatite, on which no details are available, is reported at Kuusamo in east-central Finland close to the U.S.S.R. border.

FRANCE

Columbium-tantalum minerals are reported to be minor constituents of pegmatites at many places in southern France.

Crystals of neotantalite are reported at Echassières, Allier, in kaolin derived from weathering of granite.

Skogbolite containing 82.9 percent Ta_2O_5 is reported in the department of Haute-Vienne.

Columbite is found near Chaumasse, Mesure, Runchy, and Cuzy in Saône-et-Loire, and is reported at Clap near Castelnau de Brassac, Tarn-et-Garonne.

Columbite is reported at several places in the Central Plateau area, notably at Chanteloube, Maratand et Aven, and Campaignac. Manganocolumbite is reported from Chêneville and Larmont. Tantalite occurs at Chabaunes.

GERMANY

Pyrochlore (var. koppite) occurs in a carbonatite in contact with an alkalic intrusive rock (tephrite) at Kaiserstuhl in the Breisgau district of Baden. Magnetite and apatite are closely associated with the pyrochlore, which contains about 62 percent Cb_2O_5 and 7 to 10 percent of the cerium group of rare-earth oxides. The deposit, mined from 1952 to 1955, contains several hundred thousand tons of rock containing 0.2 to 0.5 percent Cb_2O_5 . Reserves, incompletely known, are estimated to be about 5,000 tons of Cb_2O_5 .

Small amounts of columbite-tantalite have been reported in phosphate-bearing pegmatites in Bodenmais, Hagendorf, and Robenstein in Bavaria.

NORWAY

Pyrochlore deposits are being mined at Søvne in the Fen district near Ulefoss, Telemark County. The deposits are in a small alkaline rock complex cropping out over a 6-square-kilometer area. The complex has a carbonatite core, inclusions of silicate rock, with syenites and syenitic fenites, including carbonatite lenses, on the periphery. Within the carbonatite (sovite) core, the steep north-south trending brecciated zones or dikes are richer in columbium pentoxide than the en-

closing carbonatite. The lens-shaped carbonatites around the periphery are also higher in columbium pentoxide. The mineralogy of the carbonate rock is complex, consisting of calcite, dolomite, biotite, magnetite, pyrite, pyrrhotite, fluorapatite, columbite (both primary and secondary after pyrochlore), pyrochlore (koppite), feldspar, hornblende, kyanite, barite, and zircon. The columbium minerals contain from about 59 to 74 percent $(Cb, Ta)_2O_5$.

The deposit was discovered in 1918, explored by I. G. Farbenindustrie during World War II, and then explored and developed by A. S. Norsk Bergverk beginning in 1951; commercial production began in August 1953.

The three most thoroughly explored dikes are the Cappelan, Hydro, and Tufte, which are reported to contain about 6,250,000 tons of ore above the level of Lake Norsjø (Cappelen, 1,500,000 tons; Hydro, 750,000 tons; and Tufte, 4 million tons. The Cb_2O_5 reserves in these deposits are about 17,000 tons. The total resources of the Sove area are estimated to be at least 50 million tons, containing 100,000 tons of Cb_2O_5 . The tantalum pentoxide content of the ore is small (table 24). To produce one ton of the final 50-percent Cb_2O_5 product, 84 tons of raw ore is required (9).

TABLE 24.—Analyses of Norwegian columbium-tantalum minerals, percent ¹

	Cb_2O_5	Ta_2O_5	TiO_2	SiO_2	FeO	CaO	MgO	BaO	MnO	Y_2O_3	Er_2O_3	Ce_2O_3	La_2O_3	Di_2O_3
Brown koppite, Cappelen deposit, Søvne (9)	59.40		4.16		0.60	18.35	0.46							
Black koppite, Cappelen deposit, Søvne (9)	62.40		3.97	1.99	18.70	7.58	.82	1.90						
Black Columbite, Tufte deposit, Søvne (9)	74.00		3.60		20.70		.89							
Fergusonite, Berg (59)	39.30	6.25		1.44	.78	1.23	.05		0.15	35.03		0.72	2.25	
Ytrotantalite, Berg (59)	20.38	39.53	1.67	.96	7.48	1.28	.15		1.85	12.48	3.58	.42	1.71	
Polymignite, Fredricksvarn (59)	11.99	1.35	18.90	.45	2.08	6.98	.16		1.32	2.26		5.91	5.13	
Mossite, near Moss (59)	52.00	31.00			16.62									
Columbite, Annerod (59)	72.37	5.26			15.04	0.58			5.97					
Euxenite, Arendal (59)	27.64	1.27	25.68	0.17	1.13	1.08	.06		.16	27.73			2.20	
Eschynite, Hitteroe (59)	23.85	6.97	22.60	(2)	4.28	2.52				4.53		19.50		
Samarските, Odegardsletten (59)	38.83	10.70		1.82	4.40	4.30	0.13	0.38	0.86	9.07			0.89	
	UO_2	ThO_2	ZrO_2	SnO_2	H_2O	BeO	WO_3	Na_2O	Fe_2O_3	Al_2O_3	PbO	K_2O	UO_3	Total
Brown koppite, Cappelen deposit, Søvne (9)														82.97
Black koppite, Cappelen deposit, Søvne (9)														97.36
Black Columbite, Tufte deposit, Søvne (9)														99.19
Fergusonite, Berg (59)	4.68	2.51	(2)	0.98	4.00	0.40								99.77
Ytrotantalite, Berg (59)	3.85	.67	0.57	1.20	.51	.35	0.66	0.57						99.87
Polymignite, Fredricksvarn (59)		3.92	29.71	.15	.28			.59	7.66	0.19	0.39	0.77		100.19
Mossite, near Moss (59)				.18										99.80
Columbite, Annerod (59)				.67										99.89
Euxenite, Arendal (59)	5.83	3.58	(2)	.18	2.55			0.18		(2)	0.63	0.09		100.16
Eschynite, Hitteroe (59)		15.42		(2)						(2)				99.67
Samarските, Odegardsletten (59)	9.66	2.51	0.62	.57	6.54	0.30		0.76		0.36	0.77	0.08	6.78	100.38

¹ When figure is centered, the composition is not strongly predominant for one or the other, or the producing company has not differentiated between them.

² Trace.

Columbium-tantalum minerals also occur in hundreds of granitic and syenitic pegmatites in southern Norway. In a very few, small quantities of columbite-tantalite have been recovered as a byproduct of feldspar mining. Some of the localities, with the columbium-tantalum minerals which occur, are given:

Møre og Romsdal County:	
Annerode.....	Columbite, fergusonite, samarskite.
Odergardsleten.....	Columbite.
Kjaersund.....	Do.
Dillingoe.....	Yttrotantalite.
Huggeneskilen.....	Columbite.
Grevsrud.....	Do.
Hullings.....	Do.
Sameja.....	Do.
Aveno Fiord.....	Do.
Fuglevik Fiord.....	Do.
Berg (Rode Parish) near Moss.....	Columbite, mossite, yttrotantalite, fergusonite.
Trøndelag County:	
Lorebo near Aker.....	Columbite.
Kragerø region.....	Euxenite.
Hvaler Island south of Frederickstad.....	Do.
Vestfold County:	
Larvik.....	Pyrochlore, polymignite.
Fredricksvarn.....	Polymignite.
Telemark County:	
Kragerø.....	Columbite.
Lovo near Brevik.....	Pyrochlore.
Vest Agder County:	
Morefjaer.....	Euxenite, priorite.
Risør region.....	Fergusonite.
Alve on Tromo Island.....	Euxenite.
Rostol near Arendal.....	Columbite.
Tvedestrand.....	Euxenite.
Helle near Arendal.....	Fergusonite, euxenite.
Naskul.....	Fergusonite.
Hampemyr on Tromo Island.....	Do.
Vest Agder County:	
Svinor.....	Euxenite.
Christiansand.....	Columbite.
Hitteroe Island.....	Eschynite, polycrase, blomstradine, priorite, euxenite.
Eitland near Lister.....	Priorite.
Vest-Agder og Fjordane County:	
Jolster.....	Euxenite.

The commercial potentialities of these pegmatites was not reported.

PORTUGAL

Columbite-tantalite occurs with cassiterite or wolframite in placers and pegmatites in several districts in northern Portugal.

Columbite and tantalite are associated with cassiterite and gold in alluvium at Caminha,

Viana do Castello district. Waste piles from mining cassiterite contain appreciable columbite. Columbite has been recovered at the Fontainhas mine since 1952 and is also a byproduct of the Cabração mine.

Columbite concentrates or columbium-tantalum-bearing tin slags are byproducts of tin smelters at Mangualde, Vizen district; Belmonte, Castelo Branco district; and Amaranite, Porto district.

SPAIN

Tantalite and columbite are associated with tungsten minerals in pegmatites and in derived placers in the Orense, Pontevedra, and La Coruna districts of Galicia Province and in the Duero River basin in the Salamanca district of Leon Province. In Leon Province, thorium minerals are reported to be closely associated with the tantalite and columbite.

SWEDEN

At Alno Island, Klingerfjorden Bay, Västernorrland, an alkaline rock complex and associated carbonatites contain pyrochlore and cerian perovskite. The area, examined during and after World War II for commercial potentialities, consists of a series of nepheline syenite, nepheline melanite, nepheline aegerite, jacupirangite, orolivine, and magnetite rocks. Large carbonatite bodies and breccias with fenitized halos are distributed in arcuate patterns similar to those at Söve, Norway. The are local concentrations of pyrochlore and cerian perovskite with apatite, magnetite, fluorite, and barite in the carbonatites. Large low-grade resources are believed to be present, but quantitative reserve data have not been released.

Pyrochlore, aeschynite, yttrotantalite, fergusonite, and euxenite are reported in association with alkaline rocks and in pegmatites in western Sweden, particularly near Ytter in Jämtland. Similar minerals are reported with syenite and in pegmatites near Falun, Kopparberg in central Sweden. At Finbo, near Falun, tantalite occurs in pegmatites with cassiterite and yttrocerite. At Brodho, near Falun, tantalite occurs with wolframite and cassiterite in pegmatites.

U.S.S.R.

There is little detailed information on Russian columbium-tantalum deposits, but many occurrences have been reported.

Columbium and tantalum are recovered with uranium from betafite mined since 1957 at Slyudyanka near the southern end of Lake Baikal.

The Ilmen Mountains near Kusa in the Ural Range have long been a source of ti-

tanium ore. Among the minerals reported are ilmenorutile, fergusonite, and samarskite, all of which contain columbium or tantalum. Ilmenorutile is also reported at Selyankino in the Urals, and tantalite and columbite are reported in various pegmatites in the Ural region.

Tantalite-bearing pegmatites are also reported in the Kirgizia pegmatites on the north slope of the Turkestan Range, in the Georgian S.S.R., in the Ukraine, near Akmolinsk, Kazakhstan, and in the Slyudyanka-Khamar-Daban Range east of Lake Baikal.

Pegmatites containing abundant heavy black minerals, including tantalite, are reported on the Kola Peninsula in the Khibiny Range and Lovozero area. Perovskite is reported at Africanda, Kola, Peninsula. Columbite has been reported to have been mined in the Vishnevi Mountains.

Loparite and murmanite occur in alkaline intrusive rocks of the Lovozero massif, Kola Peninsula. The loparite contains 0.35 to 10.82 percent Cb_2O_5 and 0.64 to 0.67 percent Ta_2O_5 ; the murmanite contains 6.65 to 7.71 percent Cb_2O_5 and 0.50 to 0.56 percent Ta_2O_5 . The rock is said to contain an average about 4 percent loparite-murmanite. This columbium deposit, now being worked, may be the world's largest. Other alkaline complexes with associated carbonatites have been reported in the eastern part of the Sayan Mountains north of Tannu Tuva in south-central Siberia, in two areas in the northwestern part of the Siberian platform, in two areas near the Aldan River northeast of Lake Baikal in the southern part of the Yakut A.S.S.R., and at Zhdanov near the Azov Sea.

Reserves have not been reported but are very tentatively estimated to total at least 5 million tons of contained Cb_2O_5 and 250,000 tons of contained Ta_2O_5 .

Asia

BURMA

Tantalite-columbite occurs in placer deposits near Tavoy in the Tennasserim. Information is not available concerning size and grade.

CEYLON

Columbite and fergusonite are minor constituents in pegmatite dikes and placers. One columbite was reported to contain 67.35 percent Cb_2O_5 and 7.30 percent Ta_2O_5 . The gem gravels in Ratnapura district are reported to contain annerodite, fergusonite, samarskite, and tantalite.

CHINA

Extensive areas containing tantalite-bearing pegmatites are reported in remote parts of Sinkiang Province in western China.

INDIA

Columbium-tantalum-bearing pegmatites occur in several Indian states, but production has been small. In Andhra, samarskite has been reported at Griddalur and Sarlapalle; tantalite, samarskite, and fergusonite, at Sankara, Chaganam, and Razulapad.

In Bihar, columbite-tantalite has been produced near Jhā Jhā in the Monghyr district and occurs at Pichhli in the Gaya district and at Hazaribagh. Columbite, aeschynite, and euxenite are found at Othara, Tovla, Thiruvalla, and Erania Taluk in Kerala.

Tantalite has been reported in Madras: Palni, Madura district; Semmalai Hill Trichinopoly district; Mungilkaradu Hill Vayampati; Kadavur; Devepatnam; and Munampalli.

In Mysore, columbite and samarskite have been produced near Yelowal, Shettihalli, and Tagadur, and occur near Masti.

In Rajasthan columbite-tantalite has been reported at Ajmer-Merwara, Lohagal-Mawali, Mewar, Lakola, Sonaina, Sangua, Ke and Bir. Several thousand pounds have been produced from the various localities.

Reserve data are not available.

JAPAN

Both pegmatites and placers in the kawa-Machi area in Fukushima Prefecture contain columbite, euxenite, samarskite, fergusonite, and ishikawaite. Fergusonite is reported in pegmatite and placer deposits in the Suisho-Yama area. A sample of columbite from Fukushima contained 78 percent $(\text{Cb}, \text{Ta})_2\text{O}_5$.

Fergusonite occurs in a pegmatite at Nakatsugama in the Naegi-Machi area of Gifu. Placers worked for tin in the area contain fergusonite and samarskite.

Columbite occurs in pegmatites in the northern foot of Mount Tsukuba, Ibaraki Prefecture. Fergusonite is reported in pegmatites near Hakatumura, Tokushima Prefecture.

KOREA

Pegmatites and placers containing columbite, fergusonite, and samarskite are reported in Kangwon-do, Cholla-Pukto, Chugwon-do, and Hwanghae-do Provinces. At least 1 ton of columbite was produced in Kangwon-do. A sample of typical

TABLE 25.—Analyses of Malayan columbium-tantalum minerals, percent

	Cb ₂ O ₅	Ta ₂ O ₅	FeO	SnO ₂	TiO ₂	SiO ₂	Insoluble (including SnO ₂)	MgO	Al ₂ O ₃	MnO	Rare-earth oxides
Struverite, Perak (46).....	6.90	35.96	8.27	2.62	45.75	0.20
Columbite concentrate, Johore ¹ ...	28.50		25.78	35.90	1.76	(³)	(³)	3.33
Do ¹	18.72		21.75	34.78	13.04	(³)	(³)	3.70	(³)

¹ U.S. Consulate, Penang, dispatch 58, Mar. 13, 1953.

² Total (Cb, Ta)₂O₅.

³ Trace.

cobbed ore contained 58.1 percent Cb₂O₅ and 15.6 percent Ta₂O₅.

MALAYA

Columbite-tantalite occurs widely with cassiterite in alluvial and eluvial deposits derived from weathering of pegmatites and granite, particularly at Bakri in Johore and Semilang in Kedah. In addition, columbium or tantalum occur in rutile, cassiterite, ilmenite, and metamict complex multiple-oxide minerals.

The columbite-tantalite, averaging a columbium-tantalum ratio of 4:1 and a specific gravity of 5.5, may run as high as 5 to 30 percent of the cassiterite content in a given property (table 25). Some columbite-tantalite is recovered from the cassiterite; as a separated concentrate that averages up to 75 percent (Cb, Ta)₂O₅; some, intimately intergrown with cassiterite eventually reports in the tin slags at the Penang and Singapore smelters; and some, especially if the content of columbite is so low that no recovery is attempted, ends in the so-called among, which are dumps consisting mostly of ilmenite. In a few instances, such as at Kampar, the columbite has a much higher specific gravity, indicating a greater than average tantalum content. A mineral resembling tapiolite, which is high in tantalum, frequently is associated with the columbite.

Columbian rutile has been found at Larut, Kampar, Semilang, and elsewhere in Kedah, Perak, Selangor, and Johore. It is usually intergrown with columbite, cassiterite, or ilmenite.

Some cassiterite concentrates contain appreciable columbium and tantalum as ultrafine intergrowths, possibly tapiolite judging from the roughly 1:1.5 to 1:1 average tantalum-columbium ratio, compared with the 1:4 ratio existing in Malayan columbites. Various analyses of Malayan cassiterite have indicated up to 6.9 percent Ta, all of which reports in smelter slags.

Some Malayan ilmenites containing up to 1 percent columbium are of no commercial significance in 1960. Minor quantities of euxenite, betafite, pyrochlore-microlite, and other complex minerals have been recognized principally with the columbite from Bakri and Semilang.

The tin slags contain about 3 to 8 percent (Cb, Ta)₂O₅ in the ratios previously stated for cassiterite concentrates. A WO₃ content of 18 to 60 pounds per ton is a processing problem but might prove to be a valuable byproduct.

Total reserves of columbium-tantalum-gravels are not established but are believed to be large.

Struverite is reported in Perak, where the mineral and cassiterite occur in a kaolinized granite. Reserves are not known, but the occurrence may be a potential source of tantalum (30).

PAKISTAN

Columbite and tantalite in unknown quantity occur in pegmatites in the Hazara district of the Northwest Frontier Province.

THAILAND

Columbian rutile occurs in a placer at Takuapa in southern Thailand. The mineral is reported to contain 17.5 percent (Ta, Cb)₂O₅. Tantalite-columbite and euxenite comprise 2 percent of the tin tailings of the Kamunting Tin Dredging Co., Ltd., in the Phang-Nga section of southern Thailand.

TURKEY

Quartz veins in the Darmanlar area south-east of Izmir contain columbite associated with arsenopyrite.

Africa

ANGOLA

Columbite-tantalite and columbotantalates of rare earths and thorium are reported in alluvial deposits in the Lobito-Benguela Plateau area, associated with gold and cassite-

rite. Samarskite is present in pegmatites in the Malange area north of Benguela Province.

CAMEROUN

Columbite-bearing alluvial deposits have been reported in the M'Bako River, south of Betare-Oya, and near Yaounde. Tantalite is reported in alluvium near Betare-Oya, in the Lom River near Dschang, and in a tributary of the Mba River.

Reserves, not fully tabulated, are arbitrarily estimated at 100 tons of Cb_2O_5 and 50 tons of Ta_2O_5 .

CONGO, REPUBLIC OF THE (FORMERLY BELGIAN CONGO), AND RUANDA-URUNDI

What probably constitutes the world's largest tantalite reserve occurs in a zone of scattered pegmatites extending for over 700 miles in a north-south direction through Katanga, Kivu, and Orientale Provinces and Ruanda-Urundi. The mineral usually contains approximately equal amounts of tantalum and columbium pentoxides. The deposits occur both in the pegmatites and in nearby gravels derived from decomposition of the pegmatites. The most common associated pegmatite minerals are feldspar, spodumene, quartz, mica, and cassiterite. Most of the tantalite-columbite is a byproduct of tin mining. The deposits in gravel have been to a large extent exhausted and each year a greater share of production is from pegmatites. Most of the pegmatites are in mica schists close to granite bodies. Some pegmatites are as much as 15 kilometers long, but more commonly they are 2 to 5 kilometers long. The usual width is from 50 to 400 meters. Weathered material extends 1 to 80 meters deep.

Tantalum-columbium deposits have been reported in 77 places. Most of them have been mined at one time or another. The localities are as follows:

	<i>Katanga</i>
Lukasasi Basin	Luvua River
Manono	Muika
Bukena	Luizi River
Kitotolo	Lukulu River
Kiambi	Kibumba River
	<i>Kivu</i>
Kaozi	Kampulu
Kikalae (Maniewa)	Matelemana
Moemba (Maniewa)	Mubilina
Kamilanga	Kalukangala
Penekoka	Iseke
Nzovu	Lundjulu
Kasina	Mwana
Kanzuzu	Kasika
Bokumu	Tshiganda
Penekoka	Mumba
Matemba	Numbi

Kabunga	Punia (Manew)
Bokumu	Ibanga
Utú (Ulinda River Area)	Nkenge
Kalima	Kanzoro
Makalapongo	Utú (Lowa River area)
Kobokobo	Idambo
Kalamuli	Etaetu

Orientale

Teturi	Mabuka
Liha (Elota)	Enehe (Etembo)
Ngawe	Samuda

Ruanda-Urundi

Ndora	Katumba
Mogere	Bijojo
Ntungá	Kavumu
Mbuye	Buranga
Mayaga	Mont Kibingo
Kaganda	Borne
Kirengo	Ndiza
Luhanga	Kababa
Kinuoni	Lemera
Gikaya	Sinda
Bibale	Shori
Bugalula	Tshubi
Rukoma	

Since 1939 the eastern Congo-Ruanda-Urundi area has been the world's most important source of tantalum. Most of the reserves are in two pegmatite laccoliths, Manono and Kitotolo, near Manono in Katanga. These pegmatites have surface exposure of 1.2 million and 1.35 million square meters and average about 5.5 pounds of cassiterite and 0.16 pounds of tantalite-columbite per cubic meter.

The deposits, fairly extensively drilled to a depth of about 125 meters, are believed to average about 400 meters in total thickness. The total reserves for the Republic of the Congo and Ruanda-Urundi are estimated to be about 50,000 tons of Cb_2O_5 and 50,000 tons of Ta_2O_5 , almost half near Manono (table 26).

Tantalite-columbite concentrate, tin-tantalite-columbite concentrate, and tantalum-columbium-bearing tin slag (by Geomines only) are produced in the country. These products average 55, 10, and 15 percent, respectively, combined Cb_2O_5 and Ta_2O_5 . The concentrates have contained about two-thirds of the columbium and tantalum produced to date; the slag, the remainder.

A carbonatite-bearing complex has been reported in the Lueshe Valley, northeastern Kivu, near the southwest end of the Lake Edward Rift about 40 kilometers south-southwest of Lake Edward. The complex has a central core of cancrinite-syenite and an outer ring of aegerine sovite. The sovite deposit, said to contain several hundred thousand tons of pyrochlore and associated apatite, averages 2 percent Cb_2O_5 . The property has been leased by Somibuki (Société

TABLE 26.—Analyses of Republic of the Congo and Ruanda-Urundi columbo-tantalites, percent (65)

Location	Ta ₂ O ₅	Cb ₂ O ₅	TiO ₂	SnO ₂	FeO	MnO
Mundjulu, Kivu ¹	7.49	65.00	1.21	0.13	11.28	2.45
Punia, Kivu.....	13.3	64.3	1.1	.1	15.8	6.1
Buranga, Ruanda-Urundi.....	14.1	63.5	.4	.4	9.0	11.7
Mogere, Ruanda-Urundi.....	25.5	54.8	.8	.8	13.6	4.4
Mundjulu, Kivu.....	32.86	47.10	.38	.07	8.83	10.43
Inda, Ruanda-Urundi.....	32.23	46.29	.60	.23	3.52	15.2
Kikalale, Kivu.....	50.79	19.39	1.44	3.73
Moemba, Kivu.....	51.3	28.7	.4	.6	10.3	9.4
Mundjulu, Kivu.....	57.5	25.7	.5	.1	9.6	6.0
Manono, Katanga ²	58.16	16.58	.91	1.60	4.58	12.80
Cshuhi, Ruanda-Urundi.....	66.9	16.1	.4	.6	5.0	9.9
Moemba, Kivu.....	71.55	9.62	.64	1.04
Mundjulu, Kivu.....	76.69	5.74	.97	.13	14.06	0.84
Punia, Kivu ³	77.36	2.50	14.68	1.10

¹ 4.3 percent R₂O₃ of yttrium group.

² 0.21 percent ZrO₂.

³ Tapiolite.

Minière de Nyambuki). Minor amounts of other columbium-tantalum minerals other than columbite-tantalite have been reported in the placers and pegmatites. These are tapiolite, struverite, thoreaulite, samarskite, euxenite, aeschynite, and formanite.

CONGO, REPUBLIC OF (FORMERLY FRENCH CONGO)

Columbite occurs with gold in placers in the Middle Congo. It has been reported in the Mayombe region, 100 kilometers southwest of Franceville, and as a byproduct in the Mayoko region. No reserve has been established, but nominally 50 tons of contained Cb₂O₅ can be credited.

GUINEA

Pyrochlore is reported in nepheline syenite, with barkevikite and aegerite, on Los, Kassa, and Rouma Islands off the coast of Guinea.

KENYA

Four carbonatite deposits have been reported in Kenya; the one at Mrima has been explored in detail, and large reserves of pyrochlore were established.

The Mrima Hill deposit contains pyrochlore, monazite, and barite in veinlike carbonatite bodies associated with the Jombo alkalic rock complex apparently intruded into the Duruna sandstone. The bedrock is covered by a thick mantle of soil and decayed rock, which has been enriched in pyrochlore. The upper 22 feet of soil contains 34 million tons of ore, averaging 0.7 percent Cb₂O₅, or a total Cb₂O₅ content of 238,000 tons.

The other carbonatites, essentially unevaluated, are at Homa, Rangive, and Ruri.

Columbite is reported as a sparse accessory mineral in various pegmatites. No attempt has been made to recover or evaluate potentialities for recovery of this mineral.

MALAGASY REPUBLIC (FORMERLY MADAGASCAR)

Columbite, euxenite, and betafite have been produced and other minerals such as ampagabeite, strueverite blomstrandite, euxenite, tantalite samarskite, samiresite, and hatchettolite occur in various Malagasy pegmatites and in eluvium resulting from their decomposition. Reported localities with the particular columbium-tantalum minerals present are

Ampangabe, near Miandrarino.....	Columbite, strueverite, ampagabeite.
Ambatofotsikely, near Miandrarivo.....	Columbite, ampagabeite.
Tangafeno, south of Betafo.....	Blomstrandite.
Ambolotara, near Betafo.....	Euxenite, betafite.
Manjaka, Sahatsny Valley.....	Columbite.
Manendryles, near Vinanikani.....	Samarskite.
Samiresy, southwest of Laka Tritriva.....	Euxenite, samiresite.
Mount Bity.....	Hatchettolite.
Antanamaloza, south of Antsirabe.....	A columbate-tantalate of uranium.

Other localities where pegmatites contain columbium-tantalum minerals are Antandrokomby, Antsongombato, Maharitra, Tsilaisina, Lake Alaotia, and Tongafeno.

The Malagasy pegmatites are notable for the many complex minerals containing columbium and tantalum in combination with uranium and rare-earth elements and for the notable absence of cassiterite except as a very minor accessory (table 27). In struc-

TABLE 27.—Analyses of Malagasy columbium-tantalum minerals, percent (5)

Mineral and origin	Cb ₂ O ₅	Ta ₂ O ₅	FeO	MnO	TiO ₂	SnO ₂	UO ₂	UO ₃	U ₃ O ₈
Columbite, Ampangabe.....	64.60	12.60	15.00	7.30	71.15	0.40			
Struverite, Ampangabe.....		10.14	15.64		4.90			19.40	
Ampangabeite, Ampangabe.....	34.80	8.90			10.80	.30		18.10	
Blomstrandite, Tangafeno.....	23.30	28.50			19.20		¹ 16.50		
Euxenite, Ambolotera.....	33.70		1.10		18.30	.30	¹ 26.60		
Betafite, Ambolotera.....	34.80				1.42		8.70		
Samarskite, Manendrika.....	43.60	11.15	5.40		6.70	.10		21.20	
Samiresite, Samiresy.....	45.80	3.70							14.15
Hatchettolite, Mount Bity.....	² 56.61								
	(Y, Er) ₂ O ₃	(Ce, La, Di) ₂ O ₃	ThO ₂	CaO	H ₂ O	PbO	BeO	Total	
Columbite, Ampangabe.....								99.90	
Struverite, Ampangabe.....								96.93	
Ampangabeite, Ampangabe.....	4.00	0.60						73.40	
Blomstrandite, Tangafeno.....	.30	2.50						83.80	
Euxenite, Ambolotera.....	18.38	2.44	1.54	2.27				95.03	
Betafite, Ambolotera.....	.90	1.60	1.30					82.80	
Samarskite, Manendrika.....	9.50	5.05	1.05	2.45	11.44		7.30	99.76	
Samiresite, Samiresy.....		.74					2.37	85.54	
Hatchettolite, Mount Bity.....			.96					74.09	

¹ Total UO₂ and UO₃.

² Total Cb₂O₅ and Ta₂O₅.

ture and size the deposits resemble those of Brazil.

Nepheline syenites in northwestern Malagasy have been correlated with the alkalic rocks in eastern Africa, but no carbonatites have been reported in Malagasy (5).

MOROCCO

Tapiolite and columbite occur in five pegmatites near Iguerda and Timrharhrine south of Marrakech in the Atlas Mountains (62). Associated minerals include muscovite, beryl, manganese and iron phosphates, hematite, tourmaline, garnet, and apatite. The commercial potentialities of these deposits for columbite-tantalite production are not known. Columbite from Iguerda is reported to contain about 39 percent (Cb, Ta)₂O₅ with a columbium-tantalum ratio of 1.84:1. Tapiolite from Angorf-North No. 1 near Timrharhrine contained:

Oxide:	Percent	Oxide:	Percent
Ta ₂ O ₅	62.5	SiO ₂2
Cb ₂ O ₅	21.2	TiO ₂	(¹)
FeO.....	15.5	SnO ₂	(¹)
MnO.....	.1		
		Total.....	99.5

¹ Trace.

MOZAMBIQUE

Mozambique, for years producing a relatively small quantity of tantalite, greatly increased its production in 1957, 1958, and 1959. Production came from several of the numerous placer and pegmatite deposits in Zambezia, Manica, Sofala, and Niassa Provinces. Many pegmatites have not been explored at all.

Columbite-tantalite was a byproduct of beryl, lepidolite, and native bismuth occurring in high-perthite zones of pegmatites, and columbite, tantalite, and samarskite was the principal valuable material recovered from placers. Monazite, zircon, ilmenite, magnetite, garnet, and gold also are found in the placers. These alluvial and eluvial deposits, generally not exceeding 1.5 meters in thickness, average 0.5 to 1.0 pound of columbite-tantalite per ton. Reserves are unknown.

Tantalite-columbite is disseminated with cassiterite and lepidolite in greisen in the Umtabi, Odzi, and Tsungwesi reserves near the Southern Rhodesia border.

An alkalic-rock complex and associated carbonatites, at Muambe near Lupata Gorge, contains pyrochlore, but commercial potentialities have not been evaluated. A similar occurrence is at Chuara about 12 miles north of Lupata Gorge.

NIGERIA (31,88)

Columbite is found in the Younger granite series, which crops out over a 585-square-mile area, in the pegmatites associated with the older granites, and in placer deposits derived from weathering of both rock types. In the Younger granite series and derived alluvial deposits, the mineral is invariably at or close to the columbite end of the columbite-tantalite isomorphous series. In the Older granite pegmatites, the mineral may range from columbite to almost pure tantalite.

In the Younger granites, fine crystalline columbite is widely disseminated as a primary accessory constituent in biotite gran-

TABLE 28.—Columbite content of granite phases of the Jos-Bukuru complex, Nigeria (88)

Granite	Range of columbium content, lb. cu. yd.	Average columbium content, lb. cu. yd.
Jos.....	0-0.05	0.02
Delimi.....	.05- .15	.10
N'gell.....	(¹)- .25	.18
Bukuru.....	.10- .40	.30
Forum.....	.30-6.0	.50
Rayfield-Gona.....	.40-7.57	.50

¹ Trace.

ites. The Rayfield-Gona and Forum biotite granite phases of the Jos-Bukuru complex are the richest in columbite (table 28). They average 0.5 pound of columbite per cubic yard (average weight = 2,400 lb.) and in rich streaks may exceed 7.5 pounds of columbite per cubic yard. The granites are particularly susceptible to decomposition, and weathering may exceed a depth of 100 feet. Extensive areas of decayed granite have been preserved by a thin cover of laterite and by middle Tertiary fluvial and volcanic deposits.

In the alluvial deposits columbite occurs mixed with cassiterite from weathered lodes or greisen in the granite. About two-thirds of the tin leases contain columbite and in about 1 percent of these, the ratio of columbite to cassiterite is at least 1 to 3. At Odegi, Tin and Associated Minerals, Ltd., have large reserves of columbite, cassiterite, and hafnium-rich zircon in soil, clay, and rotten rock.

Columbite-tantalite has been found with cassiterite in quartz-microcline-albite-muscovite pegmatites associated with Older granite series in several provinces. The most important deposits are near Wamba-Jemoa, Plateau Province, and Egbe, Kabba Province.

RHODESIA AND NYASALAND, FEDERATION OF

NORTHERN RHODESIA.—Small pockets of betafite occur with mica in pegmatites near Fort Jameson in the Lundazi District. The extent and grade of these deposits are not reported, and their commercial potentialities are believed to be small.

Several large pyrochlore-bearing carbonatites are reported. At Nkumbwa Hill, 15 miles east of Isoka, pyrochlore, monazite, magnetite, and apatite occur in a 1-square-mile volcanic plug, which includes carbona-

tites and which is surrounded by fenitized sediments. Reserves are undisclosed but believed to be large. Average grade of the deposits is reported to be 0.25 percent Cb_2O_5 . The pyrochlore mineral is exceptionally high in Cb_2O_5 , averaging 73.46 percent.

At least four carbonatite areas occur in the Feira District about 150 miles from Lusaka. One of the areas, Nachombwa Hill, is three-quarters of a mile in diameter. All samples contain pyrochlore, and the entire hill is radioactive, but reserves and grade are not reported. Monazite, manganese, barite, and vanadium also are reported present.

NYASALAND.—Pyrochlore occurs in carbonatites and alkalic intrusive rocks of the Chilwa series. The pyrochlore is found in ringlike complexes at Chilwa Island, in Lake Chilwa (21); Tundulu, near the south short of Lake Chilwa; Songwe, south of Lake Chilwa near the Mozambique border; Nkalonje, about 4 miles south of Lake Chilwa; Kangankunde, on the west side of the Shire River valley about 50 miles west of Lake Chilwa; Nsengwa, about 22 miles southwest of Kangankunde; and Salambidwe, west of Chikwawa.

Prospecting of Chilwa Island, which contains the most promising deposits, began in 1952. Minerals of economic interest on the island include pyrochlore, apatite, manganese and iron ores, radioactive minerals, and fluorite. There are several million tons of 0.21 percent Cb_2O_5 ore proved in residual soils on the island, and even larger resources are indicated in both soil and in the carbonatites themselves. The deposits are controlled by Rhodesian Selection Trust, and total reserves have not been made public. An average analysis of Chilwa Island pyrochlore contains 64.1 percent $(Cb, Ta)_2O_5$.

Pyrochlore, columbite, and betafite occur in pegmatite and aplite dikes near Tambani.

SOUTHERN RHODESIA (79).—Tantalum and columbium minerals have been produced in small quantities from Southern Rhodesia for many years.

Tantalite, microlite, and simpsonite are byproducts of cassiterite and lepidolite mining in the Bikita area. Tantalite and simpsonite are usually associated with tin in pegmatites and derived eluvial deposits. The microlite occurs with lithium mica in greisen.

Conditions similar to those in Bikita are found in the Enterprise tin field east of Salisbury. The tantalite occurs with tin in both pegmatites and placers in the Enterprise-Victoria area.

TABLE 29.—Analyses of Southern Rhodesia columbium-tantalum minerals, percent (79)

Mineral	Ta ₂ O ₅	Cb ₂ O ₅	SnO ₂	SiO ₂	TiO ₂	ZrO ₂	Al ₂ O ₃	Fe ₂ O ₃	FeO	MnO
Bikita tantalite	71.10	5.04	2.54	7.20	(¹)	(²)	1.05	(¹)	0.69	10.00
Bikita microlite	74.36	4.78	(²)	1.20	(²)	(²)	.5677	(²)
Bikita simpsonite	60.01	6.05	.42	8.01	(¹)	0.05	22.58	0.11	.82	.03
Victoria tantalite	67.5	11.00
Conwar Concession mangantantalite	69.50	3.90	.33	4.00	12.95
	BeO	MgO	CaO	Na ₂ O	K ₂ O	H ₂ O+	H ₂ O-	P ₂ O ₅	F	Total
Bikita tantalite	0.24	1.34	0.46	0.02	99.68
Bikita microlite	(²)	(²)	11.68	5.13	0.47	.24	.02	0.30	0.58	100.09
Bikita simpsonite	(¹)	.32	.39	.23	.19	.27	.55	(¹)	100.03
Victoria tantalite
Conwar Concession mangantantalite

¹ Trace.² None.

Tantalite occurs also in greisen in the Oclyi gold belt and the Mtoko district, and in the pegmatites of the Kamativi tin field. Columbite is reported in the pegmatites of the Miami mica-beryl field. Euxenite is reported to occur widely in the Beitbridge area on the Rhodesia-Union of South Africa border (table 29).

Three carbonatite bodies are known at Shawa, Doriowa, and Chishanya in the Sabi Valley. No columbium-tantalum mineralization, however, has been reported.

SIERRA LEONE

The columbite, occurring in placers near Pujehun and in the Sula Mountains and the Kanguri Hills, was derived from weathering of quartz veins, and a few tons were mined. Reserve figures are not available.

Ilmenorutile occurs in placers extending 5 miles along the Tonkolite River. The deposits are 40 to 350 feet wide and 4 to 6 feet thick. A concentrate produced contained 2.3 percent Cb₂O₅. There are no reserve data.

SOMALI REPUBLIC

Small quantities of columbite-tantalite have been mined from pegmatites scattered over a 10-square-mile area in the Henwina Valley near Madera. Columbite is also reported northwest of Hargeisa and near Daranta Shabelah.

SOUTH-WEST AFRICA

Pegmatites occur in abundance in the Karabib-Omaruru and Orange River areas of South-West Africa. The pegmatites are

found in areas of Precambrian crystalline rocks and range in composition from barren ones containing only quartz, plagioclase, perthite, muscovite, biotite, garnet, and tourmaline to those that also contain beryl, lepidolite, amblygonite, columbite, tantalite, spodumene, cassiterite, topaz, microlite, triphylite, bismuth, and other minerals of possible economic interest. Both homogenous and zoned pegmatites are found, but only those with zonal structure are rich enough to be worked (11).

In the Karabib-Omaruru district columbite-tantalite was recovered in small quantities; beryl was a byproduct chiefly of lepidolite and petalite. The minerals occur both in the pegmatites and in derived alluvium. The tantalite concentrate contains about 65 percent Ta₂O₅.

In the Orange River district, tantalite has been recovered with beryl and bismuth from the Tantalite Valley and other areas near Warmbad. The minerals are recovered from the pegmatites and from the derived alluvium in the valley bottoms. Tantalite concentrate averaged as high as 80 percent Ta₂O₅. These pegmatites, cutting amphibolite and diorite, are rich in tantalite and bismuth, but pegmatites on the other side of the valley, in granitic gneiss, carry only columbite and are not economical to mine.

Large deposits of tantalite were said to have been discovered in 1957 near Karasburg, about 100 miles north-northwest of Warmbad.

SWAZILAND

Tin placers and pegmatites worked for lithium minerals near Embabaam in the Forbes Reef area have yielded byproduct columbite-tantalite. Euxenite and eschynite are also present.

In the Mbabane district yttriotantalite is recovered with monazite as a byproduct of tin placer mines. Old tin-mine dumps are also reworked to yield these originally neglected byproducts. Columbite is also reported in stream gravels near the headwaters of the Mbuluzi.

TANGANYIKA (18,33,34)

The largest columbium deposit reported in Tanganyika is the Mbeya, which forms part of Panda Hill, a bold knoll flanking the rift valley between Lake Rukwa and Lake Nyasa, 13 miles southwest of Mbeya. An alkalic rock complex, including carbonatites, intrudes and metasomatizes Precambrian gneisses. The deposit was discovered in 1950 during geologic mapping by the Tanganyika Geological Survey. Various lithologic types contain the following percentages of Cb_2O_5 : Fenitized (metasomatized) gneiss, 0.1 to 0.4; sovite, 0.2 to 0.4; dolomite, up to 0.2; carbonatized dikes, 0.1 to 0.7; residual soil, 0.06 to 1.80 averaging 0.62; transported soil, 0.22 average.

Reserves at Panda Hill are in residual soil and bedrock deposits. The measured reserve includes 14 million tons of ore containing 0.34 percent Cb_2O_5 and 3.8 million tons of ore containing 0.79 percent Cb_2O_5 . In addition, there is an inferred reserve of 63 million tons averaging 0.3 percent Cb_2O_5 . By 1962 the company plans to complete a plant capable of producing between 2,000 and 5,000 tons of 50 to 70 percent Cb_2O_5 concentrate annually. A pilot mill was placed in operation in 1957. Concentrate is shipped to the Arnhem works of N. V. Billiton Maatschappij for processing (table 30).

Columbium-bearing carbonatite also comprises Singeri Hill, a few miles from Panda Hill, but the area has not been examined in detail. Pyrochlore and monazite occur in sovite dikes at Wigu Hill near Kisaki south of the Uluguru Mountains. The deposit has been prospected by New Consolidated Goldfields, Ltd. Pyrochlore has also been reported in a carbonatite dike at Maji ya Weta Hill immediately south of Kisaki. Carbonatite is reported from Mount Kerimasi on the west wall of the Gregory Rift Valley, 16 miles south of Lake Natron. A pyrochlore-bearing limestone is found in the gorge of the En-

TABLE 30.—Analysis of Panda Hill (Mbeya) pyrochlore, Tanganyika

	Percent	
Cb_2O_5	55	—66
Ta_2O_55	— 2.2
CaO		13.6
Alkali as Na_2O	2	— 3.4
Rare earths including ThO_2		6.9
Equivalent ThO_205— 2.45
TiO_2	1	— 4.1
FeO3	— 2.3
MgO1	— .3
SiO_2	1.7	— 2
P_2O_51	— 2.6
U_3O_8025
Al_2O_35
F		1.8
WO_301

¹ Average, 63 percent.

danok River southeast of Ufiome Mountain. The rock contains 0.4 percent pyrochlore, which in turn contains 71.2 percent Cb_2O_5 . Carbonatite at Oldonyo Dili near Ngualla, north of Lake Rukwa in the Chunya District, assayed 0.1 to 0.2 percent Cb_2O_5 and has been prospected by Anglo-American Prospecting Company (Africa), Ltd. The area was inaccessible so that a new access road had to be built before prospecting could begin.

Columbite and tantalite have been reported with mica in pegmatite in the Aitchoscarp area north of Mbulu.

UGANDA

Past production has been limited to tantalite and columbite from pegmatites and quartz veins; interest in recent years however, is centered upon pyrochlore-bearing carbonatites. One, in the Sukulu Mountains, was being developed in 1960 (86)

At Sukulu, south of Tororo, Sukulu Mines, Ltd., a subsidiary of the Uganda Development Company, is developing a carbonatite-bearing alkaline rock complex to recover apatite and pyrochlore concentrates. The area is covered by a thick mantle of red residual soil, which contains reserves totaling more than 200 million tons (130 million proved, 70 million indicated) averaging 0.25 percent Cb_2O_5 and 13.1 percent P_2O_5 . Baddeleyite, ilmenite, magnetite, tremolite, and zircon are other valuable associated minerals. A pilot plant with a capacity of 100 tons of ore per day began operating in July 1957. In 1960 arrangements for financing £7 million to erect commercial-scale mining and milling facilities and to build a railroad were being attempted. Planned operations were to mine at a 9,000-ton-per-day rate and to produce

400,000 tons of apatite concentrate and 3 million pounds of pyrochlore concentrate annually. The final pyrochlore concentrate was expected to contain about 55 percent Cb_2O_5 .

Pyrochlore-bearing carbonatites are also reported in the Tororo Hills and at Bukusu, northeast of Tororo, but their commercial potentialities have not been evaluated. Other carbonatites, whose mineralogy has not been described, are exposed at Mount Elgan near the Kenya border, Moruangeberr in the Napak area, and at Sekululu, northeast of Tororo. Perovskite is reported in an alkalic rock complex at Budeda, northeast of Tororo. No carbonatite rocks are reported at this locality, and the occurrence has not been evaluated for its possible economic value.

Euxenite and bismutotantalite have been reported at Gamba Hill, Kagadi, and West Buganda in the Buganda district.

In the past, most columbite and tantalite has been mined from quartz veins and pegmatites in the Western Province, an extension of deposits in the former Belgian Congo. These minerals, associated with cassiterite and gold, have been produced mostly from pegmatites; small quantities came from small, erratic quartz veins. Principal localities are Dwata, Kashozo, Ngoma, Burama, Jemubir, Mingoma, Roberts Reef, Kayonza, Kakanena, Kamwezi, Migera, Kigero, Kisunu, Ruguma, Kisheki, and Nampeyo Hill. Tantalite shipments range from about 35 to 80 percent Ta_2O_5 . Columbite contains 40 to 68 percent Cb_2O_5 and 9 to 36 percent Ta_2O_5 .

UNION OF SOUTH AFRICA (63,72,75)

Tantalite is reported with lithium, bismuth, and manganese minerals in pegmatites at Richtersveld near Steinkopf, Namaqualand.

Pyrochlore has been reported 15 miles north of Eshowe in Zululand. The amount of ore is not reported, but grade is said to be 0.05 percent pyrochlore, which contains 72.2 percent Cb_2O_5 and 1.8 percent Ta_2O_5 .

In the Transvaal, tantalite occurs at Doornhook Farm in the Nylstrom tin district and has been mined since about 1937 from a pegmatite at Pietersburg. Columbite-tantalite is also reported in a pegmatite at Palakop in the Letaba area.

Carbonatites are reported at eight localities in the Transvaal, but only one has been reported to contain columbium-tantalum minerals. In the Glenover farm area, Waterberg district, apatite-rich carbonatites occur with perknite, agglomerate, syenite, and fenitized rocks. Radioactive columbite is a

reported accessory in this 4.5-square-mile area. Dikelike carbonatite bodies cut the diamondiferous kimberlite breccia pipe at the Premier mine. At Loolekop in the Palabora district, a circular alkalic complex consists of a carbonatite core surrounded by a serpentine, magnetite, apatite, vermiculite, calcite breccia and outer rings of pyroxenite and syenite. The carbonatite is a low-grade copper deposit and contains considerable uranothorite. The breccia is being quarried for the apatite content, and baddeleyite is a potential byproduct. Columbium minerals have not been reported. Other carbonatites are reported in the Goudini Farm area, Marico district; Tweerivier, Brit district; Kruidfontein, Brit district; Spitskop, Sekumiland district; and at Magnet Heights.

Oceania

AUSTRALIA (50)

Tantalum concentrates have been produced from numerous placers, pegmatites, and veins, mostly in Western Australia. Before World War II, Australia was the source of 90 percent of the world supply of tantalite. New production from other countries and exhaustion of some of the richer Australian deposits relegated Australia to a relatively minor position with output usually less than 25 tons annually.

WESTERN AUSTRALIA.—The principal deposits are at Strelley, Tabba-Tabba, Pilgan-goorra, and Wodgina in the Pilbara goldfields in northwestern Australia and in the Greenbushes district in southwestern Australia (16). Although the tantalite and other minerals occur in pegmatites and derived alluvium and eluvium, most of the known reserves in the latter two types have been worked out. At Wodgina, tantalite and microlite occur with beryl in a dike and in eluvium. Mill feed averages 3 pounds of tantalite per ton and runs as high as 25 pounds per ton. The principal pegmatite deposit has been mined to a 65-foot depth over a length of 2,300 feet. Massed crystals of tantalite in the albite zone weighed up to 90 pounds. This deposit has had the largest production in Australia. At Strelley, tantalite, microlite, and tapiolite occur with cassiterite and beryl in pegmatite and eluvium. Tantalite crystals range up to the size of a pea. The main pegmatite measures 80 by 600 by 2,000 feet. Similar deposits are reported 2 miles north of Strelley. At Tabba-Tabba, tantalite, simpsonite, microlite, and columbite occur with cassiterite and beryl in a pegmatite and associated eluvium. The main dike has been mined to depths up

to 30 feet over a length of 1,400 feet. Recovery at Tabba-Tabba is reported to have been as high as $6\frac{1}{4}$ pounds of tantalite concentrate per cubic yard. At Pilgangoora, columbite, tantalite, and tapiolite occur with cassiterite and spodumene in pegmatite dikes and derived eluvial deposits. Production has been small. At Greenbushes, tantalite, columbite, and stibiotantalite occur in granitic dikes, eluvium, and alluvium. Tantalum ore was first discovered in Western Australia at this place. Masses of tantalite up to 80 pounds have been found. Total production probably has been exceeded by Wodgina, Tabba-Tabba, and Strelley. Tantalite, columbite, and ixiolite occur in pegmatite at Londonderry south of Coolgardie, where crystals of tantalite weighing up to 5 pounds have been recovered as a byproduct of microcline feldspar. Many other tantalum-columbium occurrences are recorded in Western Australia:

Kimberly Division: Mount Dockrell, Collier Bay.

North West Division:

Pilbara District:

West Wodgina, Kangan, Stannum, Moolyella, Mt. Fransisco, Abydos, Woodstock, Hillside, Ely's, Cooglegong, Split Rock.

Gascoyne District:

Yinnietharra, Mooloo Downs, Dalgety Downs.

Murchison District:

Coodardy, Poona.

East Murchison District:

Kathleen Valley.

Central Division:

Coolgardie District:

Victoria Rocks, Gibraltar, Ubini, Larkinville, Logan's Find.

Yilgarn District:

Holleton.

Southwest Division:

Melville, Lake Moore-Lake Monger, Jumperding, Mt. Dale, Balingup (Ferndale), Smithfield, Ravenshorpe.

Eucla Division:

Dundas, Norseman, Fraser, Range, Bellinger (Israelite Bay).

Very small production has been reported from some of these localities.

OTHER.—Elsewhere in Australia tantalum and columbium minerals have been reported in the Northern Territory and Queensland. Tapiolite was produced from a pegmatite in 1952 near Bynoe Harbor, 70 miles east of Darwin, Northern Territory. In the Darwin district, small quantities of tantalite have been obtained as a byproduct of cassiterite placer mining near Mount Finnis and West Arm. Gravels contained up to 1 pound of tantalite per cubic yard. Tantalite, with associated radioactive minerals and muscovite mica, is reported in pegmatites in Harts Range, 120 miles east of Alice Springs, Northern Territory. In Queensland insignificant quantities of tantalite and fergusonite are reported in the Cloncurry district. Total Australian reserves are unknown, but probably contain 100 to 1,000 tons of contained Ta_2O_5 .

NEW ZEALAND

Tantalum minerals in small amounts have been reported in sands at the following localities:

Otorukua Point, southern Westland.....Tapiolite in beach sands.
Paringa River, southern Westland.....Tapiolite, kobeite in sands and silts.
Collingwood, Nelson.....Yttrotantalite.
Southern Nelson.....Tantaliferous cassiterite.
Northern Westland.....Tantaliferous cassiterite.
Addison's near Westport, Nelson.....Unspecified tantaliferous mineral in black sand.

SECONDARY SOURCES

Tin Slags

Columbite and tantalite occurring with cassiterite usually report with the tin concentrate. When such concentrate is smelted any columbite or tantalite present passes into the slag.

TABLE 31.—Columbium-tantalum-bearing tin slags

Country of Origin	Average grade, percent		Cb_2O_5 - Ta_2O_5 average ratio	Estimated annual production, tons
	Cb_2O_5	Ta_2O_5		
Congo, Republic of the (formerly Belgian Congo).....	6.5	6.5	1 : 1	1,000
Portugal.....	7.0	6.7	1.04 : 1	200
Nigeria.....	14.0	4.0	3.50 : 1	2,000
Malaya:				
Penang.....	3.67	2.59	1.42 : 1	4,000
Singapore.....	2.82	1.46	1.93 : 1	3,000

Tin slags, mainly from the Republic of the Congo (formerly Belgian Congo), have been an important source of columbium and tantalum. Output is largely dependent on the scale of tin mining operations and of course, on whether the columbite-tantalite is concentrated separately in the mill. Approximately one-fourth of the metal content of the columbium-containing material consumed in the United States in 1957 was from tin slags, compared with three-fourths from mineral concentrates.

Only the slags from the Congo and Portugal have been important sources of columbium and tantalum to 1960, but the slags from Nigeria and Malaya are potential sources. Table 31 summarizes data on these slags.

At least 100,000 tons of low-grade slag are estimated to have accumulated at the Malayan smelters.

Scrap

In-plant, or new scrap, is recirculated into the process. Old tantalum scrap, such as tube components and chemical equipment, is bought by scrap dealers for resale to processors, and under some conditions is accepted as partial payment for new tantalum equipment. Because of its high cost, tantalum scrap is too valuable to waste. The quantity reclaimed each year in the United States totals several tons, but exact data are lacking. Columbium is a much newer commercial product than tantalum, and to 1960 there has been little demand for columbium scrap.

CHAPTER 4.—TECHNOLOGY

EXPLORATION, MINING, AND BENEFICIATION

As previously mentioned, the four major types of columbium-tantalum ore occurrences are (1) in pegmatites, (2) in granitic rocks, (3) in alkalic igneous rocks and their associated carbonatites and fenitized haloes, and (4) in residual and placer deposits derived from weathering of the rock types mentioned.

Many techniques are employed in mining columbium and tantalum minerals. Mining methods can range from native hand labor to giant dredges and highly mechanized open pits, depending upon local labor and power supply and the geologic setting of the deposit. Underground mining is restricted to relatively shallow depths; the workings are serviced by inclined and vertical shafts. Typical concentration flowsheets consist of gravity separation based on differences in specific gravity of the ore minerals followed by magnetic and electrostatic separation. For more complex ores such as pyrochlore with associated pyrite and apatite, procedures usually evolve around gravity devices followed by flotation of pyrite, magnetic separation, and then dissolution of apatite in nitric acid. There are so many mining operations in so many parts of the world that it is not feasible to describe them all. Instead a few typical areas are described.

Bear Valley, Idaho, Euxenite-Columbite-Monazite Placer

Porter Brothers Corp. operated a unique placer mine in Bear Valley, Idaho, to recover columbium, tantalum, uranium, and other valuable elements. This mine was one of the world's largest sources of columbium-tantalum-bearing ores from 1957 through 1959.

HISTORY

The placer deposits were discovered in 1950, when prospectors noticed radioactivity in the area. Porter Brothers Corp. leased the ground that year. A preliminary examination by engineers of the Bureau of Mines focused the attention of the U.S. Atomic Energy Commission (AEC) on the area and resulted in AEC financing of a Bureau of Mines exploratory churn-drilling program in 1951 and 1952. Porter Brothers Corp. drilled the area during the same period. By 1953,

Porter Brothers Corp. felt that commercial potential had been proved and approached both Defense Materials Procurement Administration (DMPA) and the AEC for financial assistance in developing the property. A loan and agreements to purchase 1,050,000 pounds of columbium-tantalum oxides and byproduct uranium oxide were approved in 1954, and mining and concentration began in 1955. By 1957 it was estimated that the mine was one of the three largest sources of columbium-tantalum ores in the world. By 1960 the stipulated quantity of oxides had been delivered to the Government, and since private ore buyers were not found in sufficient volume, the mine was expected to close.

EXPLORATION AND SAMPLING

The usual method of exploration was by churn-drilling to bedrock, which may have been as much as 120 feet below the surface. Samples, taken at regular intervals from more than 100 drill holes, were augmented by 34 samples weighing from 50 to 100 pounds each, taken from surface test pits. Drill samples were taken every 2½ feet and two combined samples represent each 5-foot section of drill hole. Samples were dried and bagged before they were shipped to the laboratory. The samples were screened, jigged, and tabled in the laboratory to produce a black sand concentrate, from which the total pounds of black sand per cubic yard were computed. Composite samples to maximum dredgable depths were prepared for each drill hole and submitted for radiometric, petrographic, and chemical analyses.

MINING

Two bucket-line dredges mined at a combined rate of 8,000 cubic yards per day. The larger craft had 6-cubic-foot buckets; the smaller had 4¼-cubic-foot buckets. Between them, they recovered about 200 tons of black sand concentrate daily. The large dredge can dig 55 feet below the waterline; the smaller dredge, 35 feet. The smaller dredge was used to skirt the perimeter of the deposit where depth of ground rarely exceeded its limit. The dredges operated 24 hours daily for about 9 months of each year. Severe weather usually prevented operations during January, February, and March.

The dredged gravel was washed in a trommel with ¾-inch openings. The oversize

gravel dropped to a stacker and was rejected as tailings, the undersize was impounded for distribution to jigging circuits. Each dredge had parallel starboard and port circuits consisting of rougher jigs, cyclones for dewatering the rougher hutch product, and cleaner jigs. The cleaner concentrate was pumped ashore periodically through a cable-suspended rubber hose to an overhead storage tank. Trucks were driven under the tank

to load concentrate which was transported to a concrete slab for dewatering while awaiting further truck transport to the Lowman beneficiation plant. Technical data on the operation are given in table 32.

State mining laws require Porter Brothers Corp. to rehabilitate the land after their dredges have passed. Tailing piles are leveled by bulldozer; topsoil is stripped from in front of the dredges by a self-loading

TABLE 32.—*Technical data, Porter Brothers Corp., Bear Valley, Idaho, dredging operation (52)*

(Courtesy of Mining World)

Deposit characteristics:	
Total gravel dredged per day.....	7,000–8,000 cubic yards
Average black sand content of deposit.....	50 pounds per yard
Euxenite content of deposit.....	0.3–1.5 pounds per yard
Total black sand concentrates recovered.....	150–200 tons per day
Specific gravity of black sand.....	3.5–5.9
Specific gravity of gravels.....	2.7

	6-Cubic foot dredge	4¼-Cubic-foot dredge
DREDGE SPECIFICATIONS		
Ladder length.....	103 ft. 10 in. c. to c. suspension.....	62 ft. 0 in. c. to c. suspension.
Number of buckets.....	106.....	68.
Bucketline speed.....	24.3 buckets per min.....	28.4 buckets per min.
Size of buckets.....	6 cu. ft.....	4¼ cu. ft.
Bucket-line drive.....	125 hp.....	75 hp.
Type bucket-line drive.....	V-belt and double reduction gears.....	V-belt and double reduction gears.
Ladder hoist.....	125 hp. double-drum.....	25 hp. single-drum.
Maximum ladder angle.....	48°.....	45°
Hull, number of pontoons.....	36.....	26.
Hull, size.....	127 ft. 2 3/16 in. by 58 ft.-1 5/8 in. by 8 ft.-1 in.	90 ft. 5 1/2 in. by 44 ft. 0 in. by 7 ft. 1 1/2 in.
Stacker length.....	80 ft. 0 in.....	122 ft.-0 in.
Stacker belt speed, width.....	307.8 f.p.m. 28 in. belt.	345 f.p.m. 30 in. belt.
Trommel size.....	5 ft. 10 1/2 in. i.d. by 29 ft. 10 7/8 in.....	5 ft. 0 in. i.d. by 30 ft. 6 in.
Trommel speed.....	11 r.p.m.....	9.98 r.p.m.
Screen size.....	3/8 in. round openings.....	3/8 in. round openings.
Storage tank (concentrate).....	60-ton capacity.....	40-ton capacity.
Spuds.....	Single (starboard rear).....	Single (starboard rear).
Width cut.....	130 to 150 ft.....	65 to 100 ft.
Digging depth below water.....	55 ft.....	35 ft.

DIGGING PROCEDURE AND OPERATION

Means of anchorage.....	Two 7/8 in. bow lines, two 7/8 in. stern lines, D7 Cat, stern spud.	Two 7/8 in. bow lines, two 7/8 in. stern lines, two old spuds, stern spud.
Bucket life.....	Manganese steel bucket lips; approximately 9 months of life; not rebuilt; install new lips.	Manganese steel bucket lips; approximately 9 months of life; not rebuilt; install new lips.
Means of advance.....	Stern lines and spud.....	Stern lines and spud.

JIG CHARACTERISTICS AND OPERATION ¹

Size shot.....	3/8 in. steel shot.....	3/8 in. steel shot.
Other bedding.....	-1/2 in. by 1/4 in. magnetite.....	-1/2 in. by 1/4 in. magnetite.
Bed depth.....	2 in.....	2 in.
Jig adjustment.....	Required periodic adjustment by jig operator, depending on black sand.	Required periodic adjustment by jig operator, depending on black sand.
Jig stroke rougher.....	1 1/4 in.....	1 1/4 in.
Jig cycles.....	125 per minute.....	125 per minute.
Cleaners:		
"A" cell.....	1/2 in. stroke 225 cycles.....	1/2 in. stroke 225 cycles.
"B" cell.....	3/4 in. stroke 170 cycles.....	3/4 in. stroke 170 cycles.

¹ The jig settings are varied depending upon black sand content.

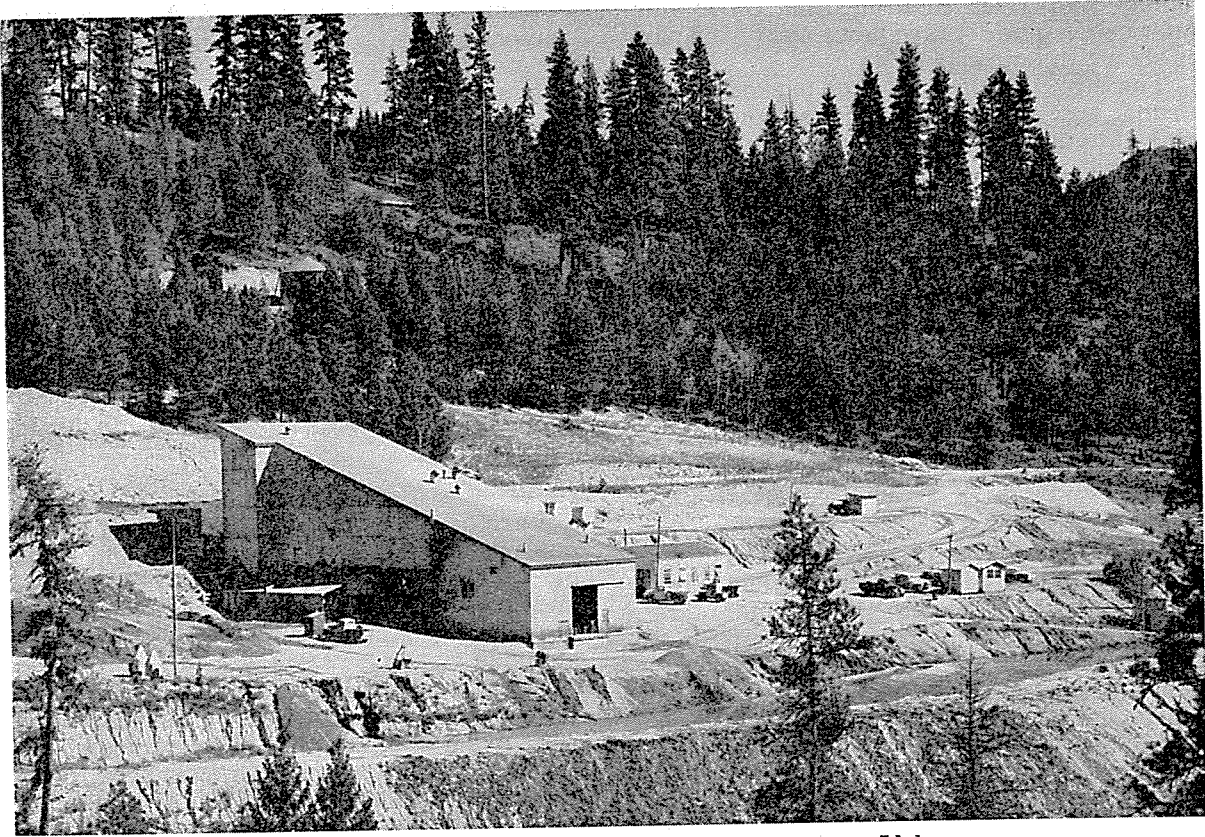


FIGURE 4.—Mill of Porter Brothers Corp. at Lowman, Idaho.
(Courtesy of Porter Brothers Corp.)

scraper and then spread over the leveled tailings. Finally, reseeded restores the land to its original grazing land character. To preserve the character of stream waters in the valley, Bear Valley Creek has been temporarily diverted around the dredging ground, and water from the dredge ponds is treated with Separan 2610 and sulfuric acid to settle the slimes. The water courses disturbed by the dredging must be replaced by the company on meander lines; the pool structure must be conducive to conservation and recreational use. In a few years it will probably be difficult to tell that the area has been dredged (52).

BENEFICIATION

Dredge concentrate was trucked to Lowman, Idaho, where it was processed in a 150- to 200-ton-per-day concentration and separation plant (fig. 4). Basically, the treatment involved a wet circuit and dry circuit. To summarize the procedure, concentrate from the plant surge pile was taken into the wet part of the plant, where it was screened and rod-milled to minus 0.064-inch, deslimed,

attrition-scrubbed to improve the surface properties of the mineral particles for subsequent separation, and passed through a Stearns MWI magnetic separator, where 99 percent of the magnetite and a small fraction of the ilmenite were removed from the pulp and piped to a stockpile. The nonmagnetic part was dried in a Cedar Rapids 48-inch-diameter, 16-foot-long, oil-fired rotary kiln. In the dry circuit a Stearns MDP electromagnetic separator pulled off an ilmenite-magnetite middling product missed in the wet circuit. Next, Stearns KT 50 induced-roll magnetic separators pulled garnet and the rest of the magnetic ilmenite from the concentrate. Then, the ore was treated in Carpco HT 460 high-tension separators to produce columbite-euxenite and monazite concentrates. The columbite-euxenite fraction was treated on Stearns crossbelt magnetic separators, tabled, and further upgraded on another crossbelt where columbite was separated from euxenite at the same time. The monazite concentrate was treated on Stearns K 30 induced rolls to reject quartz and zircon, treated on a crossbelt separator,

tabled, and given final cleaning on another crossbelt. The detailed flowsheet of the plant is shown in figure 5.

The euxenite product was packed in steel drums, trucked 70 miles to Boise, and shipped by rail to the separation plant of Mallinckrodt Chemical Works, St. Louis, Mo. Monazite and columbite also were shipped to Boise to await sale. Ilmenite, magnetite, garnet, and quartz-zircon were stockpiled at Lowman to await markets (15).

Nigerian Decomposed Granite and Alluvial Deposits

Nigerian columbite-cassiterite deposits are in the central Nigerian Bauchi Plateau region. They are the most important source of columbite in the world.

HISTORY

The potential mineral wealth of the district was first indicated about 1900, when paying quantities of cassiterite ("tinstone") were found in the Delemi River by agents of the Niger Company. During the next few years alluvial cassiterite was found in several provinces, but it was not until hostile Africans had been pacified in 1909 that commercial tin production began. Columbite was identified in the tin placers as early as 1916 but was considered valueless at the time. Recovery of byproduct columbite started in 1933, and as prices gradually rose, more and more columbite was recovered. In 1945 columbite was discovered in situ as a primary accessory mineral in the younger granites of the Jos-Bukuru complex. After this discovery, mining was begun to recover columbite from the granite saprolite.

EXPLORATION AND SAMPLING

Geologic reconnaissance is used to locate areas worthy of detailed examination. For evaluation of surficial materials, hand-panning of samples is the accepted technique. Where drilling is required to determine the thickness of the pay zone or to reveal the location of hidden ore, two methods are used. For ground less than 100 feet deep, use of hand-operated screw augers is favored. The drill holes are placed at 200-foot intervals in a square grid pattern. The holes are uncased with samples taken only from the tip of the tapered auger. Deeper ground is explored by motor-driven percussion drills, with holes usually placed at 400-foot centers on a square grid pattern. Casing is used in soft ground, and solid core samples are obtained by using an earth socket placed on the end of the drill string.

Samples obtained during drilling are split, screened, sized, deslimed, dried, and treated by magnetic separators to produce a low-grade columbite concentrate. The concentrate is leached 48 hours with hot concentrated hydrochloric acid to decompose iron oxides and ilmenite and to demagnetize magnetic zircon. The residue is then dried, and a final columbite concentrate, containing also cassiterite, magnetite, xenotime, and monazite, is produced by magnetic separation. The columbite and cassiterite content of the final concentrate and the cassiterite content of the two nonmagnetic fractions are then estimated by microscopic examination. The results agree with chemical analyses within 5 percent.

MINING

Various mining techniques are employed, depending upon local topographic and geologic factors and upon the availability of water and power. Mechanized operations, however, have virtually replaced more primitive methods.

Small surficial deposits where water is not available may be worked by African labor with picks and shovels. The excavated ore is carried by head pan or calabash to the nearest water supply for panning or sluicing. If the ore wash is buried, overburden is stripped by hand labor. When the cassiterite-columbite lies deeper than the limit to which soil can be pitched by shovel, terraced excavations are made. Debris is returned to the pit before making the next cut. The African "tributers" work in gangs under a headman who sells the product and distributes the earnings to the men.

An alternate method of hand labor, used where a water source is available, is to shovel the soil into a series of ditches, which carry the ore to a sluice; the barren alluvium is diverted into other ditches, which lead to a waste area.

In most operations, overburden is stripped mechanically, using track-mounted electric shovels with up to 1-cubic-yard-capacity dippers or draglines. The excavated overburden is dumped to one side. The pay horizon may be mined in one of several ways. The ore can be loaded on trucks or trains by shovel for transport to the washing plant, or if water is available at the mine, hydraulic mining may be employed. The cassiterite-columbite wash from hydraulicking is channeled through ditches cut in the excavation or "paddock" floor to sluices sunk in bedrock, or if the sluiceboxes are on the rim of the excavation, the wash is directed to a sump

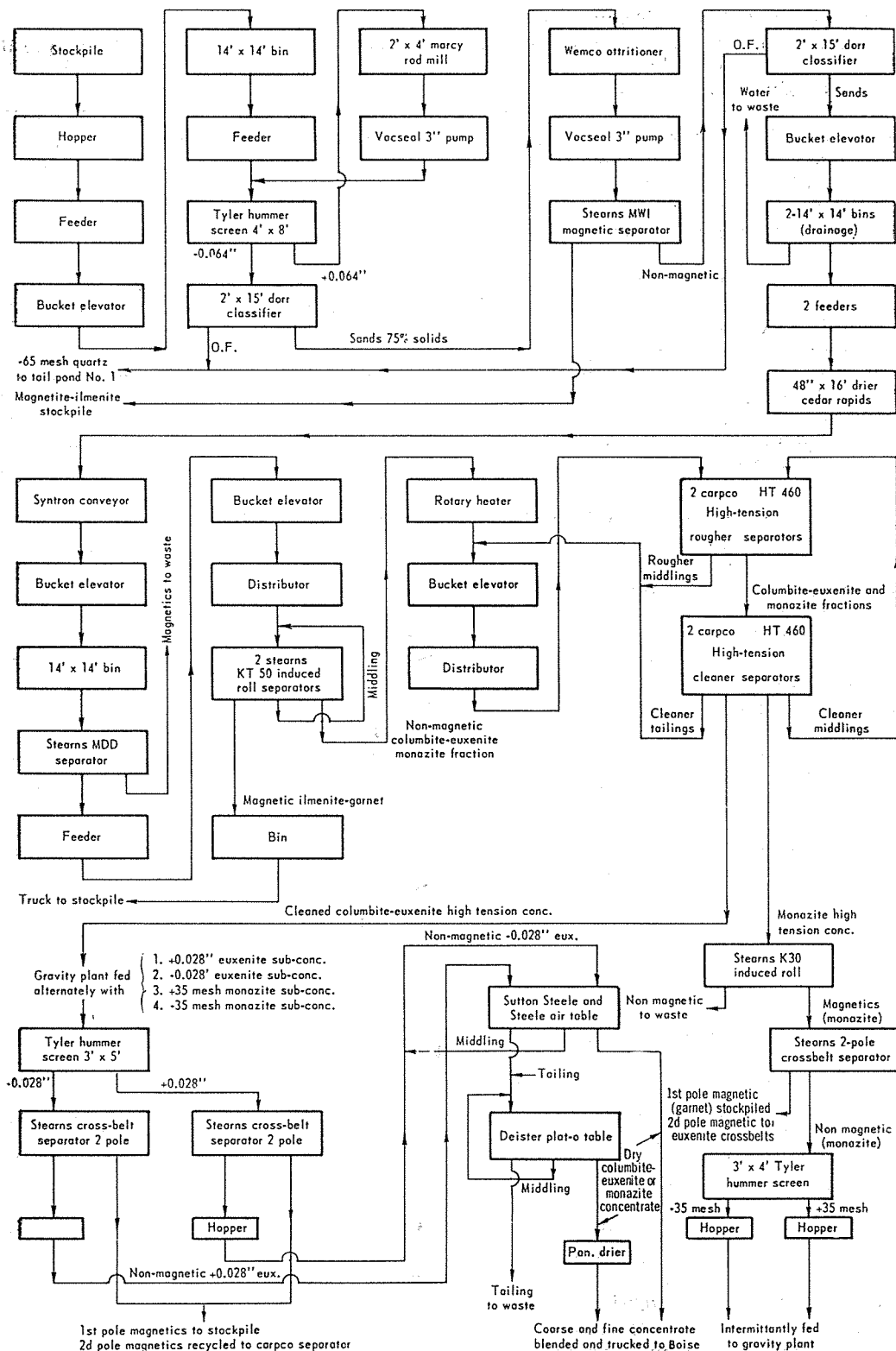


FIGURE 5.—Flowsheet, Porter Brothers Corp. Plant for Concentrating Columbite to Euxenite and Monazite. Capacity is 150 to 200 tons per day (15).
(Courtesy of Mining World)

season or passed to an oil-fired rotary drier before the columbite or tantalite is separated from the cassiterite.

Selective electromagnetic separation is used to remove the columbite-tantalite from cassiterite. The first step is to effect a rough separation of magnetic minerals (such as columbite, tantalite, iron oxides, ilmenite, tourmaline, and mica) from the cassiterite. A shaking table then is used to eliminate mica, tourmaline, some iron oxides, and other gangue minerals. Subsequently the rough columbite-tantalite is passed through two crossbelt magnetic separators. The first pass yields fractions consisting of tramp iron-magnetite, titaniferous magnetite-ferrousinginous tantalite, columbite-tantalite concentrate, and cassiterite-tantalite. The tantalite-columbite concentrate is passed through a second similar but smaller machine to produce the marketable columbite or tantalite concentrate. Recovery is 75 percent or more of the mineral in the ore. Tantalite or columbite passing into cassiterite fractions is not deleterious to succeeding tin-refining steps and eventually reports in tin-smelter slags.

Some producers market small quantities of columbite-tantalite-cassiterite concentrates without any effort to make a separation.

METALLURGY (23, 51, 64)

Extraction (81)

The first step in preparing of columbium or tantalum metal is rendering its components amenable to further treatment by decomposing the ore. The various methods for decomposing the raw materials are called extraction procedures.

CAUSTIC FUSION

The classical method for extracting columbium and tantalum from their ores (as illustrated by the Fansteel process used before 1958, fig. 6) is by fusion with hot sodium hydroxide. The concentrates are pulverized to minus 200-mesh powder in a hammermill or ballmill. The powdered ore is fused with sodium hydroxide in an iron pot or tube to convert the contained tantalum and columbium to sodium tantalate and columbate. The reaction takes place at red heat and is completed in a few seconds. The fused

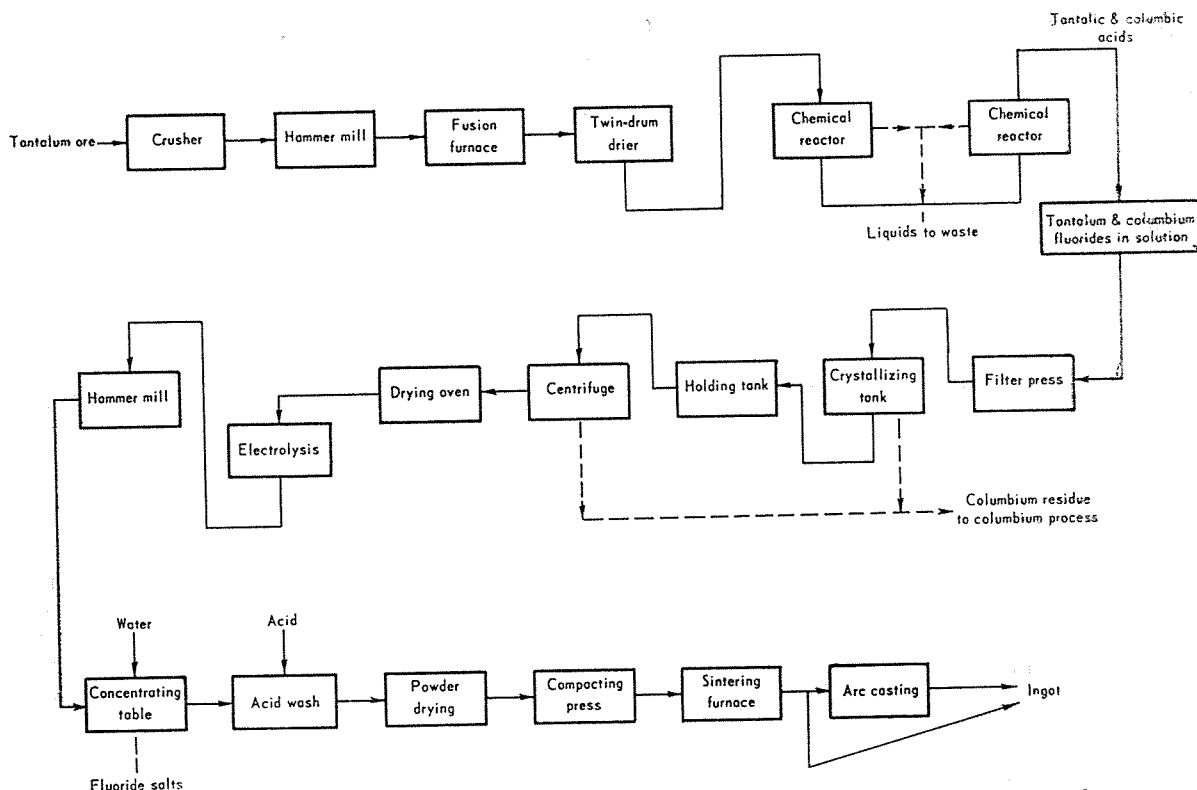


FIGURE 6.—Tantalum Flowsheet (before 1958) of Fansteel Metallurgical Corp., North Chicago, Ill. (64).

(Courtesy of Industrial and Engineering Chemistry)

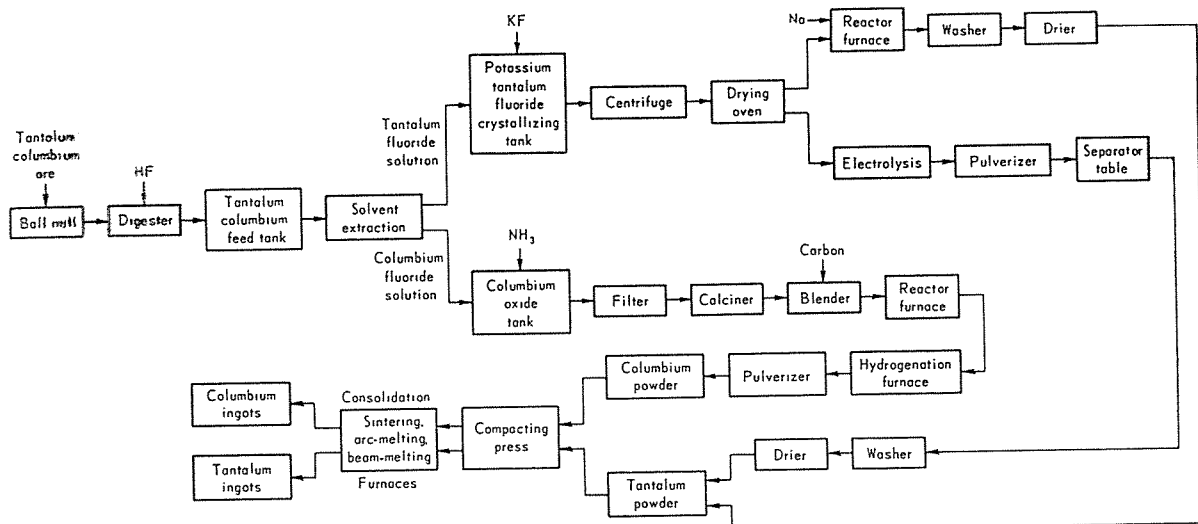


FIGURE 7.—Tantalum Flowsheet (since 1958) of Fansteel Metallurgical Corp., North Chicago, Ill.
(Courtesy of Fansteel Metallurgical Corp.)

product is flaked, leached with water to remove silica, and then boiled with hydrochloric acid in a rubber-lined vessel to remove iron, manganese, and other acid-soluble impurities and to convert the sodium salts of columbium and tantalum to hydrated oxides. Fusion techniques using other caustics have been successful in experimental trials but are not in commercial use.

ACID LEACHING

An alternate commercial method of decomposing the ore is to heat the pulverized concentrate with 98 percent sulfuric acid, with 70–72 percent hydrofluoric acid, or with a sulfuric-hydrofluoric acid mixture (fig. 7). This method requires a longer reaction time for complete decomposition of the ore and requires highly acid-resistant equipment. Many other acid-digestion schemes are described in the technical literature but are not in commercial use.

SMELTING AND CHLORINATION

The Bureau of Mines has extracted columbium and tantalum from low-grade materials such as tin slag by chlorinating a ferrotantalum-columbium alloy made from the slag. The alloy, made by smelting the ore or slag with coke in an arc furnace, is then chlorinated at 500° C. to produce mixed tantalum, columbium, and iron chlorides. A sodium chloride column can be used as a trap to form a nonvolatile, low-melting-point complex with the ferric chloride, removing it from the mixed reactor product. The tantalum and

columbium chlorides pass through the salt and are collected in a condenser. An alternate extraction and separation method used involved direct chlorination of a mixture of low-grade ore, carbon, and calcium fluoride.

OTHER METHODS

The Bureau of Mines has developed several other methods for extracting tantalum and columbium and the rare-earth compounds from ores and concentrates. In one of these, euxenite concentrate from Porter Brothers Corp. was processed by the Bureau of Mines to extract and purify the valuable metals by chlorination, solvent extraction, and hydrometallurgical techniques (48). Chlorination was used to convert the valuable constituents into chlorides and separate an iron-uranium eutectic, tantalum-columbium chlorides, titanium tetrachloride, and a rare-earth and thorium-bearing chlorination residue. Solvent-extraction techniques were used to separate the tantalum from the columbium. The rare-earth and uranium products were purified by hydrometallurgical methods. The titanium tetrachloride was 99-percent pure and did not require further treatment.

During chlorination 96 percent of the euxenite was converted to chlorides. Solvent extraction of hydrolized columbium-tantalum chloride separated high-purity columbium and tantalum oxides. Approximately 80 percent of the tantalum and 86 percent of the columbium were recovered as pure compounds after passing through the separation

system once. The rare-earth compounds in the chlorination residue were treated by hydrometallurgical procedures involving oxidation reactions and pH control, which recovered 84 percent of the rare-earth elements in a product that contained 59 percent yttrium oxide. A 99 percent uranium oxide product was obtained by processing the eutectic product from the chlorination (fig. 8).

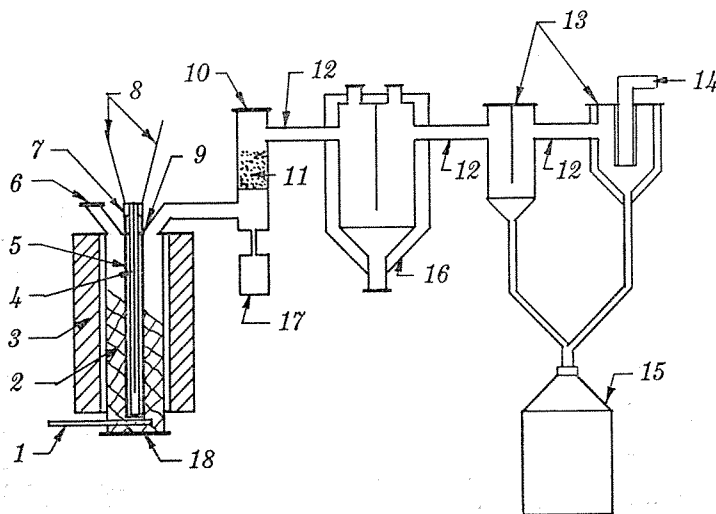
The Bureau of Mines also developed an extractive process applicable to submarginal Arkansas titanium deposits containing columbium. The process, technically but not economically feasible in 1960, consisted of reducing the mineral concentrate with coke at high temperature to form a carbide-suboxide sinter, chlorination at 400° to 500° C., and fractional condensation of the chlorides.

The chlorination procedure recovered 97, 83, and 87 percent of the titanium from brookite, rutile, and ilmenite sinters, respectively; simultaneous columbium recoveries on the same materials were 85, 50, and 87 percent. The resultant CbCl_5 contained small amounts of titanium and iron chlorides as impurities (56).

Separation of Tantalum From Columbium FRACTIONAL CRYSTALLIZATION OF THE FLUORIDES

The tantalum and columbium hydrated oxides contained in the slurry resulting from caustic fusion are dissolved by 70 percent hydrofluoride acid or anhydrous hydrogen

fluoride, and then the solution is heated to near its new boiling point. Potassium hydroxide, fluoride, or enough carbonate is added to the hot solution to produce K_2TaF_7 and $\text{K}_2\text{CbOF}_5\cdot\text{H}_2\text{O}$. When the solution has cooled, the pure potassium tantalum fluoride precipitates as needlelike crystals, leaving the 12 times more soluble potassium columbium oxyfluoride in solution for subsequent recovery (fig. 9). The K_2TaF_7 typically contains less than 1 percent impurities. The columbium solution contains impurities such as titanium, tin, tungsten, and the iron not removed in previous operations and is treated by boiling with potassium carbonate and then by cooling to crystallize the small quantity of remaining tantalum. The filtered solution is mixed with a concentrated solution of sodium hydroxide to precipitate hydrous sodium columbate; the tin, tungsten, and other impurities remain in solution. The precipitate, washed with water and sodium hydroxide, is decomposed with hydrochloric acid to produce a columbic acid slurry. This slurry is washed and dissolved in potassium hydroxide to form potassium columbate, which is purified by systematic fractional crystallization. The pure potassium columbate is then converted to pure hydrated columbium oxide by precipitation in hydrochloric acid. The hydrated oxide is washed and ignited to Cb_2O_5 . A typical analysis of such an oxide is



- 1 Chlorine inlet
- 2 Chlorination charge
- 3 Chlorinator
- 4 Split graphite resistor
- 5 Mullite electrode shield
- 6 Feed port
- 7 Water-cooled copper contact
- 8 Power cables
- 9 Electrode seal
- 10 Salt column
- 11 Sodium chloride
- 12 Transfer tubes
- 13 Water-cooled condenser
- 14 Noncondensable gas outlet
- 15 TiCl_4 receiver
- 16 Heated condenser
- 17 Eutectic receiver
- 18 Residue cleanout

FIGURE 8.—Euxenite Chlorinator.

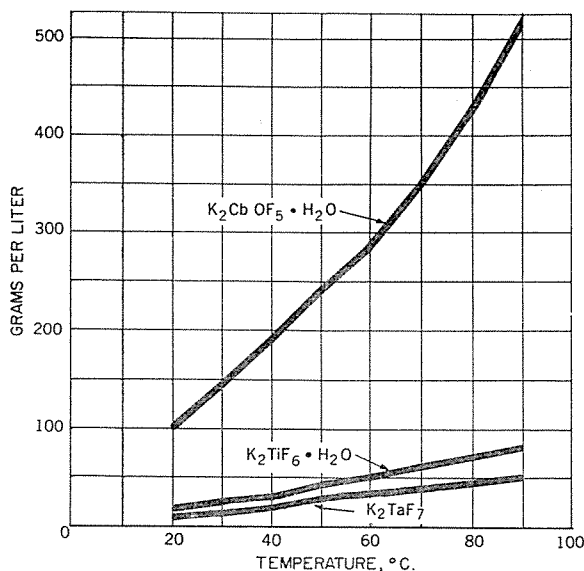


FIGURE 9.—Solubilities of Double Fluorides in 1 N Hydrofluoric Acid (81).

(Courtesy of Chemical Engineering Progress)

Oxide:	Percent	Oxide:	Percent
Cb ₂ O ₅	99.7	TiO ₂	0.05
Ta ₂ O ₅15	Fe ₂ O ₃05
SiO ₂05	ZrO ₂10

SOLVENT EXTRACTION (29, 42)

The Bureau of Mines at Albany, Oreg., developed a method for separation of columbium and tantalum compounds; this separation was readily adaptable to large-scale operation. The starting material was hydrated mixed oxides made from reacting water with the mixed chlorides produced from tin slag. These oxides dissolved in hydrofluoric acid or a hydrofluoric acid slurry of digested ore are its feed stock. Many variations of the basic process have been developed, but the Bureau of Mines has had the most success to date by using the hydrofluoric acid-sulfuric acid-methyl isobutyl ketone system (fig. 10). The basic principle of this system is the effect of pH on the solubility of the fluorides of the metals and impurities in methyl isobutyl ketone. Of the two major variations, one method dissolves the mixed hydrous oxides in weak acid in a rubber- or polyethylene-lined tank to form the aqueous feed stock. The tantalum values are extracted by contacting the feed with ketone in polyethylene counter-current towers or mixer-settler cells, then the acidity of the tantalum-free aqueous feed is increased and columbium values are extracted by fresh ketone. The alternative method is to use a strongly acid aqueous feed and extract columbium and tantalum simul-

taneously into ketone. The pregnant ketone is then back-extracted by acid at the proper concentration, which removes the columbium from the organic solvent. Multiple reextractions may be used in either process variation to produce very high-purity columbium and tantalum compounds. The separated solutions of pure tantalum or columbium fluoride are contacted with anhydrous ammonia in rubber- or polyethylene-lined tanks to precipitate the hydrated oxides of the metals. The Cb₂O₅ product, without using multistage purification steps, is more than 99.9 percent pure; a similar quality Ta₂O₅ usually takes at least two extractions. This process or its variation has been adopted by much of the industry as standard operating procedure (fig. 11).

FRACTIONAL DISTILLATION OF CHLORIDES

The Bureau of Mines has effected an experimental separation of tantalum and columbium chlorides by selective absorption of tantalum chloride by a countercurrent flow of a ferric-sodium chloride eutectic. Stauffer Chemical Co., Richmond, Calif., has installed a plant for producing separated chlorides from offgrade ferrotantalum-columbium by fractional distillation. Several other methods of effecting separation of anhydrous chlorides also have been described but have not received extensive attention.

ION EXCHANGE

Ion-exchange methods have been investigated as a means of separation by several industrial firms, and at the National Bureau of Standards ion exchange is used in analysis of tantalum and columbium ores. It has not been applied on a commercial basis to date because solvent extraction continues to be more economical, permits a higher ratio of metal to reagent, and lends itself more readily to continuous operation.

Reduction

The production of pure tantalum or columbium is difficult because the reactivity of the metals when heated makes avoiding contamination during processing difficult.

ELECTROLYSIS

Fansteel has been producing tantalum by the electrolysis of fused potassium tantalum fluoride since 1922. In 1960 it remains the most popular method of producing the metal. Pure K₂TaF₇ forms the electrolyte in an iron pot, which also serves as a cathode. A graphite rod serves as an anode (fig. 12). Tantalum pentoxide (or other oxides) is

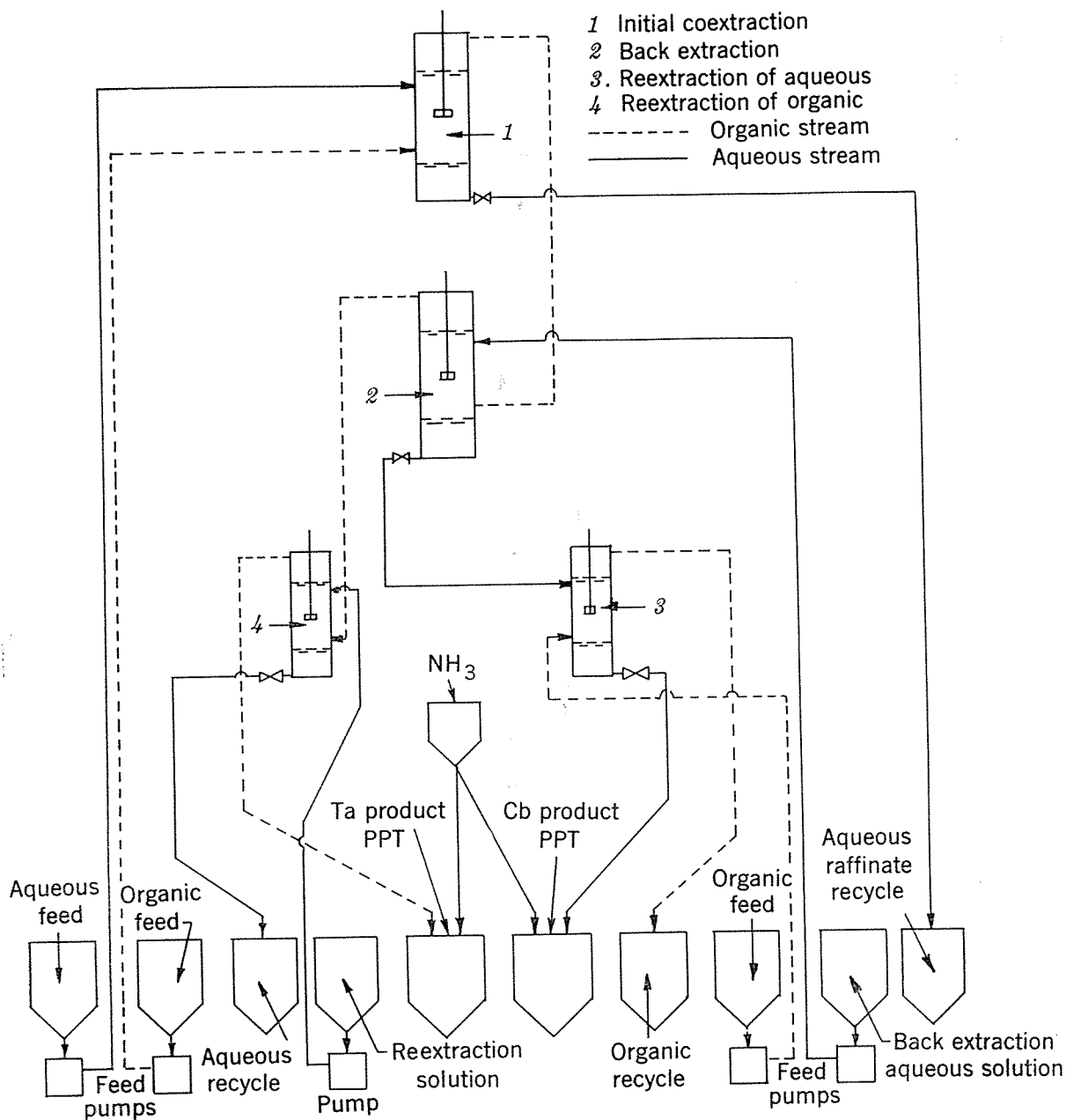


FIGURE 10.—Flow Diagram for HF-H₂SO₄-Methyl Isobutyl Ketone System for Separating Tantalum and Columbium.

added to the batch to prevent "anode effect" by depolarizing the anode surface. The charge is heated to about 900° C. and batch operated until the accumulated fine crystalline metal aggregate almost fills the pot. At the end of a run the pot is allowed to cool to room temperature. The solidified mass is removed from the pot, crushed, pulverized, tabled, and washed with strong acids to sepa-

rate the metal particles from residual salts. A typical analysis of electrolytic tantalum powder is tantalum, 99.8 percent; columbium, less than 0.05 percent; carbon, 0.12 percent; iron, 0.015 percent; and titanium, less than 0.01 percent. Typical mesh analysis is 30 percent plus 200-mesh, 40 percent minus 200- plus 400-mesh, and 30 percent minus 400-mesh.

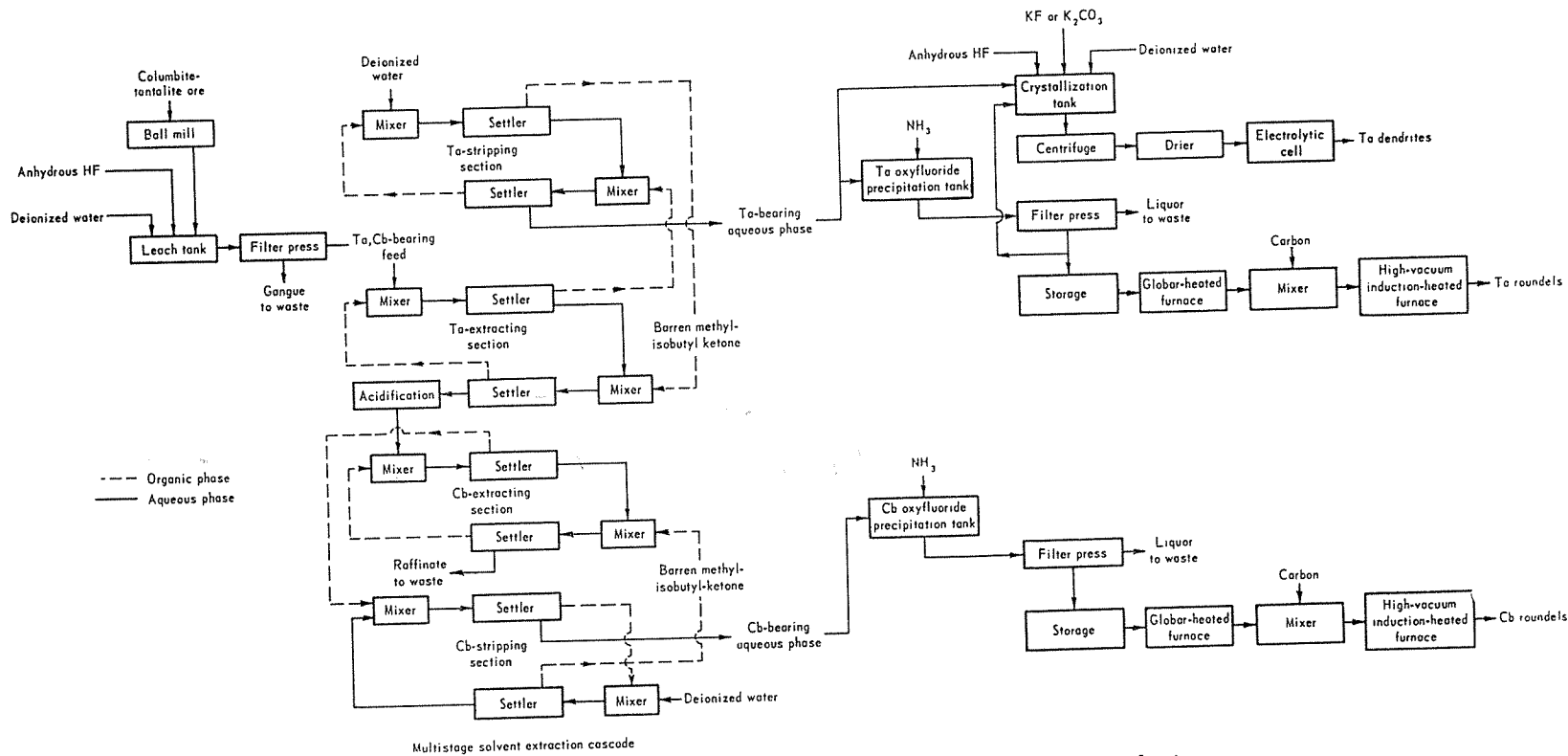


FIGURE 11.—Union Carbide Metals Co., Columbian-Tantalum Flowsheet.

(Reprinted with permission from *Chemical Engineering*, Nov. 3, 1958, copyright 1958, McGraw-Hill Publishing Co.)

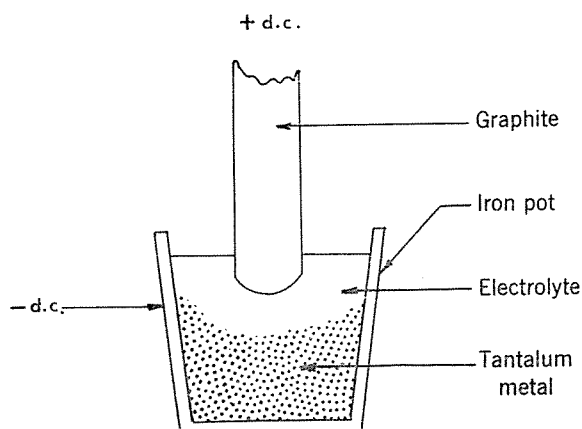


FIGURE 12.—Tantalum Electrolysis Cell (81).
(Courtesy of Chemical Engineering Progress)

Advantages of the electrolysis method are

1. The powder is relatively coarse and harmful impurities are easily washed free.
2. Ductility, particle size, and purity are ideal for powder metallurgy.

The disadvantages are

1. High equipment cost.
2. Less than half of the tantalum is reduced during the operation of the cell. The unreduced salt must be recovered by recrystallization.

A variation of the electrolysis method employs a cell composed of a heated graphite pot and removable metal cathodes. The electrolyte is fused potassium or sodium chloride to which K_2TaF_7 or K_2CbF_7 is added. The operation of the cell is almost continuous because when the metal cathodes have been loaded with deposited metal, they are removed and quickly replaced with others. The metal content of the bath is maintained by adding fluoride.

CARBON REDUCTION

Since 1935 oxides of the two metals have been reduced using a carbon source. In 1960 columbium metal is manufactured from the pentoxide in this manner.

The method consists of reacting a stoichiometric ratio of columbium carbide with the oxide. The mixture is pressed into bars, which are then heated in a vacuum between $1,600^\circ$ and $2,000^\circ$ C. by radiation from graphite or tantalum resistance elements. The result is evolution of carbon monoxide and formation of a porous metal mass containing small amounts of carbon and oxygen which may be removed by subsequent sintering. Powder is produced by grinding in a ballmill. A typical analysis of sintered columbium produced in this fashion is colum-

bium, 99.8 percent; tantalum, 0.05 percent; titanium, 0.01 percent; iron, 0.01 percent; O_2 , 0.05 percent; N_2 , 0.03 percent; and carbon, 0.02 percent.

SODIUM REDUCTION

The method of producing tantalum by sodium reduction of K_2TaF_7 (used by von Bolton) is still in use. A mixture of dried potassium fluoride and sodium pellets is placed in a steel crucible and heated in a muffle furnace at about 700° C. to produce a rapid exothermic reaction. After the metal-salt mass has cooled, it is pulverized and washed with water and acid to remove residual sodium fluoride and potassium fluoride from the metal. The resultant fine powder is notably low in carbon but contains a small amount of oxygen.

KROLL PROCESS

Columbium and tantalum metals have been produced experimentally by decomposing the chlorides with magnesium, sodium, or a magnesium-sodium mixture acting as a reductant. Columbium pentachloride feed material may be prepared by chlorinating columbium carbide at 500° C. The decomposition process involves vaporizing the pentachloride in the presence of the molten metallic reductant in a stainless steel retort heated by a two-zone resistance furnace (fig. 13). The reductant in a crucible in the lower part of the retort is melted, and then heat is supplied to the upper zone to vaporize the chloride and sustain the reaction. When the reaction is complete, the crucible is transferred to a high-vacuum retort where byproduct magnesium chloride and excess magnesium are vaporized by vacuum distillation.

CALCIUM BOMB

Columbium pentoxide has been reduced experimentally with calcium, in a steel bomb or pressure vessel. The bomb is lined with fused magnesium oxide and charged with a mixture of powdered columbium pentoxide, $\frac{1}{4}$ -inch calcium-metal granules, and powdered iodine (fig. 14). A resistance-heated columbium-wire igniter is embedded near the top of the charge, and the vessel is sealed. An exothermic reaction is initiated which is rapidly completed. The reduced metal collects as a fused regulus beneath the slag. The metal contains small quantities of oxygen and only minor quantities of other impurities.

REDUCTION OF Al_3Ta BY COPPER

A new experimental metallic reduction technique uses copper to reduce Al_3Ta . First,

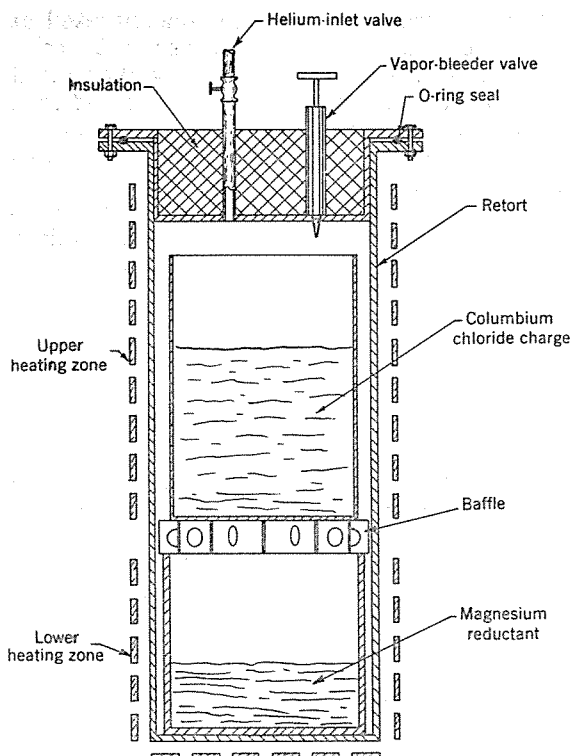


FIGURE 13.—Kroll Reactor.

K_2TaF_7 and aluminum are reacted in an induction-heated graphite crucible at $1,100^\circ C.$ to yield Al_3Ta . Next, the Al_3Ta is recovered by dissolving the aluminum matrix in acid. The Al_3Ta is then heated in the same equipment to $1,100^\circ C.$ with copper to form free tantalum suspended in an aluminum-copper alloy. The tantalum is recovered by dissolving the aluminum-copper alloy with acid. Carbon, aluminum, and copper impurities are removed during subsequent sintering operations. The method has high yield and low equipment cost but requires the recovery of the copper for reuse to approach economic feasibility.

CRYSTAL-BAR PROCESS

The crystal-bar process, also called the Van Arkel-de Boer method, can be used to produce a high quality but relatively expensive metal from either columbium or tantalum pentachloride. The process may be used to produce the metals or as a means of placing protective coatings on filaments of other metals. For example, to produce columbium, columbium pentachloride vapor, entrained in hydrogen or pumped under vacuum, is decomposed by passing it over a columbium wire heated to between $1,200^\circ$ and $2,000^\circ C.$ A

deposit of the pure metal rapidly builds up on the filament, and the released chlorine can be recycled to produce additional chlorides (45, 69, 70).

VAPOR PLATING

Tantalum has been vapor-plated on other metals by heating them to $1,100^\circ C.$ in a stream of hydrogen and tantalum pentachloride. These coatings thus far have been porous and lacking in ductility.

Purification and Production of Ingot POWDER METALLURGY

Until 1957 powder metallurgy was the only process used commercially to produce massive columbium and tantalum. In this process, the dry powders from electrolytic reduction are pressed into bars using a pressure of about 50 tons per square inch. The pressed bars weigh up to 10 kilograms. Sintering is done in a vacuum at temperatures between $2,100^\circ$ and $2,600^\circ C.$ In the United States the temperature is attained through resistance heating by holding the bars between water-cooled terminal clamps and passing a high-amperage electric current through them. In England the metal is heated in a furnace

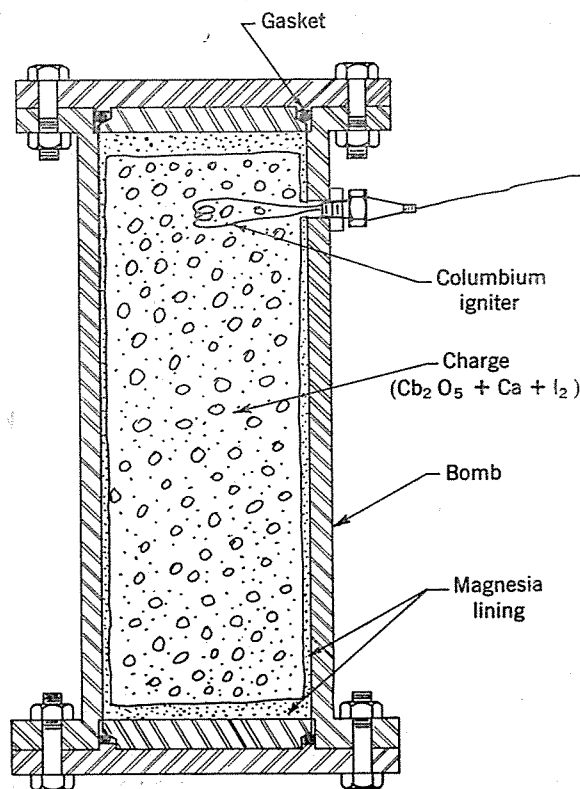


FIGURE 14.—Calcium Bomb Reactor.

using tungsten heating elements arranged cylindrically around the bar. The English procedure takes longer, and to produce highly ductile metal, the bar must be resintered in another furnace using a technique similar to the method used in the United States. Hydrogen, oxygen, and certain other impurities are evolved, and a density about 90 percent of theoretical is attained in the initial sintering. By combining subsequent cold-rolling or forging with one or two intermediate anneals in a high vacuum at about 2,500° C., almost complete consolidation is achieved.

ARC-MELTING

Sponge from Kroll process reduction and powder from electrolytic or carbon-reduction methods have been arc-melted. The powders are pressed and extruded as roundels before melting. Arc-melting largely overcomes the restriction upon ingot size imposed by powder metallurgy. In powder metallurgy the size of the compacting press and the magnitude of electric current needed for resistance sintering were limiting parameters.

The metal is melted in a vacuum or an inert argon or helium atmosphere. A water-cooled copper crucible is used to contain the molten metal. The water-cooled crucible causes rapid solidification of the metal, thus preventing reaction with the container walls from contaminating the melt.

Melting may be carried out by heating with an electric arc established between the sponge in the crucible and a carbon or tungsten electrode to produce a consolidated button in the crucible. To produce large ingots, a consumable electrode of columbium or tantalum is used. A large consumable electrode is made by welding lengths of compacted sponge or powder together as the electrode is fed into the arc furnace (fig. 15). As the tip of the electrode melts, it drips into the water-jacketed copper crucible beneath to form the first-melt ingot. These ingots may or may not be used as electrodes to form new more homogeneous or larger ingots by repeating the process.

ZONE REFINING

Zone refining has been applied to the purification of many materials and has been found to be exceptionally effective in purifying columbium. Under inert gas or in vacuum, a series of molten zones are passed along an ingot in one direction (fig. 16). Impurities travel with, or in the opposite direction to, the zones, depending on whether the particular impurities lower or raise the melting point of the metal. As a result, the

impurities gradually become concentrated at one end of the charge or the other, thereby purifying the rest of the ingot. Conventional zone refining of this sort is carried out in a horizontal boat or tube. The main problem is to find a container that will not contaminate the melt. Many methods of heating the zones have been proposed, including resistance heaters, induction or dielectric heating, gas flames, arcs, and focused radiation such as sunlight. Zone refining without a container removes the danger of contamination of the product, and is discussed in the following section.

CAGE-ZONE OR FLOATING-ZONE REFINING

In cage-zone melting, again under inert gas or in vacuum, the specimen is moved vertically by pulleys through a short induction-heated coil, or the coil is passed over the ingot. The corners of the bar remain solid and constitute the "cage" that confines the molten metal. Surface tension and the electromagnet levitation repulsion force between the inducing and induced current prevent the liquid from pouring out through the parts of the surface that are molten (fig. 17). In the floating-zone method, the molten zone is passed repeatedly at a very low rate of travel along the bar, providing the conditions for zone refining. After treatment, the corners and unmelted top and bottom parts of the bar are machined off. The floating-zone method differs from cage-zone melting in that a cross section of the bar is completely molten, eliminating the possibility of slight contamination from the unmelted cage.

After nine passes through the floating-zone apparatus, the oxygen content of columbium was reduced to 0.005 percent and the nitrogen content to 0.002 percent. The VPN hardness (50-kg. load) of cage-zone-melted bar after nine passes was (1) top, 59.7; (2) center, 59.3; (3) bottom, 65.2.

ELECTRON BOMBARDMENT

Basically, an electron-bombardment melting furnace is a large vacuum tube. Electrons are emitted from a heated tungsten filament (cathode) in high vacuum (10^{-4} to 10^{-6} mm. mercury) and acquire energy from a high electric potential (5,000 to 10,000 volts). The electrons strike the columbium target (anode) which is heated by energy transferred from the electrons. If the flow of electrons is large enough, the columbium melts. The advantage of this method derives from four factors: Crucible material, time, temperature, and pressure. Electron-bombardment melting uses a water-cooled copper mold to

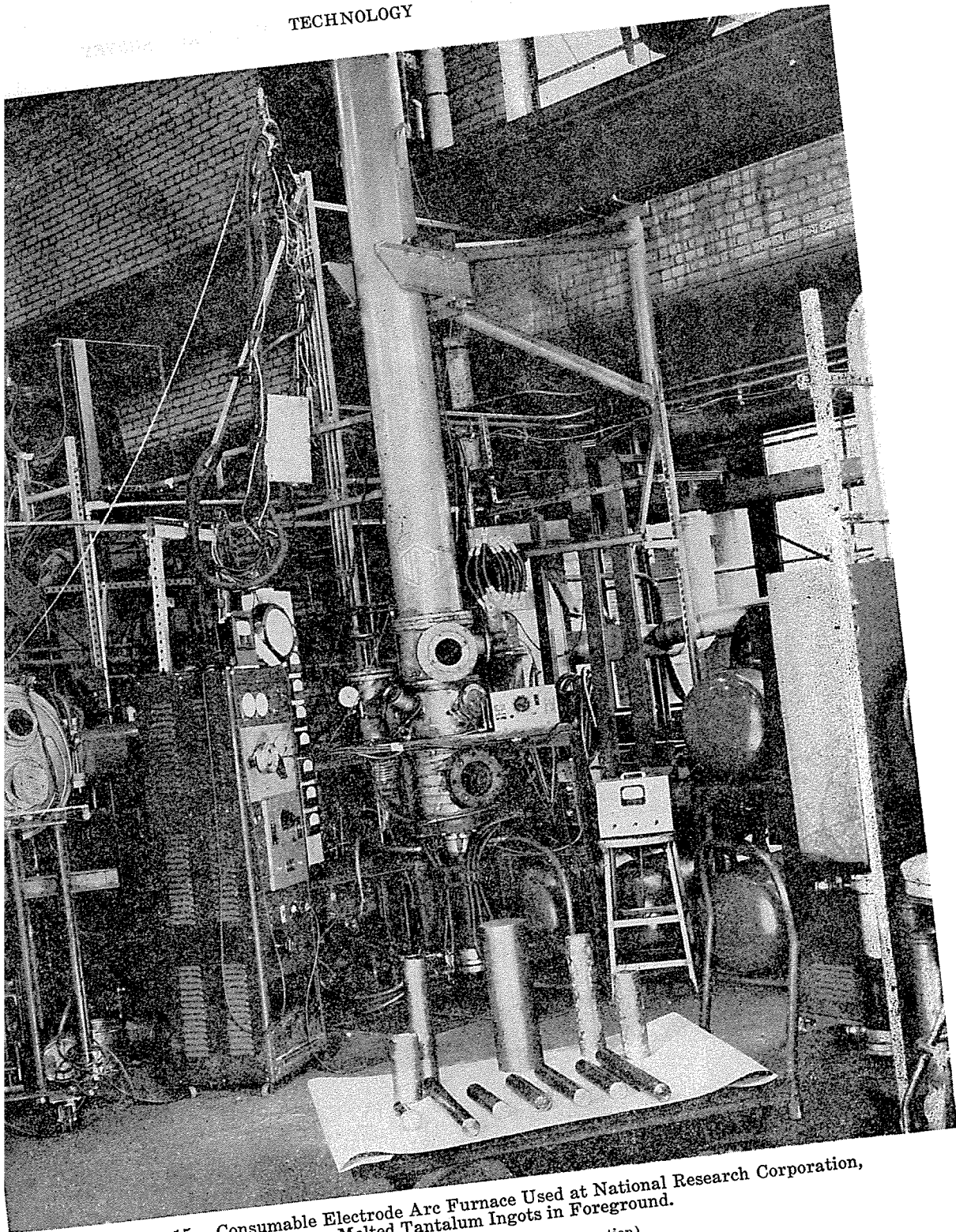


FIGURE 15.—Consumable Electrode Arc Furnace Used at National Research Corporation,
Arc-Melted Tantalum Ingots in Foreground.
(Courtesy of National Research Corporation)

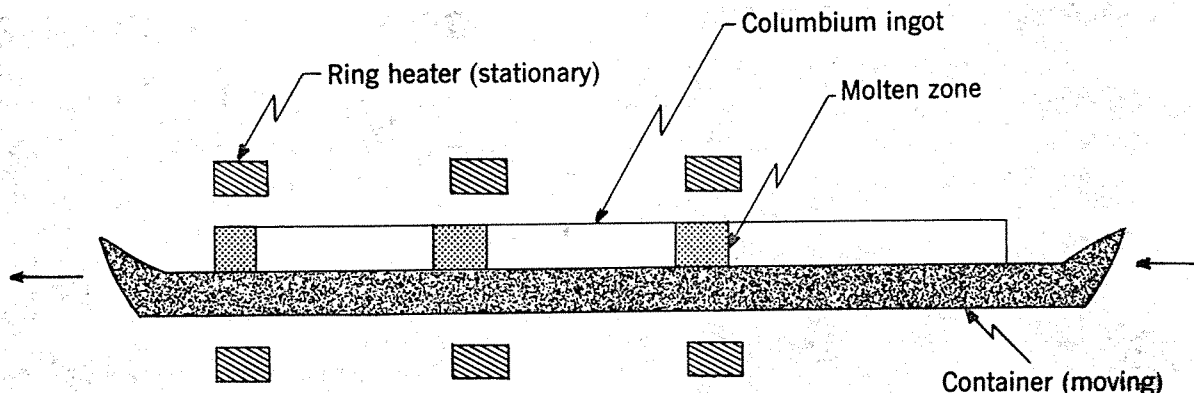


FIGURE 16.—Schematic Drawing of Horizontal Multipass Zone Refining.

receive the molten metal, employs truly high vacuum, and allows independent control of time and temperature (fig. 18). The first two factors result in decreased impurity, and the third factor permits discontinuous operation and close control of the speed of crystallization (and therefore crystal size). No other commonly applied method of melting columbium offers all of these features (36, 77, 78).

Temescal Metallurgical Corp., Richmond, Calif. (now in Berkeley, Calif.) was the first to apply the technique commercially. Temescal manufactures three types of furnace—60

kw. for experimental purposes and 120 and 225 kw. for production of 3-inch-diameter ingots or for custom melting (figs. 19 and 20). The purified metal produced is removed through a vacuum lock at the bottom of the furnace as a continuous cast ingot. The target fed through a vacuum lock at the top of the furnace may be in the form of rod, clips, or pressed powder.

A typical analysis of impurities in electron-beam melted columbium is

Impurity:	Percent	Impurity:	Percent
Oxygen	0.0106	Zirconium	0.03
Nitrogen	.0130	Titanium	.008
Carbon	.0095	Iron	.007
Tantalum	.171		

Ferroalloy Production

Columbium or tantalum is added to steel in the form of ferrocolumbium or ferrotantalum-columbium because pure metal is too expensive to use for alloying purposes. The ferroalloy may be manufactured in an electric furnace or by the aluminothermic technique. Eutectic alloys, which may eventually replace the present alloy as the additive form, can be made using the same method with appropriate alteration of feed materials (24).

ELECTRIC FURNACE

Most ferrocolumbium and ferrotantalum-columbium made in the United States is manufactured by an electric-furnace technique. This is essentially an open-arc electric-furnace operation, in which silicon is used as the reducing agent. Since silicon reduces columbium selectively, the silicon content of the charge can be varied to reduce a greater or less proportion of the tantalum in the ore, depending upon the alloy that is

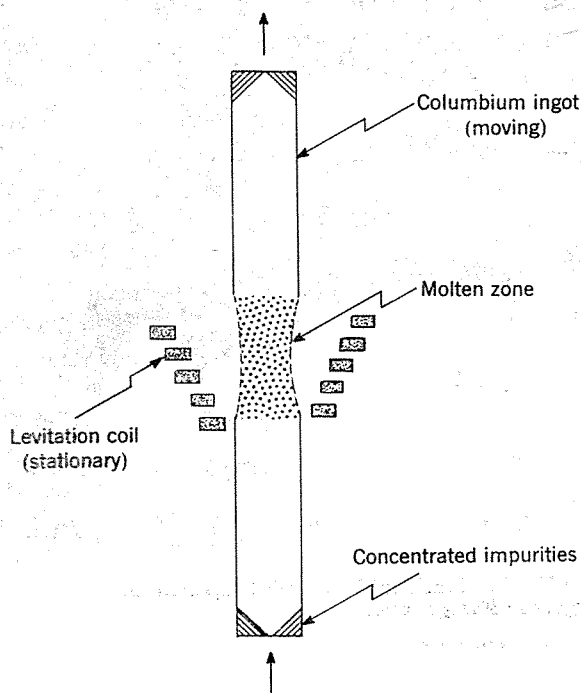


FIGURE 17.—Schematic Drawing of Floating-Zone Refining.

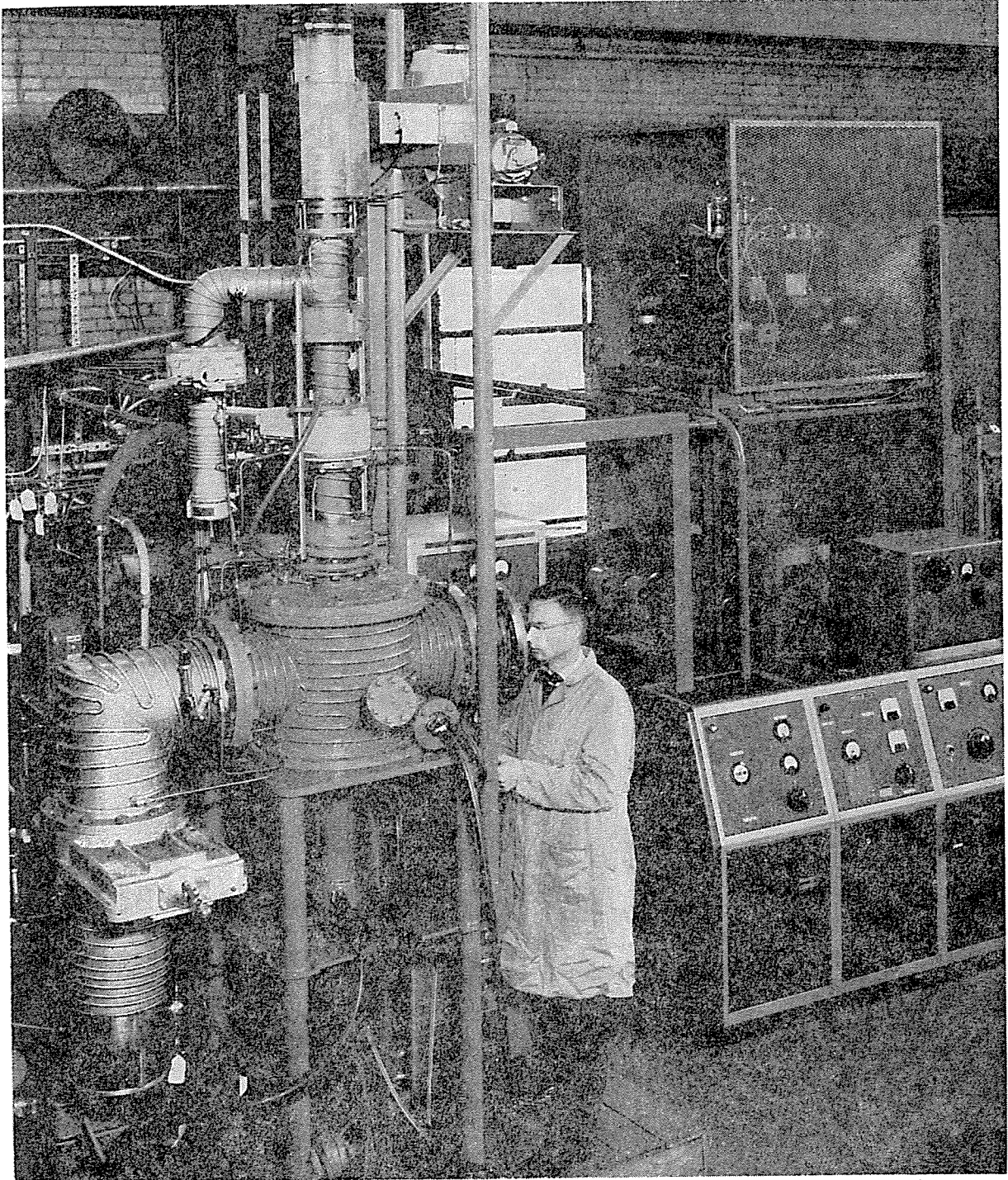


FIGURE 18.—National Research Corporation Electron-Beam Furnace in Operation.

(Courtesy of National Research Corporation)

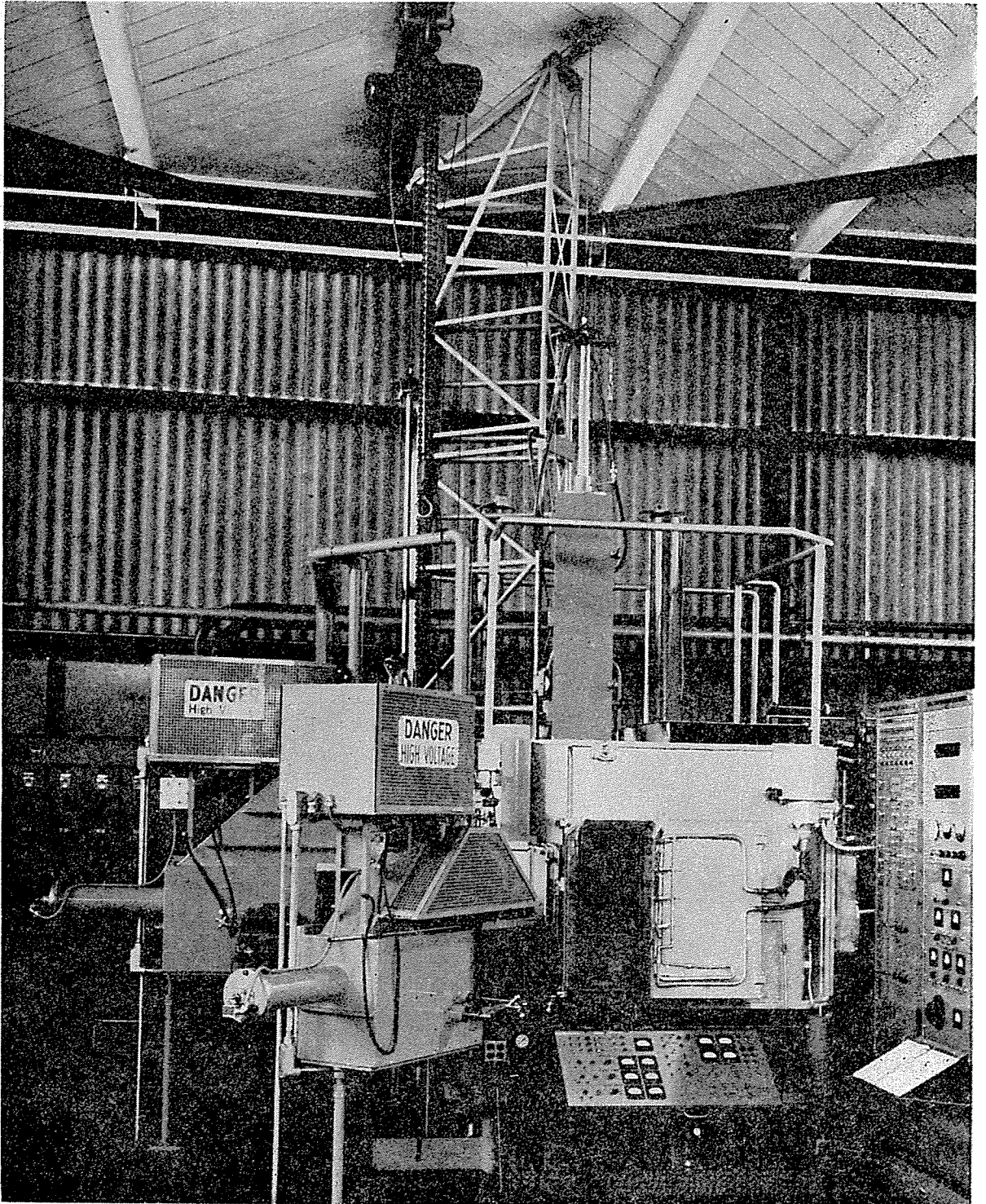


FIGURE 19.—View of Temescal 225-kw. Electron-Beam Melting Furnace.

(Courtesy of Stauffer Metals Co. and Temescal Metallurgical Corp.)

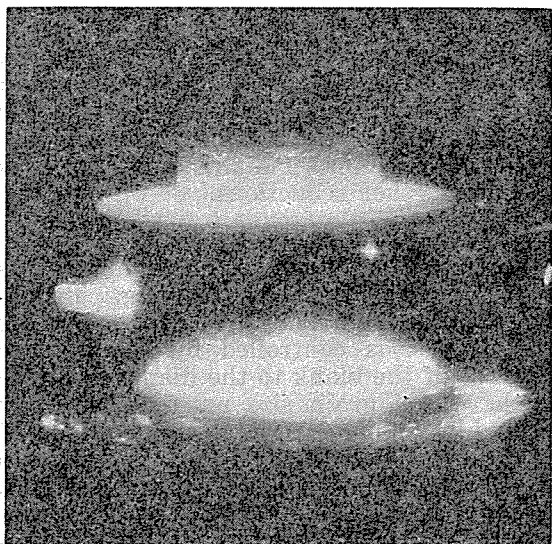


FIGURE 20.—Three-Inch-Diameter Tantalum Ingot Being Electron-Beam Melted.

(Courtesy of Stauffer Metals Co. and Temescal Metallurgical Corp.)

desired. The charge consists of crushed ore, silicon or silicon alloys, and lime or silica as a fluxing agent. To assist in the recovery of tantalum in ferrotantalum-columbium manufacture, a small quantity of aluminum is added to the charge for reducing purposes. The process is applicable to the smelting of ores containing widely varying ratios of columbium to tantalum.

The furnace is a refractory-lined tilting steel type. Graphite electrodes are suspended directly on the charge by means of water-cooled clamps. The arc is established and smelting continues until the reaction is complete. Both slag and alloy are tapped at a temperature of approximately 1,600° C. When the reduction product has cooled, the alloy is mechanically separated from the slag, cleaned, sized, graded, and packed for shipment.

ALUMINOTHERMIC

The aluminothermic method (Goldschmidt's thermite process) of producing low-carbon ferrocolumbium requires that before it is smelted the ore be carefully dressed to eliminate associated minerals that would prove deleterious to the final product. The ores usually are treated in magnetic separators at the mine and then further purified at the smelter to remove tin, titanium, tungsten, and rare-earth minerals.

Additional preparation at the smelter includes possibly presmelting to remove tin, sizing the columbite, selecting suitable alumi-

num powder, an oxidizing roast to impart satisfactory slag and metal conditions, and adding oxidizing agents such as sodium chlorate or barium peroxide to the charge. The reaction is conducted in a covered steel vessel lined with magnesia, alumina, or slag from previous smelts. The exothermic reaction is initiated by electrically igniting a small quantity of barium peroxide and aluminum powder on top of the charge. Two fundamental variations in the actual reduction procedure are followed. In one instance the entire charge is introduced to the vessel, and only one-third of the vessel is occupied when the reaction is completed in one step. The alternate procedure utilizes only a small part of the charge initially; the remainder is carefully fed in as the reaction proceeds. This technique permits a much larger quantity of alloy to be produced at a time.

The reaction mass is allowed to cool in the vessel; the product is then crushed, and the alloy is separated from the slag, cleaned, sized, and graded for shipment.

BUREAU OF MINES

To extract columbium and tantalum values from Congo tin slags, the Bureau of Mines at Albany, Oreg., used several methods, one of which consisted of smelting in an electric-arc furnace with a carbon reductant at more than 1,600° C. Columbium and tantalum (70-percent pure) were recovered as an alloy hearth product; the remainder was iron, carbon, titanium, and manganese. The only use made of the product was to crush it to ¼-inch mesh for feeding a chlorinator unit to produce mixed columbium-tantalum chloride.

Carbide Production

In preparing columbium or tantalum carbides, the metal powder may be carburized directly with a stoichiometric quantity of carbon (lampblack) and heating slowly to 2,000° C. in hydrogen or in a vacuum. A British technique prepares tantalum carbide by firing a mixture of powdered metal and carbon in a closed graphite tube in a hydrogen atmosphere at about 1,600° C. Kennametal prepares carbides directly from ore or from oxides by smelting with carbon and various molten metallic solvents such as aluminum. The resultant crystals of tantalum carbide or columbium carbide are purified by acid treating the regulus to remove aluminum and aluminum carbide. Tantalum or columbium oxides will also react with lampblack at about 1,900° C. in an inert atmosphere to form carbides. A German method sinters a mix-

ture of iron, tantalum, and columbium powders and carbon. The sinter is powdered, leached with hydrochloric acid, heated to 1,600° C. for 2 hours, again powdered, and leached with dilute hydrochloric and hydrofluoric acid to remove final traces of iron.

The carbides produced are sometimes mixed with tungsten carbide and blended with a powdered binding metal such as cobalt. Then power presses, using a pressure of approximately 30 tons per square inch, compact this powder mixture into various shapes. The compacts are then presintered, machined to final design, and reheated to 1,400° C. to produce the finished part with a hardness greater than any naturally occurring material except diamond (actually about 2,000 on the Knoop scale compared with 7,000 for diamond).

Fabrication (41)

Columbium and tantalum fabrication is affected by several characteristics of the metals. Since both metals, when hot, readily absorb gases that cause embrittlement, the forming, stamping, and drawing operations usually are done cold, unless all contaminating gases can be excluded during processing. Both metals seize and tear easily, so that caution must be used while machining them. They are comparable to mild steel in their ability to be drawn, stamped, or formed. The annealed metals take a permanent set in forming and do not spring back from the dies. They work-harden at a much slower rate than most other metals. Reductions in thickness of 60 percent or more without an intermediate anneal are standard practice.

ROLLING

Sheet and foil can be cold-rolled without difficulty on precision Sendzimir rolling mills. Tantalum foil has been rolled as thin as 0.00025 inch, and columbium foil has been made as thin as 0.0005 inches. For use of tantalum foil in capacitors, surface cleanliness, surface quality, and close tolerances are obtained by using the machine to roll only tantalum to avoid contamination of the equipment. During rolling, reductions of as much as 99 percent without intermediate anneal are possible because of the slow work-hardening rate of both metals. General practice, however, is to anneal after total reductions of 70 to 80 percent. Annealing must be conducted in a good vacuum (< 1 micron pressure) to prevent contamination-hardening by oxygen.

SWAGING

During swaging operations, dies having a bearing length 2 or 3 times the diameter and permitting about a 12-percent reduction per pass are preferred. Swaging is used only to about 2.5-millimeter diameter; drawing is used for further reductions.

CUPPING AND DRAWING

Disks are manipulated on a hydraulic press to form suitable cups for drawing. Reduction, varying from 47.5 to 51 percent during this process, is controlled by the relative diameter of the blank to the diameter of the punch. Even grain size must be carefully selected in the material to be cupped to avoid "orange-peel" effects. The cup is drawn to tubing by conventional draw-bench methods, when reductions in area up to 35 percent can be made, or a total reduction of 60 to 85 percent before annealing. The appropriate time for stress relief depends on the wall thickness-diameter ratio of the tube. Both metals tend to gall when drawn, so that effective lubrication and careful choice of tool and die material are required. Tungsten carbide and chromium-plated tools, when carefully used, are successful, but in general the metals will gall when worked with steel or carbide tools. Because copper and copper alloys have little affinity for the metals, aluminum bronzes (copper-aluminum-silicon alloys) are the recommended tool and die materials. For deep drawing, only annealed sheet should be used. The metals do not work harden as rapidly as most, and work hardening first appears at the top rather than in the deepest part of the draw. Cones and domes are usually more difficult to draw than flat-bottomed cup shapes.

MACHINING

In lathe operations, sharp, high-cutting-speed steel tools have proved most satisfactory. Tools ground with the same rakes and angles as are used with soft copper will generally give satisfactory results. A minimum speed of 100 surface feet per minute is needed for most turning operations to prevent tearing of the metal. The work is kept well flooded with carbon tetrachloride at all times. Even when filing or using emery cloth, the file or cloth is kept well wetted with carbon tetrachloride. The toxic properties of carbon tetrachloride make care necessary during these operations.

The same general rules are applied when milling, drilling, threading, or tapping the metals. Milling cutters should be of the staggered tooth type, with substantial back

and side relief. In drilling, the tool point should not rub the work. In threading large diameter tubes it is preferable to cut the threads on a lathe rather than with a threading die. When dies or taps are used, they must be kept free of chips and cleaned frequently. Polished aluminum bronze is a recommended die material.

Very light finishing cuts should be avoided. It is better to use sharp tools and light feeds and finish the work in one cut rather than to take the usual roughing and finishing cuts.

WIRE FORMING

Both tantalum and columbium wires are very malleable and ductile. They can be bent to 180° or more with virtually no radius and can be coiled around a mandrel of their own diameter. Conventional production methods may be used, except that tools and guides should be made of aluminum bronze to avoid scoring or galling the wire. The wire is anodized to enable the surface to hold a lubricant (beeswax) before it is drawn; it is then pickled in acid to remove the oxidized coating after working is completed.

COUPLING

Tantalum or columbium tubing is usually joined by welding, flare couplings or by flanges and gaskets.

SPINNING

Conventional techniques are applied, using aluminum bronze as a tool material, although yellow brass may be used for short runs. One approved lubricant is yellow soap.

EXTRUSION

Some columbium-base alloy ingots are converted to bar stock and sheet by extrusion followed by forging and swaging or rolling. Forming of cups by backward extrusion of columbium (B.h.n. 100) at room temperature has been reported. Tubing has been produced by forward extrusion at 425° C. (800° F.) without any intermediate anneal. The total reduction from the solid slug was 86 percent.

FORGING

A cold-roll forging process developed by General Electric Corp. is used in manufacturing jet-engine turbine and compressor disks. Hammer- and drop-forging are used to form bars from ingots of some high-temperature alloys. Ingots weighing from 2 to 10 tons have been forged in the range 1,175° to 1,260° C. Recently tantalum-tungsten rocket nozzles have been rough-formed from ingots in this way (figs. 21 and 22).

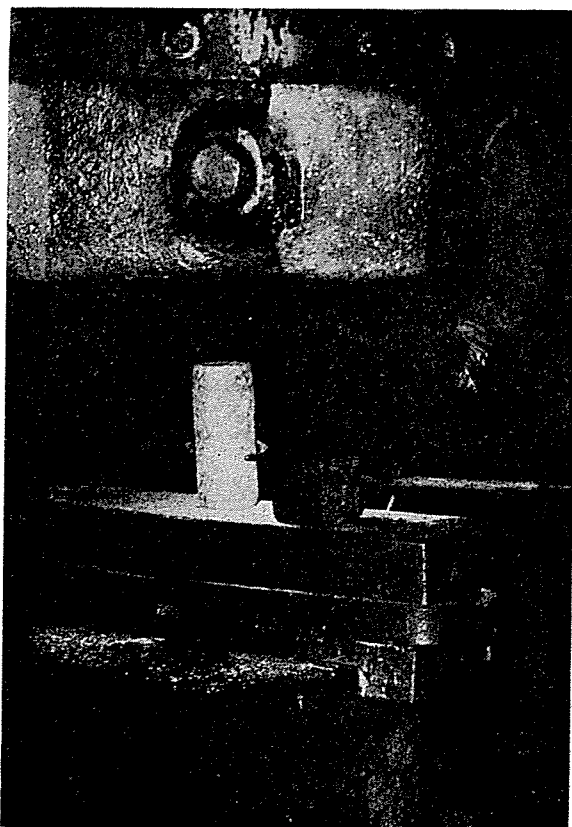


FIGURE 21.—Initial Forging of 90 Percent Tantalum
—10 Percent Tungsten Ingot in Air at 2,000° F.

(Courtesy of Stauffer Metals Co. and Temescal Metallurgical Corp.)

CASTING

Pure columbium or tantalum or columbium-tantalum-based alloys are vacuum-cast to avoid interstitial incorporation of impurities from the atmosphere. High-temperature complex columbium-bearing steels can be cast in air, but to avoid poor quality, careful consideration must be given to proper melting, the effect of tapping temperature, ingot design, and teeming rate.

SLIP CASTING

Experimental work has been conducted on slip-casting columbium and tantalum powders. Results have not been described in the literature, but a good measure of success has been attained by private industry in developing methods to produce sound castings.

CLADDING

Tantalum has been clad with platinum by drawing a platinum tube over a tantalum rod. Subsequent vacuum-firing produces a good metallurgical bond. When the platinum tubing and tantalum rod are well bonded, the

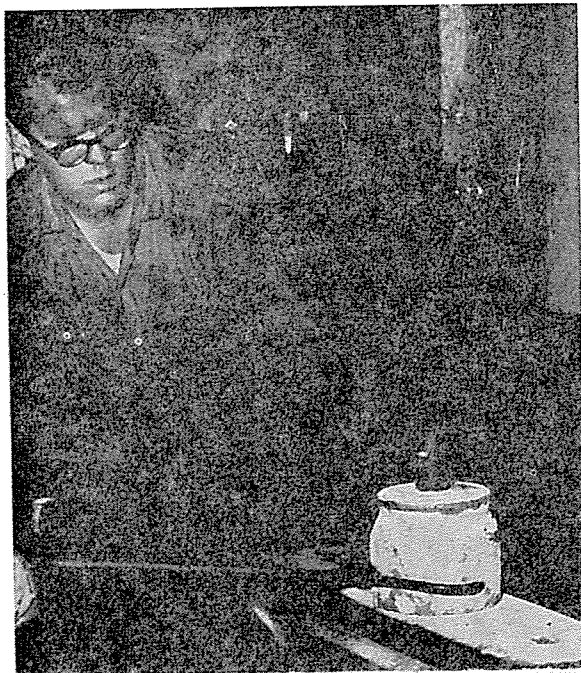


FIGURE 22.—Piercing of Partly Shaped Piece of 90 Percent Tantalum—10 Percent Tungsten Rocket Nozzle Alloy.

(Courtesy of Stauffer Metals Co. and Temescal Metallurgical Corp.)

assembly can be readily drawn to smaller diameter rod and wire.

Copper has been bonded to tantalum by inserting a copper slug in a tantalum cup and hot-extruding the assembly. Copper could also be vacuum cast into a fabricated tantalum well to accomplish the same end.

Tantalum or columbium can be bonded to nickel-plated steel using a technique similar to that described for bonding platinum and tantalum.

Battelle Memorial Institute developed a means of cladding by vapor-plating columbium on type 304 stainless steel tubing by hydrogen reduction of columbium pentachloride at reduced pressure. The coating unit was constructed of Vycor (a heat- and temperature-resistant glass with a low coefficient of expansion) and fused-silica tubing and could treat seven 21-inch-long tubes at one time. The fused-silica deposition tube was heated by a 24-inch-long chromel-wound furnace element. The Vycor vaporizer was attached to the top of the silica deposition tube. Ampoules of columbium pentachloride were placed in the vaporizer; the entire unit was flushed with argon; then the ampoules were pierced. The vaporizer was heated by a 24-inch-long sleeve heater and hydrogen introduced by a valve. When gas flow and

temperature in the plating zone are constant, uniformity of deposition depends on the vaporization rate of the halide. Since the surface area of the halide exposed to the carrier-gas stream slowly decreases owing to depletion and compaction of the halide, compensation by slowly increasing the vaporizer temperature during the run was necessary. Nickel flashing on the steel slightly improved the adhesion of the columbium coating.

WELDING

Both columbium and tantalum can be welded satisfactorily if the weld is protected from the common gases, particularly nitrogen, until it has cooled to 200° C. Inert gas arc-welding, spot and seam resistance-welding, and carbon arc-welding have been applied. Inert gas arc-melting, possible by flooding both sides of the weld with argon or helium, will produce a bright silvery weld with 85 percent of the tensile strength of the parent metal. Resistance spot and seam welding is conducted under water for protection from oxidation, and the welds have substantially the same strength as the metal itself. Carbon arc-welding is possible if the metal is submerged in carbon tetrachloride.

The most satisfactory resistance welds for tantalum are produced with a current density of approximately 85,000 amperes per square inch of electrode surface, measured at the point of contact with the tantalum. For welding tantalum 0.004 to 0.006 inch thick, a welder of at least 2 kilowatts rating should be used, having an open-circuit voltage of 1.5 to 2.0 and a closed-circuit instantaneous voltage of approximately 0.75. The circuit should be adequately wired to permit a current flow of approximately 2,000 amperes. Pure copper or alloy electrodes may be used; some alloys give longer service. When welding electronic-tube parts, where removal of copper residue may prove difficult, tungsten electrodes should be used.

Columbium has been welded by Westinghouse Electric Corp. using a tungsten arc. Welds in 0.065-inch columbium required 160 amperes when welded in commercial argon. The welding voltage was 12 to 13 volts between the contacts at the high frequency arc stabilizing unit.

Electron-beam welding of columbium and tantalum shows considerable promise.

BRAZING

Tantalum can be clad to steel by brazing it to a billet using copper alloy as a brazing medium. The assembled billet is then hot-rolled to the desired sheet thickness.

CLEANING

Fabricated parts may be cleaned without special techniques, although hot caustics must be avoided. Usually, either hot concentrated hydrochloric acid or a saturated solution of chromium trioxide in hot concentrated sulfuric acid is used. The cleaned parts must be thoroughly rinsed of cleaning solution, preferably with hot distilled water.

Electronic-tube parts require somewhat more careful treatment. These parts often are blasted with sharp particles of No. 90 steel grit at a pressure of 20 to 40 pounds to provide a greater radiation surface. Parts that have been so blasted must be cleaned

twice by immersion in hot hydrochloric acid to remove particles of iron, followed each time by rinsing with distilled water.

SCRAP RECOVERY

Columbium or tantalum can be recovered by embrittling by hydrogen absorption followed by crushing and reprocessing by sintering or melting in vacuum. Columbium- or tantalum-bearing alloys must be chemically treated to separate the alloy components if the columbium or tantalum are to be recovered as separate elements. This process varies depending upon the composition of the particular alloy.

CHAPTER 5.—SUPPLY AND DISTRIBUTION

WORLD PRODUCTION

Concentrates

World-production statistics for columbium-tantalum mineral concentrates for 1943-59 are shown in table 33. Before 1943, information is incomplete and not reliable.

Commercial production of tantalite began in Australia and the United States around 1903. Production was not reported in other countries until the early 1930's, when Rhodesia and India, followed by South-West Africa, Nigeria, and the Union of South Africa, reported columbite-tantalite production as demand for ferrocolumbium increased. Other countries reporting ore production before 1943 were Belgian Congo, Bolivia, Brazil, French Equatorial Africa, and Uganda. By 1943, because World War II had constricted European markets, most ore was being sent to the United States for processing, and Bureau of Mines world production records became more complete. During 1943-59 major concentrate producers were Nigeria, 69.7 percent; Belgian Congo (including Ruanda-Urundi), 10.6 percent; Norway, 4.0 percent; Brazil, 3.9 percent; Malaya, 3.0 percent; West Germany, 2.2 percent; Mozambique and the United States, 1.5 percent; and Portugal, 1.0 percent. Seventeen other countries produced the remainder. Total production amounted to 88,515,764 pounds, ranging from a low of 2,360,187 pounds in 1950, through a high of 11,492,027 pounds in 1955 as a result of U.S. Government stockpile purchases at a bonus price, to 4,991,053 pounds in 1958 when stockpiling of foreign ores had ceased. By 1959 private demand and new U.S. Government barter agreements had once again stimulated production which increased to 6,172,917 pounds. The columbium and tantalum content of ore produced in each country is not compiled but probably approximates the grade of U.S. imports as given later in this chapter.

Historically the major source of columbium and tantalum has been the columbite-tantalite series of minerals. Since World War

II, however, interest in pyrochlore as a source of columbium has increased. The history of pyrochlore mining began in the early part of the war, when the German government mined enough ore at Kaiserstuhl to produce about 3.0 metric tons of columbium concentrate. The same property was again worked in 1954 and 1955. A similar deposit at Sove, Norway, has been mined continuously since 1953, and pilot scale production began in Tanganyika in 1957. Facilities for an initial production rate of 4 million pounds of concentrate a year have been installed at Araxá, Brazil; production was scheduled to start during March 1961. Other deposits in Africa, South America, and Canada give bright promise as sources of columbium if future demand is great enough, and if mining and metallurgical costs can be successfully reduced.

Columbium-Tantalum-Bearing Tin Slags and Concentrates

The former Belgian Congo (including Ruanda-Urundi), South-West Africa, and Uganda also have reported production of tin-columbium-tantalum concentrates, averaging about 10 percent combined columbium and tantalum (table 34). These concentrates may later be treated in Europe to separate the columbium and tantalum, and if smelted, the columbium and tantalum content report in the slag and can subsequently be removed. Such slags are produced at smelters in the Republic of the Congo (formerly Belgian Congo), Nigeria, Portugal, and Malaya. The lower grade Malayan slags contain about 4 to 8 percent combined columbium and tantalum pentoxides and are not used in 1960 as a commercial source of columbium and tantalum. Columbium-tantalum-bearing tin slag production statistics are not obtained. However, production of such slag in the Belgian Congo is reported to have ceased in 1958.

Metals, Compounds, and Alloys

World statistics for production of metal, compounds, and alloys are not compiled, but

TABLE 33.—World production of columbium- and tantalum-mineral concentrates by continents and countries, 1943-59, pounds ¹

Continent and country	1943		1944		1945		1946		1947	
	Columbium	Tantalum	Columbium	Tantalum	Columbium	Tantalum	Columbium	Tantalum	Columbium	Tantalum
North America:										
Canada										
United States	5,771	² 9,411	3,208	² 7,204	1,149	² 5,500		² 3,475		3,259
Total	15,182		10,412		6,649		3,475		3,259	
South America:										
Argentina	1,800			200	1,000					
Bolivia					1,034		6,834			
Brazil	³ 37,500	³ 399,033	³ 116,871	³ 443,125		³ 66,138	³ 15,435	³ 98,035		³ 71,650
British Guiana										
French Guiana										
Total	438,333		560,196		68,172		120,304		71,650	
Europe:										
Germany, West										
Norway										
Portugal										
Spain										
Sweden										
Total										
Asia:										
Malaya	⁴ 3,000		⁴ 3,000		⁴ 3,000					
India	7,981		1,019							
Total	10,981		4,019		3,000					
Africa:										
Belgian Congo (including Ruanda-Urundi)	332,955		648,270		436,590		370,440		348,390	
French Equatorial Africa					10,584		2,200			
Madagascar					22					
Mozambique ⁵	11,023		9,965		3,314		440			
Nigeria	1,796,480	4,500	4,603,536	27,082	3,519,040	29,792	3,472,000	2,890	2,880,640	8,310
Rhodesia and Nyasaland, Federation of		13,820		12,640		14,740		16,900		27,300
Sierra Leone										
South-West Africa ⁶		2								493
Swaziland										
Uganda ⁷			⁸ 20,552	⁸ 9,518	⁸ 13,194	⁸ 3,036	⁸ 4,883		⁸ 2,800	
Union of South Africa		2,440		6,312		776		4,000		
Total	2,161,220		5,337,875		4,031,688		3,873,753		3,267,933	
Oceania: Australia		27,418		24,192		1,053		806		1,411
Total	27,418		24,192		1,053		806		1,411	
Grand total	2,653,134		5,936,694		4,110,562		3,998,338		3,344,253	

See footnotes at end of table.

TABLE 33.—World production of columbium- and tantalum-mineral concentrates by continents and countries, 1943-59, pounds ¹—Continued

Continent and country	1948		1949		1950		1951		1952	
	Columbium	Tantalum	Columbium	Tantalum	Columbium	Tantalum	Columbium	Tantalum	Columbium	Tantalum
North America:										
Canada.....						1,000		925		5,385
United States.....	100	500	1,020							
Total.....	600		1,020			1,000		925		5,385
South America:										
Argentina.....			³ 1,080							
Bolivia.....							1,043			
Brazil.....	³ 10,494	³ 9,202	³ 33,942	³ 91,237	³ 26,709	³ 18,700	³ 11,000	³ 8,818	5,017	49,813
British Guiana.....										
French Guiana.....										
Total.....	19,696		126,259		45,409		20,861		54,830	
Europe:										
Germany, West.....										
Norway.....						3,009		4,526		
Portugal.....										
Spain.....										
Sweden.....										
Total.....					3,009		4,526			
Asia:										
Malaya.....					17,920		56,000		105,280	
India.....										
Total.....					17,920		56,000		105,280	
Africa:										
Belgian Congo (including Ruanda-Urundi).....	319,725		255,780		297,675		209,437		231,042	
French Equatorial Africa.....	3,461		12,984		3,655				3,527	
Madagascar.....				550	7,700		8,598		5,732	
Mozambique.....							11,133		40,518	
Nigeria.....	2,455,040	8,243	1,989,120	4,980	1,935,360	2,240	2,419,200	6,720	2,896,320	2,240
Rhodesia and Nyasaland, Federation of.....		16,120		10,840		1,700				10,360
Sierra Leone.....										
South-West Africa.....		17		5,364		12,570		3,974		4,400
Swaziland.....										
Uganda.....	³ 2,285		³ 5,571		³ 11,413		³ 42,560		³ 5,914	
Union of South Africa.....						4,000		4,000		4,000
Total.....	2,804,891		2,285,189		2,276,313		2,705,622		3,204,053	
Oceania: Australia.....		12,023		3,502		16,536		5,125		15,720
Total.....	12,023		3,502		16,536		5,125		15,720	
Grand total.....	2,837,210		2,415,970		2,360,187		2,793,059		3,385,268	

See footnotes at end of table.

TABLE 33.—World production of columbium- and tantalum-mineral concentrates by continents and countries, 1943-59, pounds ¹—Continued

Continent and country	1953		1954		1955		1956		1957	
	Columbium	Tantalum	Columbium	Tantalum	Columbium	Tantalum	Columbium	Tantalum	Columbium	Tantalum
North America:										
Canada.....			90	77	42	390				
United States.....	14,867		32,829		12,954		² 216,606		² 370,483	
Total.....	14,867		32,996		13,386		216,606		370,483	
South America:										
Argentina.....			¹⁰ 11,023		¹⁰ 10,800	¹⁰ 6,614	3,968		2,205	
Bolivia.....	3,366				2,350					
Brazil.....	64,960	³ 40,320	32,320	³ 107,520	170,240	³ 127,680	394,240	³ 208,320	³ 54,500	¹⁰ 199,205
British Guiana.....	11,200		4,480		6,720					
French Guiana.....	13,228		28,250		19,805			¹⁰ 14,532		¹⁰ 3,075
Total.....	133,074		183,593		344,209		621,060		258,985	
Europe:										
Germany, West.....			267,957	62,865	849,310	594,030			1,653	
Norway.....	40,367		392,419		675,930		573,196		489,421	
Portugal.....	68,121	154,323	148,732	86,279	168,362	6,614	31,024	7,054	72,953	
Spain.....	4,410				2,525	11,276				
Sweden.....	16,713	4,242		19,251						
Total.....	288,176		977,503		2,308,047		611,274		564,027	
Asia:										
Malaya.....	116,480		248,640		529,104		619,136		317,462	
India.....										
Total.....	116,480		248,640		529,104		619,136		317,462	
Africa:										
Belgian Congo (including Ruanda-Urundi).....	623,902		967,819		967,819		921,523		¹⁰ 905,989	¹⁰ 491,124
French Equatorial Africa.....	3,514		6,261		2,672					
Madagascar.....	8,377		36,596		38,801		19,400		¹⁰ 3,075	¹⁰ 6,835
Mozambique ⁵	58,133		94,031		82,884		51,971		288,582	
Nigeria.....	4,388,160		6,527,360	22,400	7,047,040	35,840	5,832,960	33,600	4,307,520	40,320
Rhodesia and Nyasaland, Federation of.....	5,100	27,060	18,060	15,552	12,240	4,660	5,080	29,320	760	76,960
Sierra Leone.....			8,960		8,960					
South-West Africa ⁶	17,634		22,439	3,868	8,299	2,924	9,607	3,740	9,325	14,676
Swaziland.....										32,920
Uganda ⁷	23,542		23,117		34,003		10,080		4,032	
Union of South Africa.....		38,000		46,000		24,000		2,900		⁴ 2,000
Total.....	5,193,422		7,792,463		8,270,142		6,920,181		6,184,118	
Oceania: Australia.....	18,124		117,767		27,139		159,655		65,000	
Total.....	18,124		117,767		27,139		159,655		65,000	
Grand total.....	5,764,143		9,352,962		11,492,027		9,147,912		7,760,075	

See footnotes at end of table.

TABLE 33.—World production of columbium- and tantalum-mineral concentrates by continents and countries, 1943-59, pounds¹—Continued

Continent and country	1958		1959		Total	Percent of total
	Columbium	Tantalum	Columbium	Tantalum		
North America:						
Canada.....			¹⁰ 14,000		14,599	<0.1
United States.....	¹² 428,347		¹² 189,263		1,313,256	1.5
Total.....	428,347		203,263		1,327,855	1.5
South America:						
Argentina.....	¹⁰ 2,262	¹⁰ 11,635	¹⁰ 3,591	¹⁰ 1,611	57,739	<.1
Bolivia.....					14,627	<.1
Brazil.....	³ 302,030		³ 30,864	³ 207,232	3,452,150	3.9
British Guiana.....					22,400	<.1
French Guiana.....					78,890	.1
Total.....	315,927		243,298		3,625,856	4.1
Europe:						
Germany, West.....	¹⁰ 46,628	¹⁰ 135,431	¹⁰ 11,578		1,969,452	2.2
Norway.....	630,516		756,178		3,558,027	4.0
Portugal.....	¹⁰ 65,461	¹⁰ 32,513	¹⁰ 38,083	¹⁰ 27,227	914,281	1.0
Spain.....					18,211	<.1
Sweden.....		¹⁰ 992			41,198	<.1
Total.....	911,541		833,066		6,501,169	7.3
Asia:						
Malaya.....	356,160		268,800		2,643,982	3.0
India.....					9,000	<.1
Total.....	356,160		268,800		2,652,982	3.0
Africa:						
Belgian Congo (including Ruanda-Urundi) ¹¹	553,355		535,718		9,417,553	10.6
French Equatorial Africa.....					48,858	<.1
Madagascar.....	28,880		22,046		178,362	.2
Mozambique ⁶	378,916		320,004		1,359,164	1.5
Nigeria.....	1,803,200	49,930	3,559,875	31,114	61,743,052	69.7
Rhodesia and Nyasaland, Federation of.....		96,260		116,820	532,292	.6
Sierra Leone.....					17,920	<.1
South West Africa.....	4,152	6,574	2,610	1,539	134,207	.3
Swaziland.....					32,920	<.1
Uganda ⁷	6,384		5,264		223,748	.3
Union of South Africa.....		37,920		11,500	187,848	.2
Total.....	2,965,571		4,606,490		73,880,924	83.5
Oceania:						
Australia.....	13,507		4 18,000		526,978	.6
Total.....	13,507		4 18,000		526,978	.6
Grand total.....	4,991,053		6,172,917		88,515,764	100.0

¹ Frequently the composition (Cb₂O₅-Ta₂O₅) of these concentrates lies in an intermediate position; neither Cb₂O₅ nor Ta₂O₅ is strongly predominant; or the producing country has not differentiated between columbite and tantalite. Therefore, the production figure is centered.

² Principally microlite. ³ Exports. ⁴ Estimate.

⁵ In addition, 176 pounds of samarskite was produced in 1951 and 132 pounds in 1953.

⁶ In addition, tin-tantalite-columbite concentrates were produced as follows: 1943, 560 pounds; 1944, 2,000 pounds.

⁷ In addition, tin-columbium-tantalum concentrates were produced as follows: 1943, 15,700 pounds; 1947, 329 pounds; 1948, 210 pounds; 1951, 336 pounds; 1952, 3,248 pounds; 1953, 4,480 pounds; 1954, 6,720 pounds; 1955, 515 pounds.

⁸ Contained in 28,334 pounds mixed concentrates in 1944; in 17,687 pounds in 1945; in

7,706 pounds in 1946; in 3,651 pounds in 1947; and in 3,203 pounds in 1948.

⁹ Includes 6,720 and 670 pounds of bismutotantalite in 1944 and 1945.

¹⁰ U.S. imports.

¹¹ In addition, tin-columbium-tantalum concentrates were produced as follows: 1947, 597,555 pounds; 1948, 1,148,050 pounds; 1949, 1,944,457 pounds; 1950, 2,431,674 pounds; 1951, 2,597,019 pounds; 1952, 2,813,070 pounds; 1953, 3,575,861 pounds; 1954, 5,970,057 pounds; 1955, 5,456,335 pounds; 1956, 6,501,365 pounds; 1957, 4,360,699 pounds; 1958, 3,196,670 pounds; 1959 (est.), 2,750,000 pounds; columbium-tantalum content averaging about 10 percent.

¹² Columbium-tantalum concentrate plus columbium-tantalum oxide content of euxenite concentrate.

TABLE 34.—*Production of tin-columbium-tantalum concentrate, 1943-59, pounds*

Year	Belgian Congo (including Ruanda Urundi)	South-West Africa	Uganda
1943.....		560	15,700
1944.....		2,000	
1945.....			
1946.....			
1947.....	597,555		329
1948.....	1,148,050		210
1949.....	1,944,457		
1950.....	2,431,674		
1951.....	2,597,019		336
1952.....	2,813,070		3,248
1953.....	3,575,861		4,480
1954.....	5,970,057		6,720
1955.....	5,456,385		515
1956.....	6,501,365		
1957.....	4,360,699		
1958.....	3,196,670		
1959.....	¹ 2,750,000		
Total...	43,342,862	2,560	31,538

¹ Estimated.

foreign production is only a fraction of the production in the United States. Foreign producers in 1956, 1957, 1958, or 1959 by country, company, and product were

Country and firm:	Products
Austria:	
Metallwerke Plansee..	Columbium and tantalum carbides; tantalum wire, rod, and sheet.
Treibacher Chemische Werke.	Columbium, ferrocolumbium.
Belgium:	
Société Générale Métallurgique de Hoboken.	Ferrocolumbium; ferrotantalum-columbium; columbium and tantalum carbides and oxides.
Société Anonyme Sadaci.	Tantalum carbide.
Canada:	
Quebec Metallurgical Industries, Ltd.	Columbium powder, sponge, and oxide; ferrocolumbium, tantalum oxide.
Atlas Steel, Ltd.....	Ferrocolumbium and ferrotantalum-columbium.
Fahr Alloy Canada, Ltd.	Do.
England:	
Murex, Ltd.....	Ferrocolumbium; ferrotantalum-columbium; columbium and tantalum powder, rod, sheet, and wire.
Blackwell's Metallurgical Works, Ltd.	Ferrocolumbium.

Country and firm—Continued:	Products
England—Continued	
Minworth Metals, Ltd.	Ferrocolumbium.
London & Scandinavian Metallurgical Co., Ltd.	Do.
Borax Consolidated, Ltd.	Columbium boride.
Imperial Chemical Industries, Ltd.	Columbium fabricated shapes.
Accles & Pollock, Ltd.	Do.
France:	
Fabriques de Produits Chimiques de Thann et Mulhouse S.A.	Columbium; columbium oxide.
Compagnie Pechiney..	Tantalum fabricated shapes.
Société d'Électro-Métallurgia d'Ugine.	Ferrocolumbium.
Société Kuhlmann.....	Columbium pentoxide.
Germany:	
Herman C. Starck A.G.	Columbium and tantalum powders, oxides, and carbides.
Heraeus Quarzschmelze G.m.b.H.	Columbium and tantalum rod, sheet, and wire.
Gesellschaft für Elektrometallurgie.	Ferrocolumbium, tantalum.
Italy:	
S.P.A. Leghe e Metalli.	Ferrocolumbium.
Japan:	
Showa Denko.....	Do.
Sweden:	
A. B. Ferrolegeringär.	Do.
Switzerland:	
Société des Produits Pharmaceutiques Ciba.	Tantalum oxide.
Norway:	
Electric Furnace Products Company, Ltd.	Ferrocolumbium.
A. S. Norsk—Bergverk.	Columbium oxide, ferrocolumbium.
Finland:	
Kovametalli Oy.....	Columbium and tantalum carbides.

No information is available on commercial production in Soviet bloc countries, but both columbium and tantalum are undoubtedly produced.

DOMESTIC PRODUCTION Concentrates

The earliest domestic production of tantalite was reported in 1884, when 3,000 pounds was mined in the Black Hills of South Dakota for sale to museums as mineralogical speci-

mens. Commercial production began in 1903 or 1904 in the same State, and a small but persistent domestic production was maintained through 1955. In 1956 production increased tremendously when the Bear Valley, Idaho, euxenite-monazite-columbite placer deposit of Porter Brothers Corp. began full-scale production. In 1958 domestic production of concentrate reached a record high of 428,347 pounds (total weight of columbite-tantalite plus combined oxide content of euxenite concentrate). In 1959 production dropped to 189,263 pounds owing to the closing of Porter Brothers mine during the year, when mining the ore necessary to fulfill its

TABLE 35.—*Domestic mine shipments of columbium-tantalum mineral concentrates reported, 1884–1959, by year, in pounds*

Year ¹	Columbium concentrates		Tantalum concentrate	
	Pounds	Value	Pounds	Value
1884.....			3,000	¹ \$1,000
1904.....			1 500	1 250
1905.....			1 500	1 250
1906.....			1 800	1 400
1907.....			1 500	1 250
1908.....				
1909.....			1 500	1 250
1910.....			1 500	1 250
1911.....			1 500	1 250
1912.....				
1913.....				
1914.....				
1915.....				
1916.....				
1917.....				
1918.....			4,500	2,250
1919.....			300	90
1920.....			4,000	1,450
1921.....			3,400	1,150
1922.....			600	240
1923.....			1,350	540
1924.....			1,197	598
1926.....			2,100	650
1927.....			1,100	378
1928.....			34,899	26,322
1929.....			22,117	17,261
1930.....			5,100	3,350
1931.....			700	490
1932.....			390	234
1933.....			300	180
1934.....			2,425	1,968
1935.....			7,681	4,521
1937.....	¹ 2,000	¹ \$1,000	14,307	¹ 12,317
1938.....	2,000	1,000	34,189	34,127
1939.....	280	140	60	60
1941.....			250	219
1942.....			200	175
1943.....	5,771	1,465	9,411	27,621
1944.....	3,208	917	7,204	23,317
1945.....	1,149	287	5,500	13,366
1946.....			3,475	8,793
1947.....			3,259	8,677
1948.....	100	42	500	600

TABLE 35.—*Domestic mine shipments of columbium-tantalum mineral concentrates reported, 1884–1959, by year, in pounds—Continued*

	Columbium-tantalum concentrate (undifferentiated)	
	Pounds	Value
1949.....	1,020	\$1,785
1950.....	1,000	2,150
1951.....	925	1,528
1952.....	5,385	16,723
1953.....	14,867	29,779
1954.....	32,829	57,262
1955.....	12,954	22,125
1956.....	² 216,606	(³)
1957.....	² 370,483	(³)
1958.....	² 428,347	(³)
1959.....	² 189,263	(³)
Total.....	1,465,501
Value.....	(³)

¹ Estimate.

² Total weight of columbite-tantalite concentrates plus (Cb, Ta)₂O₅ content of euxenite concentrate.

³ Company confidential.

Government contract was completed and stockpile purchases under Public Law 733, 84th Congress, had ceased on December 31, 1958. Domestic mine shipments from 1884 to 1959 are shown in table 35. State totals for the same period are shown in table 36.

The statistics present a revealing picture. Pegmatites, despite their long production history, are not a significant domestic source of large amounts of ore. Their very physical characteristics and the mining methods used to exploit them set severe limitations upon their production capabilities. Unless some of the larger domestic pegmatites are proven richer than suspected or new ones discovered, the United States must turn to other ores for domestic supplies. These other sources are placers, such as that worked by Porter Brothers Corp., carbonatites such as those at Gunnison, Colo., columbium-bearing titanium minerals and bauxite, and alumina plant wastes such as in Arkansas. Of course, the possibility also exists that large granite bodies containing abundant accessory columbite (such as are mined in Nigeria) may eventually be found in the United States, although columbium-bearing granites of economic grade have not been reported to 1960.

Metal, Compounds, and Alloys

All figures on production of columbium and tantalum metals, compounds, and alloys were

TABLE 36.—*Domestic mine shipments of columbium-tantalum mineral concentrates by State, 1884-1959, pounds*

State	Concentrate, pounds	Percent of total	Rank
Idaho ¹	1,192,351	81.5	1
South Dakota.....	196,393	13.4	2
New Mexico.....	34,352	2.3	3
Colorado.....	19,592	1.3	4
North Carolina.....	15,627	1.0	5
Arizona.....	3,105	.2	6
New Hampshire.....	2,455	.2	7
Maine.....	764	.1	8
Wyoming.....	542		9
Connecticut.....	253		10
Virginia.....	67		11
Total United States ¹	1,465,501		

¹ Weight of columbite-tantalite concentrates plus (Cb, Ta)₂O₅ content of euxenite concentrate.

company confidential through 1956. However, examination of ore consumption records, maintained since 1952, indicates an almost steady growth of all phases of the industry.

Statistics for combined production of ferro-columbium and ferrotantalum-columbium could be published for the first time in 1957. Production was 526 tons containing 313 tons of columbium and tantalum. Shipments weighed 430 tons, with 252 tons of contained columbium and tantalum valued at \$2,336,383. In 1959 production was 607 tons containing 355 tons of columbium and tantalum. Shipments weighed 564 tons, containing 330 tons of columbium and tantalum valued at \$2,247,351. Metal production still cannot be revealed exactly, but first reduction-metal production in 1957 was about 90 tons of tantalum and 10 tons of columbium; in 1959, corresponding figures were approximately 120 and 60. Data on production of compounds are not published. Several hundred tons each of columbium and tantalum compounds are produced annually. Very little of the output is sold; most of it is consumed within the plant to produce the metals.

CONSUMPTION

Minerals of the columbite-tantalite series and columbium- and tantalum-bearing tin slags are the principal raw materials for the columbium-tantalum industry. Euxenite, microlite, and concentrates produced from pyrochlore are of minor importance. The

low-grade tin slags are used almost exclusively to produce ferroalloys. During 1958, as measured by the metal content of the raw material consumed, 80 percent of columbium was contained in columbite or pyrochlore, 19 percent in tantalite, and 1 percent in tin slag, compared with 63 percent, 11 percent, and 26 percent, respectively, in 1957. All euxenite was processed for government stockpiling and none was considered as industrial consumption. In 1958 tantalum was contained 58 percent in tantalite, 40 percent in columbite, and 2 percent in tin slag, compared with 44 percent, 33 percent, and 23 percent, respectively, in 1957.

Significant domestic consumers of such materials numbered 11 in 1957 and 12 in 1959. In order of importance, measured by the tantalum content of concentrates and slag reported consumed, domestic consumers of tantalum ores were

1957

Fansteel Metallurgical Corp., North Chicago, Ill., and Muskogee, Okla.
 Union Carbide Metals Co., Division of Union Carbide Corp., Niagara Falls, N.Y.
 Kennametal, Inc., Latrobe, Pa.
 Kawecki Chemical Co., Boyertown, Pa.
 Molybdenum Corporation of America, Washington, Pa.
 Wah Chang Corp., Albany, Oreg., and Glen Cove, N.Y.
 Shieldalloy Corp., Newfield, N.J.
 Vanadium Corporation of America, Cambridge, Ohio
 Transition Metals and Chemicals, Inc., Walkill, N.Y.
 Reading Chemical Co., Wyomissing, Pa.
 E. I. duPont de Nemours and Co., Newport, Del.

1959

Fansteel Metallurgical Corp.
 Kawecki Chemical Co.
 Wah Chang Corp.
 Union Carbide Metals Corp.
 Kennametal Inc.
 Molybdenum Corporation of America
 National Research Corp., Cambridge, Mass.
 Shieldalloy Corp.
 Vanadium Corporation of America
 Reading Chemical Co.
 E. I. duPont de Nemours and Co.
 Transition Metals & Chemicals, Inc.

Consumers of columbium in concentrate and slag, in order of size, were

1957

Union Carbide Metals Co.
 Fansteel Metallurgical Corp.
 Molybdenum Corporation of America
 Vanadium Corporation of America
 Transition Metals and Chemicals Corp.
 Reading Chemical Co.
 Kennametal, Inc.
 Shieldalloy Corporation
 Wah Chang Corp.
 Kawecki Chemical Co.
 E. I. duPont de Nemours and Co.

1959

Union Carbide Metals Co.
Molybdenum Corporation of America
Shieldalloy Corp.
Wah Chang Corp.
Kawecki Chemical Co.
Vanadium Corporation of America
Fansteel Metallurgical Corp.
Kennametal, Inc.
Reading Chemical Co.
National Research Corp.
E. I. duPont de Nemours and Co.
Transition Metals and Chemicals Corp.

An additional firm, Mallinckrodt Chemical Works, St. Louis, Mo., processed euxenite to produce mixed columbium-tantalum oxide for Government stockpiling and extracted columbium and tantalum compounds from ores on a toll basis for one of the foregoing firms.

Consumption of columbium and tantalum ores and slags, measured by metal content, is shown in figure 23 for 1952-59; it grew from 350 tons in 1952 to 890 tons in 1957 or more than 150 percent in 6 years. In 1958, there was a sharp recession-generated decline, but in 1959 consumption rebounded to 828 tons, consisting approximately of 595 tons of columbium and 235 tons of tantalum.

Estimates of industrial end uses of the contained columbium and tantalum in 1952-53 and 1956-57 are included in table 37. Data for estimating other years are not available. Tantalum for capacitor use increased significantly. Columbium in alloys

TABLE 37.—Domestic consumption of columbium and tantalum by use, percent

	1952	1953	1956	1957
Columbium:				
Steel.....	60	73	58	55
Other alloys.....	30	18	32	35
Carbides.....	5	4	4	4
Miscellaneous.....	5	5	6	6
Tantalum:				
Ferrotantalum-columbium...	28	46	40	30
Capacitors.....	7	10	26	33
Other electronic.....	18	7	8	8
Chemical equipment.....	20	18	10	10
Carbides.....	16	9	10	13
Miscellaneous.....	11	10	6	6

other than stainless steel increased the most.

The percentage-of-use figures cannot be directly correlated with the metal content of ore consumed owing to processing loss, government stockpiling of some intermediate products, and producer stock adjustments of columbium and tantalum products.

IMPORTS

Concentrates

Columbium mineral concentrates imported in 1935-59 are shown in table 38. Before

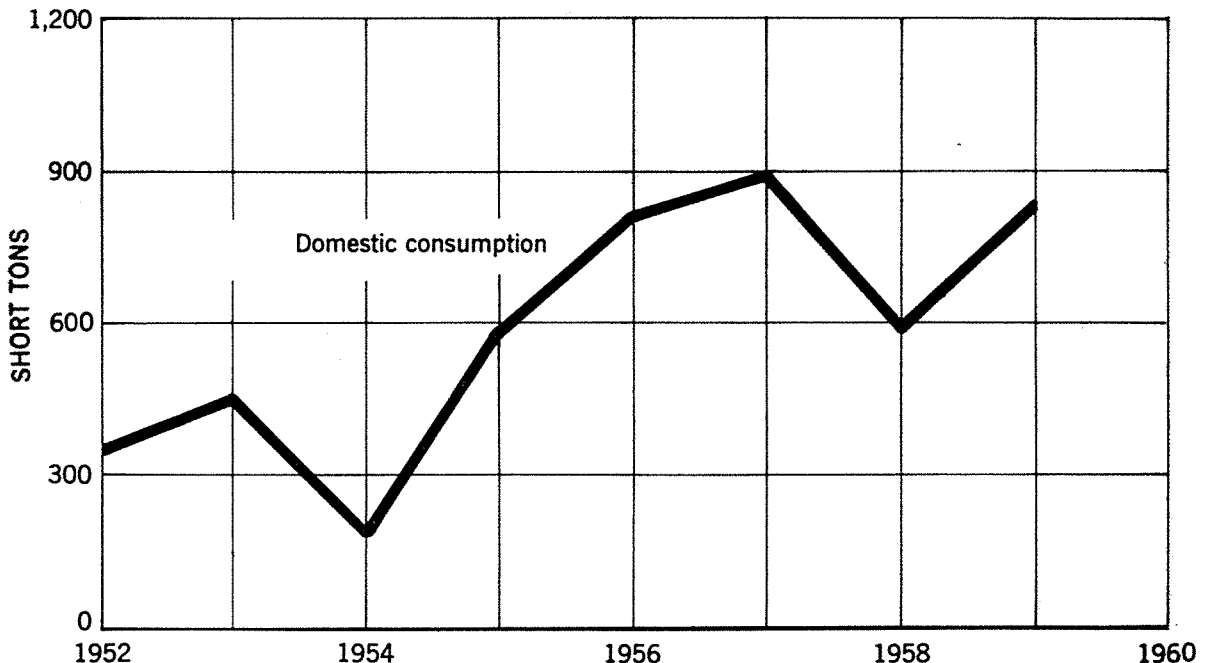


FIGURE 23.—Domestic Consumption of Columbium-Tantalum Concentrates and Tin Slags. (Metal content in tons.)

TABLE 38.—Columbium-mineral concentrate imported for consumption in the United States, 1935-59, by continents and countries, in pounds

Continent and country	1935	1936	1937	1938	1939	1940	1941	1942	1943	1944
North America: Canada.....										
Total.....										
South America:										
Argentina.....							2,914		2,685	
Bolivia.....							2,229	1,133		
Brazil.....			540							
British Guiana.....										
Total.....			540				5,143	1,133	2,685	
Europe:										
Belgium-Netherlands- Luxembourg ¹										
Germany, West.....										
Norway.....										
Portugal.....										
Spain.....										
Sweden.....										
United Kingdom ¹										
Total.....										
Asia:										
Aden ¹									21,600	1,470
India.....										
Japan ¹										
Korea, Republic of.....										
Malaya.....										
Thailand.....										
Total.....									21,600	1,470
Africa:										
Belgian Congo.....								36,422		1,373
British West Africa.....										
French Equatorial Africa.....										
Madagascar.....										
Mozambique.....										
Nigeria.....	1,184,315	996,000	922,114	645,141	109,132	595,220	1,435,312	1,724,800	2,350,329	3,658,084
Rhodesia and Nyasaland, Federation of.....									3,111	23,603
Uganda ²									4,325	
Union of South Africa.....										
Total.....	1,184,315	996,000	922,114	645,141	109,132	595,220	1,435,312	1,761,222	2,357,765	3,683,060
Oceania Australia.....										
Total.....										
Total:										
Pounds.....	1,184,315	996,000	922,654	645,141	109,132	595,220	1,440,455	1,762,355	2,382,050	3,684,530
Value.....	\$397,737	\$257,686	\$306,086	\$228,078	\$37,062	\$210,526	\$504,537	\$608,917	\$844,544	\$1,196,899

See footnotes at end of table.

TABLE 38.—Columbium-mineral concentrate imported for consumption in the United States, 1935-59, by continents and countries, in pounds—Continued

Continent and country	1945	1946	1947	1948	1949	1950	1951	1952	1953	1954
North America: Canada										
Total										
South America:										
Argentina										11,023
Bolivia	1,034	46,834						14,678	10,375	5,714
Brazil		7,717		6,926	8,568	10,981	6,377	5,017	34,391	124,460
British Guiana								800	2,324	
Total	1,034	14,551		6,926	8,568	10,981	6,377	20,495	47,090	141,197
Europe:										
Belgium-Netherlands-Luxembourg ¹				27,125						207,957
Germany, West									40,367	342,886
Norway									68,121	148,732
Portugal									4,410	
Spain						2,103			16,713	
Sweden										
United Kingdom ¹				1,200						
Total				28,325		2,103			129,611	759,575
Asia:										
Aden ¹										
India										
Japan ¹						31,835				
Korea, Republic of									2,000	
Malaya								20,264	101,967	180,225
Thailand										
Total						31,835		20,264	103,967	180,225
Africa:										
Belgian Congo			2,734	113,813	198,585	400,868	177,273	354,732	580,232	976,832
British West Africa										
French Equatorial Africa										11,060
Madagascar										31,183
Mozambique	22,046				1,200		17,082	21,205	57,894	4,575,648
Nigeria	4,220,691	2,411,695	2,818,900	1,822,843	1,349,126	1,280,930	1,336,041	1,450,787	3,167,344	
Rhodesia and Nyasaland									520,460	11,788
Federation of									4,622	4,446
Uganda ²	33,381							6,030	19,891	76,714
Union of South Africa				1,821					34,472	
Total	4,276,118	2,411,695	2,821,634	1,938,477	1,548,911	1,681,798	1,530,396	1,837,376	3,880,293	5,687,671
Oceania: Australia									25,119	35,408
Total									25,119	35,408
Total:										
Pounds	4,277,152	2,426,246	2,821,634	1,973,728	1,557,479	1,726,717	1,536,773	1,878,135	4,186,080	6,804,076
Value	\$1,312,346	\$742,804	\$857,550	\$658,950	\$561,945	\$752,926	\$1,362,393	\$2,368,769	\$6,890,914	\$14,191,142

See footnotes at end of table.

TABLE 38.—Columbium-mineral concentrate imported for consumption in the United States, 1935-59, by continents and countries, in pounds—Continued

Continent and country	1955	1956	1957	1958	1959	Total	Percent of Total
North America: Canada					14,000	14,000	<0.1
Total					14,000	14,000	<0.1
South America:							
Argentina	10,800			2,262	3,591	33,275	<0.1
Bolivia		3,791				42,426	<.1
Brazil	233,012	160,462	54,500	101,992	137,648	895,953	1.4
British Guiana	7,033					10,157	<.1
Total	250,845	164,253	54,500	104,254	141,239	981,811	1.4
Europe:							
Belgium-Netherlands-Luxembourg ¹					13,000	40,125	<0.1
Germany, West	849,310		1,653	46,628	11,578	1,177,126	1.7
Norway	562,759	521,003	236,147	310,858	454,535	2,468,555	3.7
Portugal	168,362	31,024	72,953	65,461	38,083	592,736	.9
Spain	2,525					9,038	<.1
Sweden						16,713	<.1
United Kingdom ¹		11,200	29,621			42,021	<.1
Total	1,582,956	563,227	340,374	422,947	517,196	4,346,314	6.4
Asia:							
Aden ¹		1,350				1,350	<0.1
India						23,070	<.1
Japan ¹						31,835	<.1
Korea, Republic of						2,000	<.1
Malaya	515,688	521,741	127,524	709,077	151,881	2,328,367	3.6
Thailand					13,596	13,596	<.1
Total	515,688	523,091	127,524	709,077	165,427	2,400,168	3.6
Africa:							
Belgian Congo	1,247,901	758,919	905,989	507,725	519,712	6,783,110	10.1
British West Africa	14,521					14,521	<.1
French Equatorial Africa	4,700					4,700	<.1
Madagascar	36,412	10,621	3,075	9,920	11,939	83,027	<.1
Mozambique	64,974	43,124	81,422	171,164	85,249	596,543	<.1
Nigeria	5,739,526	3,593,114	1,804,631	543,925	1,936,296	51,671,944	76.5
Rhodesia and Nyasaland, Federation of	13,529	6,652				52,429	<.1
Uganda ²	24,399	18,780		5,771	2,205	140,209	.2
Union of South Africa	55,539	17,772	31,191	81,159		309,023	.5
Total	7,201,501	4,448,982	2,826,308	1,319,664	2,555,401	59,655,506	88.4
Oceania: Australia	61,586				2,553	124,666	0.2
Total	61,586				2,553	124,666	0.2
Total:							
Pounds	9,612,576	5,699,553	3,348,706	2,555,942	3,395,816	67,522,465	100.0
Value	\$19,912,381	\$8,386,659	\$3,037,706	\$2,345,890	\$2,651,783	\$70,326,210	

¹ Presumably country of transshipment rather than original source.

² Classified by Bureau of the Census as British East Africa.

³ Before 1935 all imports were classed as tantalum ores and are reported in Table 3.

⁴ Classified by Bureau of the Census as from Chile, which is believed to be the country of transshipment only.

⁵ Southern Rhodesia.

Source: Bureau of the Census.

1935 ore imports were all classed as ores of tantalum.

Columbium concentrate imports have totaled 67,515,649 pounds valued at \$70,326,210. The major suppliers during the 25 years were Nigeria, 51,671,944 pounds; Belgian Congo, 6,783,110 pounds; Norway, 2,468,555 pounds; Malaya, 2,328,367 pounds; and Germany, 1,174,126 pounds. The columbium and tantalum pentoxide contents of imports in 1954 are in table 39. These figures have not been obtained since 1954 so that 1959 averages are for receipts by U.S. dealers and consumers.

Imports of tantalum mineral concentrates 1925-59 are shown in table 40.

Tantalum concentrate imports have totaled 13,264,467 pounds valued at \$18,190,740.

TABLE 39.—Grade of U.S. columbium ore imports, 1954 and 1959

Country	1954		1959	
	Cb ₂ O ₅ , percent	Ta ₂ O ₅ , percent	Cb ₂ O ₅ , percent	Ta ₂ O ₅ , percent
Argentina	50.0	15.0		
Bolivia	53.0	18.0		
Brazil	49.8	22.3	40	29
Germany, West	40.7	32.9		
Norway	41.3	10.7	51	1
Portugal	37.4	28.8		
Malaya	55.0	15.5	58	16
Belgian Congo	41.3	30.8		
Rhodesia and Nyasaland, Federation of	55.9	14.2		
Madagascar	40.3	30.9		
Mozambique	46.8	25.8		
Nigeria	54.4	13.0	65	6
Uganda	43.6	20.3	54	0
Union of South Africa	40.0	24.3		
Australia	41.5	22.2		

TABLE 40.—*Tantalum mineral concentrates imported for consumption in the United States, 1925-59, by continents and countries, pounds*

Continent and country	1925	1926	1927	1928	1929	1930	1931	1932	1933	1934	1935
North America: Canada.....											
Total.....											
South America:											
Argentina.....											
Brazil.....											
French Guiana.....											
Total.....											
Europe:											
Belgium-Luxembourg ¹											
Germany, West.....											
Netherlands ¹											
Norway.....											
Portugal.....											
Spain.....											
Sweden.....											
United Kingdom ¹											
Total.....											
Asia:											
India.....											
Japan ¹											
Malaya.....											
Thailand.....											
Total.....											
Africa:											
Ango-Egyptian Sudan ¹											
Belgian Congo.....											
Madagascar.....											
Mozambique.....											
Nigeria.....											
Rhodesia and Nyasaland, Federation of.....											
Uganda ²											
Union of South Africa.....											
Total.....											
Oceania: Australia.....	5,022	350	15,119	15,250	8,474	6,288	36,131	14,257	24,630	6,083
Total.....	5,022	350	15,119	15,250	8,474	6,288	36,131	14,257	24,630	6,083
Grand total:											
Pounds.....	5,022	350	15,119	15,250	8,474	6,288	36,131	14,257	24,630	6,083
Value.....	\$1,347	\$149	\$20,012	\$19,418	\$3,350	\$6,289	\$51,033	\$20,530	\$35,441	\$9,342

See footnotes at end of table.

TABLE 40.—*Tantalum mineral concentrates imported for consumption in the United States, 1925-59, by continents and countries, pounds—Continued*

Continent and country	1936	1937	1938	1939	1940	1941	1942	1943	1944	1945
North America: Canada.....									700	
Total.....									700	
South America:										
Argentina.....								2,420	8,233	
Brazil.....					(³)	161,996	237,210	416,874	440,460	68,229
French Guiana.....										
Total.....						161,996	237,210	419,294	448,693	68,229
Europe:										
Belgium-Luxembourg ¹										
Germany, West.....										
Netherlands ¹	2,032									
Norway.....										
Portugal.....										
Spain.....										
Sweden.....										
United Kingdom ¹										
Total.....	2,032									
Asia:										
India.....								1,805	2,442	
Japan ¹										
Malaya.....										
Thailand.....										
Total.....								1,805	2,442	
Africa:										
Anglo-Egyptian Sudan ¹									98	
Belgian Congo.....					(³)	146,904	309,843	157,073	332,312	485,986
Madagascar.....								3,567	4,751	
Mozambique.....								5,757	18,116	31,410
Nigeria.....										
Rhodesia and Nyasaland, Federation of.....					(³)	⁴ 64,773	⁴ 18,971	⁴ 40,481	⁴ 12,794	⁴ 9,967
Uganda ²	4,480							3,063	7,277	11,348
Union of South Africa.....						217		1,332	632	2,027
Total.....	4,480					211,894	328,814	211,273	375,980	540,738
Oceania: Australia.....	14,246	20,897	41,706	56,561	(³)	29,574	1,470	10,708	9,315	21,125
Total.....	14,246	20,897	41,706	56,561	(³)	29,574	1,470	10,708	9,315	21,125
Grand total:										
Pounds.....	20,758	20,897	41,706	56,561	490,460	403,464	567,494	643,080	837,130	630,092
Value.....	\$30,751	\$40,742	\$80,092	\$82,900	\$258,514	\$188,936	\$467,418	\$724,066	\$699,473	\$453,141

See footnotes at end of table.

TABLE 40.—*Tantalum mineral concentrates imported for consumption in the United States, 1925-59, by continents and countries, pounds—Continued*

Continent and country	1946	1947	1948	1949	1950	1951	1952	1953	1954
North America: Canada.....									
Total.....									
South America:									
Argentina.....			1,074						
Brazil.....	98,072	71,634	9,202	63,478	13,378		49,813	46,146	255,533
French Guiana.....								10,987	24,809
Total.....	98,072	71,634	10,276	63,478	13,378		49,813	57,133	280,342
Europe:									
Belgium-Luxembourg ¹		3,199			85,683	20,876			
Germany, West.....									62,865
Netherlands ¹				29,500					
Norway.....									
Portugal.....							35,428	154,323	86,279
Spain.....							741		
Sweden.....								4,242	19,251
United Kingdom ¹									
Total.....		3,199		29,500	85,683	20,876	36,169	158,565	168,395
Asia:									
India.....									
Japan ¹					10,691				
Malaya.....							2,087	3,639	1,479
Thailand.....									
Total.....					10,691		2,087	3,639	1,479
Africa:									
Anglo-Egyptian Sudan ¹									
Belgian Congo.....	263,097	311,526	93,939	38,086	211,433	210,402	236,701	507,282	420,562
Madagascar.....									6,173
Mozambique.....									10,893
Nigeria.....		7,998	14,559	4,480	7,543	5,700	2,273		50,018
Rhodesia and Nyasaland, Federation of.....		4 14,928	4 8,914				4 233	4 8,163	4,944
Uganda ²								2,050	2,158
Union of South Africa.....	1,884			1,120				2,036	4,480
Total.....	264,981	334,452	117,412	43,686	218,976	216,102	239,207	519,531	499,228
Oceania: Australia.....	500	9,468				1,467	1,590	20,541	32,428
Total.....	500	9,468				1,467	1,590	20,541	32,428
Grand total:									
Pounds.....	363,553	418,753	127,688	136,664	328,728	238,445	328,866	759,409	981,872
Value.....	\$302,397	\$386,934	\$82,799	\$237,292	\$244,205	\$190,383	\$398,849	\$1,229,534	\$1,972,321

See footnotes at end of table.

TABLE 40.—Tantalum mineral concentrates imported for consumption in the United States, 1925-59, by continents and countries, pounds—Continued

Continent and country	1955	1956	1957	1958	1959	Total	Percent of total ⁶
North America: Canada.....						700	<0.1
Total.....						700	<0.1
South America:							
Argentina.....	6,614	4,409		11,635	1,611	35,996	0.3
Brazil.....	221,834	140,039	199,205	159,015	205,898	2,858,016	22.3
French Guiana.....	23,085	14,532	3,075			76,488	.6
Total.....	251,533	158,980	202,280	170,650	207,509	2,970,500	23.2
Europe:							
Belgium-Luxembourg ¹			6,391	10,681	21,871	148,701	1.2
Germany, West.....	594,030			135,431		792,326	6.2
Netherlands ¹						31,532	.2
Norway.....	11,729			32,513	27,227	71,469	.6
Portugal.....	6,614	7,054	5,966			295,664	2.3
Spain.....	11,276					12,017	.1
Sweden.....				992		24,485	.2
United Kingdom ¹	28,533					28,533	.2
Total.....	652,182	7,054	12,357	179,617	49,098	1,404,727	11.0
Asia:							
India.....						4,247	<0.1
Japan ¹						10,691	.1
Malaya.....	5,853			6,000		19,058	.2
Thailand.....					4,515	4,515	<.1
Total.....	5,853			6,000	4,515	38,511	0.3
Africa:							
Anglo-Egyptian Sudan ¹						98	<0.1
Belgian Congo.....	539,214	953,092	491,124	370,120	166,317	6,245,013	48.9
Madagascar.....	10,693	20,165	6,835	7,716	9,375	60,957	.5
Mozambique.....	57,184	4,409	24,046	149,777	68,343	322,970	2.5
Nigeria.....	⁵ 303,692	31,174	16,815	34,537	50,902	584,974	4.6
Rhodesia and Nyasaland, Federation of.....	18,326	22,166	38,975	77,667	44,720	386,022	3.0
Uganda ²	8,507			2,034	2,690	43,607	.4
Union of South Africa.....	14,428	6,511	6,910	27,368	24,805	93,750	.7
Total.....	952,044	1,037,517	584,705	669,219	367,152	7,737,391	60.6
Oceania: Australia.....	46,074	109,314	28,923	10,102	24,565	622,178	4.9
Total.....	46,074	109,314	28,923	10,102	24,565	622,178	4.9
Grand total:							
Pounds.....	1,907,686	1,312,865	828,265	1,035,588	652,839	13,264,467	100.0
Value.....	\$4,820,453	\$1,180,118	\$948,638	\$1,838,338	\$1,165,536	\$18,190,740

¹ Presumably country of transshipment rather than original source.

² Classified by Bureau of the Census as British East Africa.

³ Not available.

⁴ Southern Rhodesia.

⁵ Presumably includes material classified by producers as columbite.

⁶ Because 490,460 pounds imported in 1940 is not distributed by country, percentage is therefore calculated against a total of 12,774,007.

Source: Bureau of the Census.

TABLE 41.—Grade of U.S. tantalum ore imports

Country	1954		1959	
	Ta ₂ O ₅ , percent	Cb ₂ O ₅ , percent	Ta ₂ O ₅ , percent	Cb ₂ O ₅ , percent
Brazil.....	48.9	21.2	53	19
French Guiana.....	48.2	19.7
Germany, West.....	60.8	22.4
Portugal.....	38.8	32.2	33	34
Sweden.....	30.3	21.7
Malaya.....	24.9	8.8
Belgian Congo.....	34.2	26.7	33	39
Rhodesia and Nyasaland, Federation of.....	38.9	29.0	63	7
Madagascar.....	42.5	33.7	51	27
Mozambique.....	49.5	28.5	46	30
Nigeria.....	46.8	26.3	30	46
Uganda.....	45.3	12.2
Union of South Africa.....	29.4	29.9	45	29
Australia.....	39.9	27.6	46	23

The major suppliers over the 35 years were Belgian Congo, 6,245,013 pounds; and Brazil, 23,858,016 pounds. The Ta₂O₅ and Cb₂O₅ contents of imports have not been obtained since 1954. The average grades of 1954 imports by country of origin are given in table 41, with the average grade of 1959 receipts for comparison.

Import statistics on columbium-tantalum-bearing tin slags are not obtained, but in 1957 several million pounds were imported from Africa. In 1959 such imports totaled only a few hundred thousand pounds. They constitute an important resource for ferroalloy manufacture and are potential commercial sources of both elements in the pure metal form.

Metals, Alloys, and Compounds

Import statistics for most columbium and tantalum alloys and compounds cannot be obtained because they are included in miscellaneous metal categories. The small amount of data obtained on imports of ferro-columbium indicates that between 10 and 25 tons were imported from 1952 through 1959 from the United Kingdom, Norway, and Canada.

Very little columbium and tantalum metal was imported before 1955. About 10,000 pounds of tantalum and 12,000 pounds of columbium, in all forms, were imported from the United Kingdom, West Germany, and Austria from 1955 through 1959.

TOTAL DOMESTIC SUPPLY

Except for 1956-59 the total domestic ore supply base has been essentially the same as ore imports (fig. 24). In 1956 domestic production surged tremendously, but the ore was produced mostly under special contract, and

the oxides made from it were placed in Government stockpiles.

In addition to imports of concentrate and domestic concentrate production, large imports of columbium-tantalum-bearing tin slags have augmented supply. From 1952 to 1959 slag imports, much of which was consigned to the Government stockpile, totaled more than 60 million pounds. From this figure, plus the previously presented totals for concentrate imports and domestic concentrate production, the great oversupply of raw materials during the period is readily apparent. In 1957, the year of highest industrial ore consumption, 5,750,000 pounds of concentrates and tin slags combined containing about 1,200,000 pounds of columbium and 600,000 pounds of tantalum were consumed by industry. Most of the excess imports over consumption were delivered to the U.S. Government stockpile, and the remainder, amounting to a 1- to 2-year supply, was stockpiled by dealers and consumers as a protection against erratic ore supplies, which have plagued them in the past.

Exports

Exports of columbium-tantalum-bearing materials, 1942-59, are itemized in table 42. An unknown quantity of material exported in such categories as "wire of other metals" or "articles of metal not specially provided for" is not included and cannot be estimated. From 1952 to 1959, exports of all listed classes totaled 366,602 pounds, valued at \$2,761,084. In 1959, exports totaled 33,880 pounds worth \$338,800, compared with 645 pounds valued at \$21,337 in 1942.

Destinations for each class of materials were as follows:

Columbium Ore and Concentrate	
<i>Major</i>	<i>Minor</i>
Canada	Switzerland
West Germany	France
Japan	
United Kingdom	
Sweden	
Columbium Metal and Alloys in Crude Form and Scrap	
<i>Major</i>	<i>Minor</i>
United Kingdom	Canada
West Germany	France
	Finland
Columbium Semifabricated Forms	
<i>Major</i>	<i>Minor</i>
United Kingdom	France
	Belgium-Luxembourg
Tantalum Ore and Concentrate	
<i>Major</i>	<i>Minor</i>
United Kingdom	France
Sweden	West Germany
Canada	Italy
	Austria
	Japan

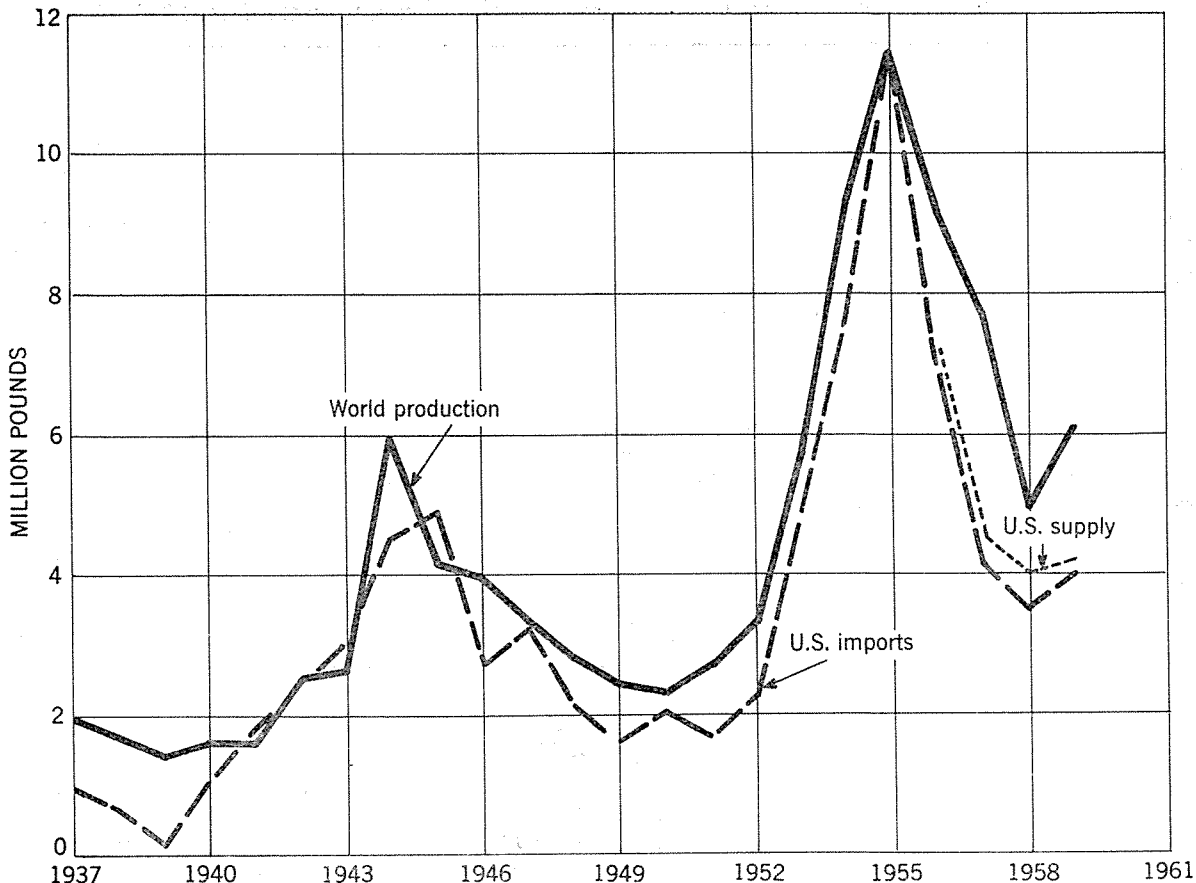


FIGURE 24.—World Production and U.S. Imports of Columbium-Tantalite Concentrates, 1937-39, and U.S. Imports Plus U.S. Production, 1956-59.

Tantalum Metal and Alloys in Crude Form and Scrap

- | | |
|--------------|-----------------------|
| <i>Major</i> | <i>Minor</i> |
| West Germany | Austria |
| Canada | United Kingdom |
| | Sweden |
| | France |
| | Italy |
| | Union of South Africa |

Tantalum Powder

- | | |
|--------------|--------------------|
| <i>Major</i> | <i>Minor</i> |
| West Germany | Brazil |
| | United Kingdom |
| | France |
| | Austria |
| | Canada |
| | Japan |
| | Switzerland |
| | Belgium-Luxembourg |

Tantalum Semifabricated Forms

- | | |
|--------------|--------------------|
| <i>Major</i> | <i>Minor</i> |
| West Germany | Canada |
| France | Mexico |
| | Chile |
| | Norway |
| | United Kingdom |
| | Belgium-Luxembourg |
| | Switzerland |
| | Italy |

Major

Minor

- Ceylon
- Japan
- Australia
- Israel
- Argentina
- Sweden
- Spain
- Iraq
- Union of South Africa
- Kuwait
- New Zealand
- Brazil
- Denmark
- Netherlands
- Finland
- Yugoslavia
- Southern Rhodesia

STOCKS

No data on stocks were collected before 1957, and data collected since then are incomplete. The estimated stocks of ore in the hands of domestic consumers and dealers at the beginning of 1957 and close of 1959 are given in table 43.

TABLE 42.—Exports of columbium- and tantalum-bearing materials, 1942-59

Material	1942		1943		1944		1945		1946	
	Pounds	Value	Pounds	Value	Pounds	Value	Pounds	Value	Pounds	Value
Columbium ores and concentrates							5,000	\$188	7,924	\$3,566
Columbium metal and alloys in crude form, including scrap	² 200	\$277	² 27	\$37			1,334	3,098	4,076	5,296
Tantalum ores and concentrates									21	319
Tantalum metal and alloys in crude form, including scrap	³ 445	21,060	184	15,447	5,052	77,463	11	801	494	26,801
Total, all classes	645	21,337	211	15,484	5,052	77,463	6,345	4,087	12,515	35,982
	1947		1948		1949		1950		1951	
	Pounds	Value	Pounds	Value	Pounds	Value	Pounds	Value	Pounds	Value
Columbium ores and concentrates	10,511	\$4,720	660	\$1,980	18,034	\$11,996	109	(¹)		
Columbium metal and alloys in crude form, including scrap	75	869			90	460	17	\$1,231	42	\$2,646
Tantalum ores and concentrates					3,222	3,450			41	2,221
Tantalum metal and alloys in crude form, including scrap	2,622	30,872	867	33,995	3,483	92,082	1,282	61,322	1,676	74,814
Total, all classes	13,208	36,461	1,527	35,975	24,829	107,988	1,408	62,553	1,759	79,681
	1952		1953		1954		1955		1956	
	Pounds	Value	Pounds	Value	Pounds	Value	Pounds	Value	Pounds	Value
Columbium ores and concentrates					192	850	6,370	\$9,700	10,500	9,312
Columbium metal and alloys in crude form, including scrap	4	\$146	7	\$274	22	880				
Columbium semifabricated forms ^{4 5}			2	559	64	13,320				
Tantalum ores and concentrates	1,058	36,230	77,990	43,964					1,926	2,071
Tantalum metal and alloys in crude form, including scrap	4	146			1,060	3,395	2,000	5,380		
Tantalum powder ⁶	14	639	62	3,025	110	4,750	594	24,767	6,080	245,359
Tantalum semifabricated forms ^{4 6}	1,360	89,054	822	64,613	1,196	89,781	1,390	101,868	1,721	112,930
Total, all classes	2,440	126,215	78,883	112,435	2,644	112,976	10,354	141,715	20,227	369,672
	1957		1958		1959		Totals			
	Pounds	Value	Pounds	Value	Pounds	Value	Pounds		Value	
Columbium ores and concentrates	59,200	\$43,886	54,624	\$37,335	15,060	\$12,730	188,184		\$136,263	
Columbium metal and alloys in crude form, including scrap	15	1,721	44	842	114	2,149	6,067		19,926	
Columbium semifabricated forms ^{4 5}	6	1,335	43	3,820	240	6,101	355		25,135	
Tantalum ores and concentrates			6,600	9,350	10,337	25,021	101,195		122,626	
Tantalum metal and alloys in crude form, including scrap	2,047	44,628	7,757	163,326	4,121	40,283	33,105		691,815	
Tantalum powder ⁶	5,997	228,014	5,773	212,048	1,988	75,870	20,618		794,472	
Tantalum semifabricated forms ^{4 6}	2,830	206,627	5,719	129,328	2,020	176,646	17,058		970,847	
Total, all classes	70,095	526,211	80,560	556,049	33,880	338,800	366,582		2,761,084	

¹ Not available.² Ferrocolumbium.³ Includes compounds.⁴ Not elsewhere classified.⁵ Before 1952 included in columbium metal, alloys, and scrap.⁶ Before 1952 included in tantalum metal, alloys, and scrap.

TABLE 43.—Domestic stocks, columbium-tantalum ores, pounds

Material	Beginning stocks, 1957	End stocks, 1959
Columbium concentrate.	1,300,000	3,500,000
Tantalum concentrate...	500,000	2,500,000
Tin slags.....	¹ <3,000,000	¹ >6,000,000
Total.....	<4,800,000	>12,000,000

¹ Exact figure company confidential.

Total quantities in Government stockpiles cannot be revealed. However, beginning with World War II large quantities of columbium- and tantalum-bearing materials have been purchased and bartered so that total stockpile objectives have been greatly exceeded, and stocks amount to many millions of pounds of contained columbium and tantalum. Materials in various stockpile inventories (National Stockpile, Defense Production Act, and Interior Department) include columbite, tantalite, metal-bearing tin slags,

columbium pentoxide, tantalum pentoxide, columbium-tantalum pentoxide, potassium-tantalum fluoride, ferrocolumbium, tantalum metal, and tantalum fabricated forms. Totals that can be revealed in 1960 are 1,050,000 pounds of contained (Cb, Ta)₂O₅ purchased from Porter Brothers Corp.; 59,104 pounds of contained (Cb, Ta)₂O₅ purchased under Public Law 733 (p. 105); and at least 15 million pounds of additional contained columbium and tantalum metal in various forms in DPA stockpiles.

PRICES

Ores

Columbite and tantalite prices during 1939-59 are compared in table 44. The price of columbite was very stable until 1950. During World War II, Government price controls precluded a price rise, but during the Korean war, demand for ore to produce ferrocolumbium rapidly outstripped the supply available. To assure emergency supplies the Government began buying ore containing 50 percent combined Cb₂O₅ plus Ta₂O₅ in unspecified ratios for \$3.40 per pound of contained oxides. From 1952 until 1955 this

TABLE 44.—Comparison of columbite and tantalite prices, 1939-59

Year	Columbite		Tantalite	
	Price	Basis per pound of contained Cb ₂ O ₅ +Ta ₂ O ₅	Price	Basis per pound of contained Ta ₂ O ₅ only
1939.....	\$0.35	65 percent combined pentoxides.....	\$2.50	60 percent tantalum pentoxide.
1940.....	.35	do.....	2.50	Do.
1941.....	.35	do.....	2.00	Do.
1942.....	\$.50-.56	do.....	2.25	Do.
1943.....	.50	50 percent contained pentoxides.....	2.25	Do.
1944.....	.50	do.....	3.50	Do.
1945.....	.54	50-55 percent contained pentoxides....	3.50	Do.
1946.....	.54-.45	do.....	3.50	Do.
1947.....	.45-.58	do.....	3.00	Do.
1948.....	.58-.67	do.....	3.00	Do.
1949.....	.62-.72	do.....	3.00	Do.
1950.....	.69-1.62	do.....	3.25	Do.
1951.....	1.56-1.62	do.....	3.50	Do.
1952.....	1.60-3.40	50 percent contained pentoxides.....	8.00	65 percent tantalum pentoxide.
1953.....	3.40	do.....	8.00	Do.
1954.....	3.40	do.....	8.70	Do.
1955.....	3.40-2.25	do.....	8.70	Do.
1956.....	1.25-1.35	65 percent pentoxides, Cb : Ta = 10 : 1	7.00	Do.
	1.05-1.15	65 percent pentoxides, Cb : Ta = 8½ : 1	\$3.90-6.25	60 percent tantalum pentoxide.
1957.....	1.40-1.15	65 percent pentoxides, Cb : Ta = 10 : 1		
	1.20-1.05	65 percent pentoxides, Cb : Ta = 8½ : 1	6.25-4.25	Do.
1958.....	1.15-1.05	65 percent pentoxides, Cb : Ta = 10 : 1		
	1.05-.95	65 percent pentoxides, Cb : Ta = 8½ : 1	4.25-3.50	Do.
1959.....	1.05-1.20	65 percent pentoxides, Cb : Ta = 10 : 1		
	.95-1.05	65 percent pentoxides, Cb : Ta = 8½ : 1		

price was an effective price support. When the purchase program ceased in 1955, the oversupply from the greatly overexpanded ore production facilities caused a sudden price decline as the market sought its true level. Marginal producers, who could not operate profitably at lower prices, halted production, and by 1960 the supply-demand picture and prices reached a more or less stable plateau.

The history of prices for tantalite is slightly more complex. Until 1950 it resembled, although at a higher price, columbite pricing. As with columbite, the Government buying program during the Korean war caused prices to rise drastically. A 65 percent Ta₂O₅ concentrate during this period sold for as high as \$8.70 per pound of contained Ta₂O₅. The situation was further aggravated by consumers purchasing more than their requirements as a safeguard against future shortages. When the Government withdrew from the market, prices began to decline. However, compared with columbite, tantalite production had not expanded to the same degree, and many optimistic estimates were released on the future for tantalum. As a result, industrial firms continued to stockpile ore well into 1958. By that time it became apparent that tantalum market developments would not be as swift as previously believed and that tantalite ore supplies were not as short as supposed. Consumers began to use part of their excess ore stocks, and the price for 60-percent concentrates was about \$6 in 1958 and 1959. This price continues very sensitive to supply-demand changes such as presence of the Government in the market or civil strife and political upheaval in supplier nations.

Metal

Prices for columbium and tantalum metals since 1939 are presented in table 45. Columbium was really a laboratory curiosity until about 1955, and price reductions since that date reflect upscaling of production facilities and increased competition. Tantalum metal was turned out by only one producer until about 1956; price reductions since then reflect new process efficiencies, lower ore prices, and increased competition. Prices for both metals vary greatly depending upon specifications, form, and quantity ordered. The prices listed in table 45 represent an idealized history for typical products. A true reflection of the 1959 price structure can be gained in table 46. Historical price changes for each item in table 46 would be roughly

TABLE 45.—*Metal prices, 1939–59*

Year	Columbium		Tantalum ¹ price
	Price	Basis per pound	
1939.....	\$227–254	Metal.....	\$36
1940.....	227–254	...Do.....	36
1941.....	227–254	...Do.....	36
1942.....	227–254	...Do.....	36
1943.....	227–254	...Do.....	36
1944.....	250	...Do.....	36
1945.....	250	...Do.....	36
1946.....	250	...Do.....	36
1947.....	250	...Do.....	36
1948.....	62.75	Powder.....	36
1949.....	62.75	...Do.....	28
1950.....	62.75	...Do.....	28
1951.....	62.75	...Do.....	28
1952.....	62.75	...Do.....	28
1953.....	75	...Do.....	28
1954.....	75	...Do.....	51
1955.....	55–70	...Do.....	51
1956.....	55–70	...Do.....	49
1957.....	57–70	...Do.....	49
1958.....	40–50	...Do.....	49
	55–70	Roundels...	
	65–80	Rough ingot	
1959.....	30	Powder.....	40
	36	Roundels...	
	50	Rough ingot	

¹ All tantalum prices based on powder.

TABLE 46.—*Typical columbium and tantalum metal prices, 1959, per pound¹*

Columbium	Purity, percent	Price
Powder.....	99.5+	\$30–\$60
Powder, technical grade.....	(²)	17– 31
Roundels.....	99.5+	36– 50
Pellets.....	99.8+	31– 61
Pellets, technical grade.....	(²)	16– 25
Sintered bar.....	99.8+	45– 65
Rough ingot.....	99.5+	50– 65
Finished ingot.....	99.8+	55– 90
Wire.....		70–130
Strip or sheet.....		50–100

Tantalum	Purity, percent	Price
Powder.....	99.5+	\$35–\$50
Powder capacitor grade.....	(³)	48– 60
Roundels.....	99.5+	35– 60
Sintered bar.....	99.8+	45– 65
Rod.....		50– 80
Wire.....		60–325
Sheet.....		45–100
Foil.....		70–160

¹ Range due to company variations, specifications, and size of order.

² Purity of technical grade varies.

³ Purity of capacitor grade varies.

TABLE 47.—*Ferrocolumbium and ferrotantalum-columbium prices, 1939-59*

Year	Ferrocolumbium, price (per lb. of Cb.) ¹	Ferrotantalum-columbium, price (per lb. of Cb+Ta) ²
1939	\$2.25-\$2.35	
1940	2.25- 2.35	
1941	2.25- 2.35	
1942	2.25- 2.30	
1943	2.25- 2.30	
1944	2.25- 2.30	
1945	2.25- 2.30	
1946	2.25- 2.30	
1947	2.25- 2.60	
1948	2.60- 2.90	
1949	2.90	
1950	2.90- 4.90	\$2.67-\$3.75
1951	4.90	3.75
1952	4.90	3.75
1953	4.90- 6.40	3.75- 4.75
1954	6.40-12.00	4.75- 6.25
1955	12.00- 6.90	6.25- 4.65
1956	6.90	4.65
1957	6.90- 4.90	4.65- 4.25
1958	4.90- 3.35	4.25- 3.05
1959	3.35	3.05

¹ Basis, 50-60 percent Cb.
² Basis, 40 percent Cb + 20 percent Ta.

TABLE 48.—*Typical tantalum foil and sheet prices, 1958, per pound, annealed¹*

Thickness, inch	Finish	Size of order		
		1 pound	10 pounds	20 pounds
Foil:				
0.0005..	Capacitor grade.	\$101-\$108	\$87-\$101	\$79-\$97
.001..	do.....	91- 97	78- 91	72- 87
Sheet:				
.003..	Standard..	74- 86	71- 74	67- 68
	Select.....	80- 94	77- 80	73- 74
.006..	Standard..	58- 68	56- 58	53- 54
	Select.....	64- 74	61- 64	58- 59
.009..	Standard..	56- 66	54- 57	51- 52
	Select.....	61- 72	58- 62	56- 57
.011-.015..	Standard..	56- 65	53- 56	50- 51
	Select.....	61- 71	58- 61	55- 56
.060-.100..	Standard..	50- 60	48- 50	45- 46
	Select.....	54- 64	52- 55	49- 50

¹ Foil: 4¼ width; sheet: random width by random length.

proportional to those listed in table 45 for the average metals.

Ferroalloys

Ferroalloy prices since 1939 are shown in table 47. The results of short supply during

TABLE 49.—*Typical prices, tantalum wire and rod, 1958, per pound*

Diameter, inch	Size of order		
	10 pounds	75 pounds	150 pounds
0.010.....	\$88-\$102	\$88-\$94	\$88-\$89
.020.....	77- 92	73- 84	73- 80
.040.....	72- 87	69- 80	69- 80
.100.....	63- 73	60- 69	60- 66
.250.....	61- 70	58- 67	58- 64

TABLE 50.—*Tariffs on columbium and tantalum metal and alloys (ad valorem), percent*

	1930 Tariff Act	GATT	
		Effective 1957 ¹	Effective 1958 ¹
Ductile tantalum, ductile columbium, and ductile nonferrous alloys of tantalum or columbium ²	40		
Columbium or tantalum metal.....	25	12.5	
Alloys of columbium, tantalum, or both with two or more metals of the group: Ca, Zr, Ti, U, B, Ba, Sr, Th, or V.....	25	12.5	
Alloys containing columbium or tantalum and 1 or more of the group: Al, Cr, Co, Cu, Mn, Ni, and Si.....	25	18	17
Ferrocolumbium or ferrotantalum-columbium and alloys used in the manufacture of steel or iron not elsewhere specified....	25	12.5	
Abrasives and manufactured abrasives containing more than 0.2 percent of tantalum or columbium.....	60	53	50
Columbium or tantalum ore or concentrate.....	(³)		
Metallic mineral substances crude: includes drosses, residues, skimmings, flue dust, slags, etc.....	(³)		

¹ The tariffs became effective on June 30 of the years specified.
² This class dates back to 1909 and apparently was designed to encompass material for incandescent lamp filament use. Today this high tariff rate is bypassed by entry as semifabricated shapes. For example, tantalum foil classed as "article of metal not specially provided for" is dutiable at 22.5 percent. Tantalum wire entered as "wire of other metals" is dutiable at 12.5 percent.
³ Free.

and after the Korean war are readily apparent.

Mill Shapes

Prices for columbium shapes were not regularly quoted in 1958. Prices for tantalum sheet, foil, rod, and wire are quoted in tables 48 and 49. Particular items vary in price

because pricing policies and purities differ with each firm. Of the many more regularly quoted tantalum mill products, those in the table represent the general price structure for primary shapes.

TARIFFS

Tariff rates of columbium- or tantalum-bearing items are shown in table 50. Import duties on columbium and tantalum-bearing metal scrap were suspended by Public Law 869, 82nd Congress (1950), as amended, and extended. Modifications of the Tariff Act

of 1930 rates were made by General Agreement on Tariffs and Trade in Geneva (GATT), 1947. These changes and their effective dates also are shown in the table.

For Soviet-bloc countries, 1930 Tariff Act remains in force. However, most columbium- and tantalum-bearing materials cannot be shipped to the Soviet nations owing to embargo under the Mutual Defense Assistance Control Act of 1951 (Battle Act). Such items are administered by the Department of State under the East-West security trade control program.

CHAPTER 6.—STRUCTURE OF THE INDUSTRY

In 1959 the industry had several years of spectacular growth behind it, and further rapid growth was expected. The pattern the industry would have at maturity was still emerging. Certain firms stressed production of only one of the metals; others placed equal emphasis on the production of both. Only Wah Chang Corp. and Fansteel Metallurgical Corp. could be truly called fully integrated, although Union Carbide Corp., in its various operating divisions, was rapidly approaching full columbium-tantalum integration. In general, most firms purchased ore and carried the metal to ingot. A few also were semifabricators to a limited extent, but most had their metal formed by specialized mills on a toll basis.

ORE PRODUCERS

Most columbium-tantalum ore production is controlled by foreign firms. In the United States, ore production has been of minor importance, except for the output of Porter Brothers Corp. of Boise, Idaho. To maintain proper context, however, one should bear in mind that most of the known world columbium resources are controlled by U.S. companies or their wholly or partly owned subsidiaries. Wah Chang Corp., Molybdenum Corporation of America, Kennecott Copper Corp., and E. I. Du Pont de Nemours and Co. own large potential future columbium resources so that in the foreseeable future most production of columbium ore will probably be controlled by U.S. firms.

Among the major columbium-tantalum mining companies by nation are

United States: Porter Brothers Corp., Boise Idaho.
Nigeria:

Amalgamated Tin Mines of Nigeria, Ltd., and its subsidiary Keffi Tin Co., Ltd., London.
Bisichi Tin Co., Ltd., London.
Tin and Associated Minerals, Ltd., a subsidiary of Kennecott Copper Corp., New York.
Jantar Nigeria Co., Ltd., London.
Naraguta Tin Mines
Minerals Research Syndicate, Ltd., London.
Forum Mines, Ltd.
Gold and Base Metals Mines of Nigeria, Ltd., London.
London Tin Corp., Ltd.
United Tin Areas of Nigeria, Ltd., London.
London Nigerian Mines, Ltd.

Brazil:

São João del Rei Tin Co.,
Rio Grande do Norte Society
DEMA—Distribuidora e Exportadora de
Minerios e Adubos, S.A., Araxa.

Australia: Tin and Strategic Minerals, Ltd.

Congo and Ruanda-Urundi (Belgian-controlled at time of listing):

Compagnie Géologique et Minière des Ingenieurs et Industriels Belges, (GEOMINES) Brussels (partly owned by Société Générale de Belgique) and its subsidiary Compagnie de Recherches et d'Exploitations Minières au Ruanda-Urundi (COREM).

Compagnie Minière des Grands Lacs Africains, (M.G.L.) Brussels and its subsidiaries: Compagnie Minière de L'Urega (MINERGA), Compagnie Minière du Lualaba (MILUBA), and Compagnie Minière du Ruanda-Urundi (MIRUDI); Comité National du Kivu (C.N.Ki), Brussels, partially owned by M.G.L.

Syndicat Minier d'Étain (SYMETAÏN), Brussels, a subsidiary of Banque de Bruxelles.

Compagnie des Mines d'Or et d'Étain de la Belgika (BELGIKAOR) Brussels.

Société Minière du Nyangwe, Brussels.

KINORETAIN (?), Brussels.

Société d'Exploitations et de Recherches Minières du Katanga (SERMIKAT), Brussels.

Société des Mines d'Étain du Ruanda-Urundi (MINETAÏN), Brussels.

Compagnie Belge d'Entreprises Minières (COBELMIN).

Société Minière de Muhinga et de Kigali (SOMUKI), Antwerp.

Uganda:

Tororo Exploration Co., Ltd., and Sukulu Mines, Ltd., Tororo (jointly owned by Uganda Development Corp., Olin Mathieson Chemical Corp., and Frobisher, Ltd.).

Kagera Mines, Ltd., Ankole.

Tanganyika: Mbeya Exploration Co., Ltd., Mbeya (jointly owned by N. V. Billiton Maatschappij and Colonial Development Corp.).

Rhodesia and Nyasaland, Federation of: Anglo-American Corp. of South Africa, Lusaka.

Mozambique:

Empresa Mineira do Alto Ligonha (EMDAL), Lourenço-Marques.

Adilia Santa Rita de Lima.

Alice Ataide Campos Costa Co., Nampula.

Marie Alzina Simao.

Monteminas, Ltd., Alto Ligonha.

João da Costa Pinheiro.

Société Minière da Mocubela.

Société Minière da Zambesia.

South-West Africa:

South West Africa Lithium Mines, Ltd., Windhoek.

Tantalite Valley Minerals, Ltd., Karasburg.
P. J. Human, Omaruru.

Canada:

Boreal Rare Metals, Ltd.
 Quebec Metallurgical Industries, Ltd.
 Multi-Minerals, Ltd., Lackner Lake.
 Dominion Gulf Co., Ltd., Nemogosenda.
 Quebec Columbium, Ltd., Oka (jointly owned by Molybdenum Corp. of America and Kennecott Copper Corp.).
 Nova Beaucage Mines, Lake Nipissing (controlled by Consolidated Mining and Smelting Co., Ltd., a subsidiary of the Canadian Pacific Railway).
 Columbium Mining Products, Ltd., Oka (jointly owned by Coulee Lead and Zinc Mines, Ltd., and Headway—Red Lake Gold Mines, Ltd.).
 Oka Rare Metals Mining Co., Ltd., Oka.
 Columbium Metals Corp., Ltd., Oka.
 Chemalloy Minerals, Ltd., Montgary, Manitoba.

Portugal:

Sociedade Mineira de Arga, Ltd., Porto.
 The Portuguese-American Tin Co., Belmonte.
 Sociedade Mineira do Santo Antão, Viana do Castelo Unidos, Ltd., Guarda Estacao.
 Industrial do Minerios de Matozinhos, Matozinhos.

Malaya:

Semeling Tin, Ltd., Semeling.
 Redong Tin Mines, Ltd., Semeling.
 Thai Nyen Tin Mines, Semeling.
 Siau Hin Kongs, Semeling.
 Teh Wan Sang Mine, Bakri.
 Bakri Mining Co., Bakri.
 Kampong Kamunting Tin Dredging Co., Karagan.
 Malayan Tin Dredging, Ltd., London.

Norway: Norsk-Bergverk, Ulefoss.

DEALERS

Although some ore is imported directly by consumers, it is imported mostly by dealers whose activities are centered in New York, N.Y. Columbium and tantalum metals, compounds, and alloys produced in Europe also are handled by certain of these dealers. The leading dealers (in addition to direct import by consuming firms) in columbium-tantalum ores are African Metals Corp., Metallurg, Inc.; Metal Traders, Inc.; Philipp Brothers, Inc.; Standard Ore & Alloys Corp.; and C. Tennant and Sons, all of New York, N.Y. Samincorp, New York, has been named to handle pyrochlore to be produced by Columbium & Metals Corp. at Oka, Quebec, beginning in late 1961.

In addition manufactured products of Murex, Ltd., England, are handled by C. Tennant and Sons, while those of H. C. Starck A. G., Germany, are marketed by Shieldalloy Corp., Newfield, N.J.

METAL AND COMPOUND PRODUCERS

In 1959 all the primary metal producers except National Research Corporation also manufactured compounds as feed for their reduction plants. A few other firms produced intermediate compounds only. The companies, plant locations, products, capacities, and opening dates are shown in table 51.

TABLE 51.—Plant data, major columbium-tantalum producers

Company	Plant location	Opening date	Products	Capacity
Fansteel Metallurgical Corp....	{ North Chicago, Ill....	1922.....	Columbium and tantalum metals, alloys, and compounds.	250 tons of tantalum and columbium.
	{ Muskegee, Okla....	1958.....		
Wah Chang Corp.....	Albany, Oreg.....	1957.....	...Do.....	100 tons of tantalum and columbium.
Union Carbide Metals Co.....	Niagara Falls, N.Y....	1957.....	...Do.....	50 tons of tantalum and columbium.
E. I. duPont de Nemours and Co.	Newport, Del.....	1958.....	...Do.....	10 tons of columbium.
Kawecki Chemical Co.....	Boyertown, Pa.....	1956.....	Columbium and tantalum metals and compounds.	65 tons of tantalum and columbium.
Kennametal, Inc.....	Latrobe, Pa.....	1956.....	Columbium metal, alloys, and compounds.	34 tons of tantalum and columbium.
Minerals Refining Co.....	Murray, Utah.....	1957.....	Columbium and tantalum oxides..	(¹)
Stauffer Chemical Corp.....	Richmond, Calif..	1959.....	Columbium and tantalum chlorides.	480 tons of columbium and tantalum chlorides.
National Research Corp.....	Cambridge, Mass....	1958.....	Tantalum.....	15 tons of tantalum.
United States Industrial Chemicals Co.	Cincinnati, Ohio....	1957.....	Tantalum and columbium metal...	10 tons of tantalum plus columbium.
Mallinckrodt Chemical Works..	St. Louis, Mo.....	1957.....	Columbium-tantalum oxide and separated oxides.	250 tons of oxides.
Murex, Ltd.....	Rainham, England...	1955.....	Columbium and tantalum metals and oxides.	10 tons of tantalum and columbium a year.

¹ Not available.

TABLE 51.—*Plant data, major columbium-tantalum producers—Continued*

Company	Plant location	Opening date	Products	Capacity
Herman C. Starek G.m.b.H....	Goslar, Germany....	1956.....	Tantalum and columbium metals and oxides.	10 tons of metal.
Heraeus Platinschmelze G.m.b.H.	Hanau, Germany....	Tantalum.....	(1)
Metallwerk Plansee G.m.b.H...	Reutte, Austria....	Tantalum and columbium.....	(1)
Compagnie Pechiney.....	France.....	Tantalum metal and oxide.....	5 tons of metal.
Société des Produits Pharmaceutiques CIBA.	Switzerland.....	Tantalum oxide.....	(1)
Société Kuhlmann.....	France.....	Columbium pentoxide.....	12 tons of oxides.
Fabriques de Produits Chimiques de Thann et Mulhouse, S.A.	Do.....	Columbium.....	(1)
Gesellschaft für electro-metallurgie.	West Germany.....	Tantalum.....	(1)
Société Générale Métallurgique de Hoboken.	Belgium.....	Columbium pentoxide.....	(1)

¹ Not available.

FERROALLOY, MASTER ALLOY, AND CARBIDE PRODUCERS

Several ore-consuming firms produced ferroalloys, nonferrous master additive alloys, and columbium or tantalum carbides, borides,

or hydrides. Firms and principal products were

Wah Chang Corp.....	Ferrocolumbium, ferrotantalum-columbium.
Zirconium Metal Corp. (subsidiary of National Lead Co.)....	Ferrocolumbium-aluminum.
Copper Metallurgical Associates.....	Columbium and tantalum borides.
Metal Hydrides, Inc.....	Columbium and tantalum hydrides.
Union Carbide Metals Co.....	Ferrocolumbium, ferrotantalum-columbium, assorted master additive alloys with aluminum, titanium, etc.
Kennametal, Inc.....	Columbium and tantalum carbides.
Reading Chemical Co.....	Chrom-columbium, nickel-columbium, ferrocolumbium.
Transition Metals and Chemicals Co.....	Ferrocolumbium, chrom-columbium.
Shieldalloy Corp.....	Ferrocolumbium, ferrotantalum-columbium.
Haynes Stellite (division of Union Carbide).....	Tantalum carbide.
General Electric Co., Metallurgical Dept.....	Do.
Firth-Sterling.....	Do.
Metal Carbides.....	Do.
Vascoloy-Ramet (subsidiary of Fansteel).....	Do.
Molybdenum Corporation of America.....	Ferrocolumbium.
Atlas Steel, Ltd., Canada.....	Ferrocolumbium, ferrotantalum-columbium.
Fahr Alloy Canada, Ltd., Canada.....	Do.
Metallwerke Plansee, Reutte, Austria.....	Columbium and tantalum carbides.
Treibacher Chemische Werke, Austria.....	Ferrocolumbium.
Société Anonyme Sadaci, Belgium.....	Tantalum carbide.
Kovametalli Oy, Finland.....	Columbium and tantalum carbides.
Société d'Électrometallurgie d'Ugine, France.....	Ferrocolumbium.
Gesellschaft für Electrometallurgie, West Germany.....	Do.
S. P. A. Leghe e Metalli, Italy.....	Do.
Norsk Bergverk A. S., Norway.....	Do.
Electric Furnace Products Co., Norway.....	Do.
A. B. Ferrolegeringar, Sweden.....	Do.
Murex Ltd., England.....	Do.
Blackwell's Metallurgical Works, Ltd., England.....	Do.
Minworth Metals, Ltd., England.....	Do.
London & Scandinavian Metallurgical Co., Ltd., England.....	Do.
Borax Consolidated, Ltd., England.....	Columbium boride.
Showa Denko, Japan.....	Ferrocolumbium.

SEMIFABRICATORS

Some ingot and mill shapes are produced by the primary metal producers and some by firms that specialize in melting, rolling, drawing, or other operations. Facilities were more than sufficient to treat all the primary metal produced.

In 1959, six firms had facilities for pressing columbium or tantalum powder and sintering the resultant compacts to dense bars: Fansteel Metallurgical Corp., Kennametal, Inc., Kawecki Chemical Co., Wah Chang Corp., Allegheny-Ludlum Steel Corp., and General Electric Co. Consumable electrode arc-melting facilities, used at least in part for casting columbium or tantalum, were installed at Universal-Cyclops Steel Corp., Oregon Metallurgical Corp., Wah Chang Corp., General Electric, Union Carbide Metals Co., E. I. duPont de Nemours and Co., Al-

legheny-Ludlum Steel Corp., Federal Bureau of Mines, Kennametal, Inc., Kawecki Chemical Co., National Research Corp., and Haynes Stellite Co. (division of Union Carbide Corp.). Ingot diameter averaged 3 to 6 inches with 16 inches the maximum. Electron-beam melting facilities and experience with columbium or tantalum existed at Temescal, Inc., Wah Chang Corp., Federal Bureau of Mines, and National Research Corporation.

Custom rolling and extrusion facilities are widely available. Among those with experience on columbium or tantalum are Wilbur Driver Co., M & C Nuclear Corp., Allegheny-Ludlum, E. I. du Pont de Nemours and Co., Bridgeport Brass Co., Fansteel Metallurgical Corp., Haynes Stellite, Kawecki Chemical Corp., Kennametal, Inc., and Union Carbide Metals Co. Tubing and other shapes were produced by Fansteel, Superior Tube Co., and others.

CHAPTER 7.—RESEARCH (53, 55)

Research on columbium and tantalum has increased greatly in the past 10 years. Industry, private research institutions, universities, and Government agencies have investigated all facets of technology including geology and ore reserves, analytical techniques, properties, and preparation and use of metals, their alloys, and compounds.

The Bureau of Mines has been evaluating resources and studying extraction, separation, reduction, purification, consolidation, fabrication, and alloy systems. Outstanding work has included sampling and evaluation of the Bear Valley placers in Idaho and other northwestern placers; determination of columbium reserves in Arkansas, and the development of methods to concentrate the minerals and extract the metals; beneficiation studies of columbium-tantalum-bearing minerals in alluvial black-sand deposits in the Northwest; extraction of columbium and tantalum from low-grade ores and tin slags; separation by solvent extraction; anhydrous separation; reduction techniques; and preparation of high-purity metals.

The Bureau of Mines authorized a comprehensive research program for Fiscal Year 1960 (July 1959–June 1960). Research components included halogenation studies, halogenation reactor research, general separation-reduction studies, studies on columbium-tantalum-base alloys, production and properties of high-purity columbium, columbium electrorefining, physical constants of columbium-tantalum-bearing metamict minerals, a field test for columbium-tantalum-bearing minerals, the nature of columbium-tantalum fluorocomplexes, the effect of nuclear irradiation upon columbium and tantalum, and cooperative metallurgical studies with Wah Chang Corp., Stauffer Chemical Co., and Porter Brothers Corp.

Other U.S. Government agencies prominently engaged in or sponsoring research included the Atomic Energy Commission (AEC), Geological Survey, National Aeronautics and Space Administration (NASA), and various Department of Defense agencies. The AEC sponsored basic research on properties of the metals and on alloy systems at Oak Ridge National Laboratory, Ames Laboratory, New York University, University of Illinois, University of Florida, Massachusetts Institute of Technology, Case Institute of

Technology, Armour Institute, Sylvania Electric Products Corp., Horizons, Inc., Carnegie Institute of Technology, and Battelle Memorial Institute. The Department of Defense and agencies of the Air Force, Army, and Navy sponsored programs on consolidation, working, and fabrication; high-temperature properties of the metals and columbium- or tantalum-based alloys; oxidation and protective coating research; and basic studies on phase equilibria, properties, and kinetics. Projects were being conducted at Climax Molybdenum Co., Westinghouse Electric Corp., General Electric Co., Southern Research Institute, Sentralinstitutt for Industriell Forskning (Oslo, Norway), National Bureau of Standards, Ohio State University, University of Illinois, Armour Institute, Nuclear Metals, University of Pennsylvania, Rennslear Polytechnic Institute, Watertown Arsenal, Stanford Research Institute, Battelle Memorial Institute, and Crucible Steel Co. (the most recent Air Force contract for forging and sheet rolling of columbium and columbium-base alloys). NASA has sponsored research at its Lewis Research Center on high-temperature oxidation of alloys. The Geological Survey studies geology and ore deposits. In total, the 1959 rate of Government and Government-supported research and development of columbium and tantalum was probably about \$2 million a year.

Industry and institutions have numerous research programs underway, which include investigations of high-purity metal production, improved high-temperature alloys, processes for concentrating pyrochlore-type ores, improved impact- and corrosion-resistant steels, and numerous basic problems. The producing companies in the industry have been outstanding in this respect. An exact figure on industry research expenditures in 1959 is not available, but it probably was considerably more than the total for Government-sponsored research.

Total research and development expenditures by Government and industry from 1955 through 1959 probably were about \$20 million. The pressing current need is to learn where and when to apply the knowledge that has been gained concerning the properties, purification, and fabrication of these metals, their alloys, and compounds.

CHAPTER 8.—GOVERNMENT PROGRAMS AND MOBILIZATION EXPERIENCE

WORLD WAR II

By 1940, due to the constriction of the European market as a result of Nazi conquests, a large share of the world output of columbite and tantalite was being imported into the United States, where industrial expansion to make the country "the arsenal of democracy" was already underway. The solitary U.S. tantalum producer, Fansteel Metallurgical Corp., North Chicago, Ill., began to expand its facilities to meet the new demands during the same year.

On May 1, 1941, the Office of Production Management (OPM) issued General Metals Order No. 1, which included ferrocolumbium on a list of materials to be placed under inventory control to prevent consumer hoarding. The action was a result of the excessive diminution of available supplies caused by overbuying by consumers.

Imports of columbium and tantalum ores were placed under control by Amendment 4 of War Production Board (WPB) Order M-63 on April 8, 1942. Government stockpiles of both ores to provide for essential needs were established and maintained during the emergency by the Government-owned Metals Reserve Company.

Full allocation control of tantalum in all forms was instituted May 22, 1942, by WPB General Preference Order M-156, and for ferrocolumbium on March 25, 1943, by Order M-296. Rapid expansion of tantalum-production facilities was deemed necessary, and the Government-owned Defense Plant Corporation provided \$5,350,000 in 1942-43 for the Fansteel subsidiary, Tantalum Defense Corp., to erect new facilities next to the Fansteel plant at North Chicago.

The intense enemy submarine campaign necessitated airlifting of urgently needed tantalum-columbium ores into the United States; in 1943 two-thirds of all tantalite imports and part of the columbite imports were brought in by air. By December 1943, use of ferrocolumbium was restricted to those applications in aircraft construction where ferrotitanium or other substitute materials had proved ineffective.

By October 1944, the ferrocolumbium and tantalum supplies had improved sufficiently to permit the WPB to classify the items as in adequate supply to meet all war and essential industry needs. On December 6, 1944, all restrictions on tantalum were lifted by revocation of WPB General Preference Order M-156. Order M-296 pertaining to ferrocolumbium was revoked on July 9, 1945, but Order M-21, which had been issued May 4, 1945, continued control on usage until its revocation on August 21, 1945.

By 1947 the supply of both tantalum and columbium ores far exceeded demand, and Order M-63 was revoked on May 1, 1947.

KOREAN WAR

Allocation Program

The involvement of the United States in hostilities in Korea in June 1950 brought about a defense program and mobilization effort that quickly resulted in growing shortages of many commodities including columbium and tantalum.

Regulation I issued by the National Production Authority (NPA) on September 18, 1950, placed inventory controls on scarce materials including ferrocolumbium, potassium columbium fluoride, columbium oxide, columbium carbide, and all scrap or secondary material containing commercially recoverable columbium. Amendments in 1951 added ferrotantalum-columbium, tantalum metal, and tantalum chemicals to the list. The regulation was terminated effective May 1, 1953.

On October 20, 1950, the NPA issued Order M-3 stipulating that production, distribution, and use of ferrocolumbium and ferrotantalum-columbium be limited to "DO" defense-rated orders, which were reserved under the NPA priorities system to procurement by the Department of Defense and the AEC. The order provided further that use of ferrocolumbium-bearing steels be prohibited wherever ferrocolumbium-tantalum steels might be substituted and that use of either type of steel be prohibited where any other substitute

was available. Subsequent amendments in 1951 placed columbium and tantalum ferroalloys under defense allocation and limited the proportion of columbium and tantalum that could be used in stainless steels.

NPA Delegation 5, December 18, 1950, delegated authority to regulate the allocations and priorities of some commodities, including columbium ores and tantalum ores, to the Department of the Interior. It also delegated claimant responsibilities over mines and plants producing columbium or tantalum metals, alloys, or compounds. An amendment to NPA Delegation 5 in 1952 redelegated authority for allocation of columbium-tantalum ores and claimant responsibilities over mines and concentrating plants to the Defense Materials Procurement Administration (DMPA).

Another NPA order was the Designation of Scarce Materials 1 (DSM-1) issued December 27, 1950. This order closely paralleled NPA Regulation 1 and specifically listed materials not to be hoarded, including ferrocolumbium and ferrotantalum-columbium. Defense Minerals Administration (DMA) issued a parallel order, MO-1, on December 29, 1950, designating columbium and tantalum ores as scarce materials. These restrictions were terminated on March 12, 1953.

NPA Order M-49, March 15, 1951, placed columbium and tantalum metals and their ferroalloys under complete allocation. This order complemented M-3, which had placed columbium- and tantalum-bearing steels under allocation. Order M-1, as amended April 6, 1951, established procedures for virtually complete control over the use of ferroalloys in producing alloy iron, steel, or nonferrous products.

NPA began issuing "Lists of Basic Materials and Alternates" on June 20, 1951. These lists reviewed about 550 materials grouped according to three degrees of available supply at the time: (1) Very short, (2) tight, and (3) fair. Columbium and tantalum were placed in group (1) and remained there during the Korean war.

NPA Order M-80 issued August 15, 1951, superseded orders M-3, M-49, and the part of M-1 that referred to columbium and tantalum products. The new order required all melters and processors to file proposed melting or processing schedules and data concerning inventories. This order was revoked November 1, 1953, by Business and Defense Services Administration, successor to NPA.

Price Controls

Office of Price Stabilization (OPS) General Ceiling Price Regulation, issued January 26, 1951, froze virtually all commodities, including columbium and tantalum, at the prices in effect during the period December 19, 1950, through January 25, 1951. General Overriding Regulation 9, Amendment 4 of OPS, effective August 10, 1951, exempted from price controls all columbium-tantalum ore in crude, concentrated, or beneficiated form.

FACILITIES EXPANSION

To increase production capacity for columbium-tantalum ores and products, the U.S. Government provided funds to producers to construct plants, allowed accelerated tax amortization, and executed contracts to purchase all or part of the production from the expanded facilities.

In 1942-43 the Defense Plant Corporation, a subsidiary of the Reconstruction Finance Corporation, provided \$5,350,000 to finance construction of the plant of the Tantalum Defense Corporation, North Chicago, Ill. The plant was Government-owned but operated by Fansteel Metallurgical Corp. The facilities were later purchased by Fansteel. In 1952, Fansteel was granted an accelerated Tax Amortization Certificate, when it expanded the facility further, costing \$485,000. These Certificates of Necessity permit companies to depreciate a designated percentage of the cost of a new facility over a 5-year period, rather than over the customary 20- to 25-year period. In addition, the Government agreed to purchase for a period of 5 years the crude columbium oxide and potassium tantalum fluoride output of the additional facilities to the extent that the production was not required for normal commercial channels (amounts not to exceed 328,950 pounds of columbium pentoxide and 650,000 pounds of potassium tantalum fluoride).

In 1954, Certificates of Necessity were granted Porter Brothers Corp., Boise, Idaho, for \$1,650,000 for mining and milling facilities for euxenite in Idaho, and to Mallinckrodt Chemical Works, St. Louis, Mo., for \$1,550,000 for a plant in St. Louis to separate valuable contained components such as columbium-tantalum oxides and U_3O_8 . The Government agreed to purchase 1,050,000 pounds of columbium-tantalum oxides through a General Services Administration (GSA) contract (DMP-108), and the AEC purchased coproduct U_3O_8 .

STOCKPILING

The Strategic Materials Act (Public Law 117, 76th Cong.) of June 7, 1939, included provisions for initiation of stockpiling for seven strategic metals. After United States entrance in World War II, the program was expanded to include all critical commodities including columbium and tantalum ores. Stocks were held by the Government Metals Reserve Company. After World War II, stockpiling was extended under the Strategic and Critical Materials Stock Piling Act (Public Law 520, 79th Congress). The avowed purpose was to provide for the industrial and essential civilian wartime needs, not just those of a military nature. Stocks acquired by the Metals Reserve Company were transferred to the new National Stockpile maintained by the GSA. Both the basic and maximum objectives for columbium- and tantalum-bearing materials had been achieved by 1959. Stockpile subobjectives were established in 1960 for tantalum metal, columbium metal, tantalum carbide, columbium carbide, ferrocolumbium, and ferrotantalum-columbium. None of these new objectives was reached during the year. Both commodities are on the List of Strategic and Critical Materials for Stockpiling. Materials were being acquired for the stockpile in 1959 only under the barter program announced by the Department of Agriculture on November 14, 1958. The aim of the program was to exchange perishable farm products for strategic minerals produced in foreign countries.

EXPLORATION LOANS AND PURCHASING AS AN AID TO EXPANSION

Under the Defense Production Act of 1950, columbium and tantalum ores were eligible

for Government assistance to exploration. This section of the act was implemented by DMA Order MO-5, April 6, 1951, which set forth procedures under which Government loans, constituting 90 percent of approved costs, could be obtained to finance exploration for columbium-tantalum ores. DMA was superseded by the Defense Minerals Exploration Agency (DMEA), which issued a similar DMEA Order 1 on March 7, 1952. On March 19, 1954, Amendment 1 to the DMEA order reduced Government participation in columbium-tantalum exploration to 75 percent. Revision of the order on October 18, 1957, further reduced the ratio of Government contribution to 50 percent. On September 13, 1958, DMEA was succeeded by the Office of Minerals Exploration (OME) which continued the policy of supporting 50 percent of the allowable costs of exploration as specified by contract terms.

Under the Defense Production Act, a program of Government purchase of both foreign and domestic columbium-tantalum ores was authorized on January 1, 1952. The purpose of the program was to encourage the expansion of ore-production facilities. Under the regulation, dated May 28, 1952, the U.S. Government agreed to purchase a minimum of 15 million pounds of contained Cb_2O_5 plus Ta_2O_5 in ores. Purchases were halted under the program in May 1955 when the quantity of concentrates received, plus forward commitments to buy, equaled the amount authorized for purchase.

Domestic ores were purchased until December 31, 1958, under Public Law 733, 84th Congress (designed to lessen the impact upon the industry when defense purchases stopped). The GSA regulation governing this program was dated October 3, 1956.

CHAPTER 9.—STRATEGIC FACTORS

Until about 1954 columbium-tantalum resources were assumed to be small. They were believed to be almost entirely in the Eastern Hemisphere and, to a lesser extent, in Brazil. During both World War II and the Korean war, rapid expansion of industrial demand for the ores resulted in serious shortages and stringent Government controls (see Chapter 8). During World War II at the height of the German submarine warfare campaign, supplies of columbite-tantalite were in such critically short supply that shipments of the concentrates from Africa and Brazil had to be airlifted to meet urgent mobilization needs.

To preclude future similar supply shortages the U.S. Government has conducted extensive foreign and domestic purchase and stockpiling programs and has extended loans for domestic exploration (see chapter 8). As a result of these incentive and aid programs many new columbium deposits were discovered, measured reserves in known deposits were increased, and the heavy dependency upon Eastern Hemisphere ores was partially alleviated. Government stockpiles will assure emergency supplies even if access to all overseas sources were denied. Total world resources of columbium are for practical purposes inexhaustible, based on present consumption trends, and tantalum reserves are at least sufficient to satisfy all requirements at present consumption levels.

During both World War II and the Korean war, shortages of production and fabricating facilities arose, which caused many designers

and engineers to exclude columbium or tantalum from their designs unless absolutely necessary. Again in 1956-57 rapidly increasing demand for tantalum foil in space-age electronic devices could not be satisfied because of a lack of rolling facilities. Since that time, however, large increases in domestic processing facilities from ore treatment plants through fabrication installations have been completed.

Some potential supply problems remain. Domestic ores are either low grade, contain unusual mineral suites, or are more difficult to mine or treat than foreign ores. Consequently, although North America is potentially self-sufficient in columbium resources and the Western Hemisphere is potentially self-sufficient in tantalum, lack of actual mine development leaves the United States dependent upon the Eastern Hemisphere for a large part of its total requirements. This dependency probably will be maintained in part. The economy of Nigeria, for example, would be affected if U.S. purchases of columbite were to cease.

The relatively small size of the columbium-tantalum industry might cause problems during a sudden sharp rise in demand. Although present capacity could probably satisfy at least twice the current demand, a rise of greater proportions would require facility expansion. If such a demand developed without precognizance, facility expansion would lag behind requirements during an interim period while new plants were built or old ones expanded.

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