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RECOVERY OF NIOBIUM FROM
NIOBIUM-URANIUM ALLOYS—
AN ANNOTATED BIBLIOGRAPHY

James D. Navratil

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RECOVERY OF NIOBIUM FROM
NIOBIUM-URANIUM ALLOYS
AN ANNOTATED BIBLIOGRAPHY

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Albuquerque Operations Office
U. S. Atomic Energy Commission

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INTRODUCTION

AN ANNOTATED bibliography has been prepared to assist those interested in developing a process to recover niobium-uranium alloys. With the exception of classified literature and most progress reports, this bibliography is complete as of July 1, 1970.

Most of the references contained herein were obtained from *Nuclear Science Abstracts*, and to a minor extent from other sources. The resumes do not necessarily represent an entire summary of the papers presented because emphasis was placed only on the recovery of niobium metal from niobium-uranium alloys.

This bibliography has been divided into four main sections: alloy dissolution, alloy separation, niobium metal preparation, and refining of niobium. A subject index is provided for overlapping subject matter, since each reference was abstracted only once. A book listing, index of organizational symbols, and author index are also provided.

SECTION I

Alloy Dissolution

Nitric Acid

- 1
DIFFERENTIAL SPECTROPHOTOMETRIC DETERMINATION OF URANIUM AND NIOBIUM, C.V.
Banks, K.E. Burke, J.W. O'Loughlin, and J.A. Thompson, *Anal. Chem.*, 29, 995 (1957).

In the analysis of niobium-uranium alloy, a mixture of nitric and hydrofluoric acid is used to dissolve the alloy.

- 2
EXPLOSIVE REACTIONS DURING REPROCESSING OF REACTOR FUELS CONTAINING URANIUM AND ZIRCONIUM OR NIOBIUM, T.A. Gens, CF-58-11-31 (1958).

Niobium-bearing fuel alloys can be explosive when contacted with nitric acid. As with uranium-zirconium-niobium alloy, the explosive reaction can be avoided by addition of fluoride ion, although the explosive conditions have not been thoroughly studied.

- 3
DISSOLUTION OF URANIUM METAL AND ITS ALLOYS, R.P. Larsen, *Anal. Chem.*, 31, 545 (1959).

Dissolution of uranium and its alloys in mixtures of nitric-hydrofluoric acid, bromine-ethyl acetate, and sodium hydroxide-hydrogen peroxide was found satisfactory. Dissolution was found unsatisfactory in nitric acid, aqua regia, and mixtures of hydrochloric acid-oxidant and hydrochloric acid-ethyl acetate.

- 4
DISSOLUTION OF EBWR-TYPE FUEL PLATES SUBSEQUENT TO DISINTEGRATION OF THE CORE WITH WATER, C. Melches, *Energia Nuclear (Madrid)*, 12, 48 (1959).

Two dissolution methods were examined to avoid explosive reactions produced by direct nitric acid dissolution of uranium-zirconium-niobium alloy in the absence of fluoride. They are: pretreatment of the alloy (U, 93.5; Zr, 5; Nb, 1.5%) with heated water prior to nitric acid dissolution and substitution of nitric acid by 12M hydrochloric acid. Under the conditions studied, the former treatment gave a maximum alloy disintegration rate of 1500 mg. per sq. cm. - hr. at 300° and the latter method provided a dissolution rate of 103 mg. per sq. cm. - hr. at 25°.

Ammonium Fluoride

- 5
MODIFIED ZIRFLEX PROCESS FOR DISSOLUTION OF ZIRCONIUM AND NIOBIUM-BEARING NUCLEAR FUELS IN AQUEOUS FLUORIDE SOLUTIONS: LABORATORY DEVELOPMENT, T.A. Gens and F.G. Baird, ORNL-2713 (1959).

Process flow sheets are presented for dissolving uranium-zirconium-niobium alloy in six molar ammonium fluoride and precipitating the uranium as NH_4UF_5 . The niobium can then be separated as the insoluble niobic oxide after nitric acid addition.

- 6
NEW DEVELOPMENTS IN URANIUM-ZIRCONIUM ALLOY FUEL REPROCESSING, T.A. Gens, *Nucl. Sci. Eng.*, 9, 488 (1961).

Processing of uranium-zirconium-niobium alloy in refluxing six molar ammonium fluoride is described. The uranium is precipitated as NH_4UF_5 after ammonium fluoride dissolution and the niobium filtrate contains 0.3% uranium. The NH_4UF_5 is redissolved in nitric acid and the remaining niobium is separated from the soluble uranium as the insoluble niobic acid which contains 0.04% uranium.

- 7
THE CHEMISTRY OF NIOBIUM IN PROCESSING OF NUCLEAR FUELS, T.A. Gens, ORNL-3241 (1962);

A review of recent chemical and industrial work concerning niobium-uranium alloys is presented. Also mentioned is an ammonium fluoride dissolution method to separate the alloy as well as chlorination techniques.

Miscellaneous Solvents

- 8
NUCLEAR ENGINEERING DEPARTMENT PROGRESS REPORT (FOR) MAY 1 - SEPTEMBER 30, 1957, F. Maslan, Ed, BNL-477 (1957).

The product of dissolving niobium-uranium alloy in 85% phosphoric acid - 0.26 molar hydrofluoric acid at 170° was a black liquid which gave a clear green solution upon addition of an oxidizing agent. In a 100 ml volume, 3.46 grams of the alloy dissolved at the rate of 1700 mg/cm/hr.

9

COMPLETE ANALYSIS OF URANIUM-NIOBIUM-ZIRCONIUM ALLOY BY X-RAY ABSORPTION EDGE TECHNIQUE, J.H. Stewart, Jr., T.H. Barton, Jr., and M.R. Ferguson, *Anal. Chem.*, 40, 27 (1968).

The uranium-niobium-zirconium alloy was dissolved in six molar hydrochloric acid and the bulk of the uranium removed by tri-n-butyl phosphate (TBP) extraction.

10

EFFECT OF THE CONCENTRATION OF HYDROFLUORIC AND SULFURIC ACIDS IN A MIXTURE ON THE KINETICS OF NIOBIUM PENTOXIDE AND TANTALUM PENTOXIDE DISSOLUTION, I.I. Baram, *Izv. Vyssh. Ucheb. Zaved., Tsvet. Met.*, 12(4), 78, (1969).

The rate of niobium pentoxide dissolution was found dependent on hydrofluoric and sulfuric acid interaction and concentration. At a ratio of hydrofluoric acid to sulfuric acid of five, niobium pentoxide dissolution rate was maximum.

11

ELECTROLYTIC DISSOLUTION OF TANTALUM, NIOBIUM, AND OTHER REFRACTORY METALS,

A. Aladjem, *Anal. Chem.*, 41, 989 (1969).

A method for the electrolytic dissolution of niobium and its alloys is described. The dissolution is accomplished using a 5% solution of ammonium chloride in methanol. Borosilicate glass containers are used, and the metal serves as the anode and platinum serves as the cathode. An anode current of 20 to 30 mA/sq cm is recommended for providing short dissolution times. Complete dissolution is accomplished using the recommended conditions. The method can also be used for powdered samples contained in graphite or a heavily-anodized tantalum dish.

12

ANODIC DISSOLUTION STUDY OF URANIUM ALLOYS IN FUSED SALTS, J.C. Fondanaiche, *Bull. Soc. Chim. Fr.*, No. 5, 1579 (1969).

The anodic dissolution mechanism for uranium-niobium alloys was studied by analyses of the anodic sludges. During electrochemical dissolution of alloy in molten chlorides, the waste formed on the alloy corresponded to weakening of the alloy for the slow extraction of uranium.

SECTION II

Alloy Separation

Precipitation and Scavenging Techniques

- 13**
INVESTIGATIONS INTO THE ANALYTICAL CHEMISTRY OF TANTALUM, COLUMBIUM, AND THEIR MINERAL ASSOCIATES. XXV. SEPARATION OF URANIUM FROM TANTALUM, COLUMBIUM, AND TITANIUM, W.R. Schoeller and H.W. Webb, *Analyst*, 58, 143 (1933).
 Tannic acid added to a solution of ammonium oxalate and chloride containing niobium and uranium precipitates the niobium from solution. In tartaric acid hydrolysis, uranium interferes with the precipitation of niobium.
- 14**
LABORATORY DEMONSTRATION OF REDOX FEED HEAD-END TREATMENT; RUTHENIUM VOLATIZATION AND MANGANESE DIOXIDE SCAVENGING, H.G. Hicks, C.G. McCormack, and W.E. Roake, HW-22,076 (1951).
 Several parameters are discussed involving the manganese dioxide scavenging of niobium from fission product solutions containing uranium. Included are the decontamination of uranium from niobium as functions of concentration and a preparation method for manganese dioxide.
- 15**
CONTRIBUTIONS TO THE CHEMICAL ANALYSIS OF COPPER IN Zr, Si IN U-Si ALLOYS, AND Nb IN U-Nb ALLOYS, E.B. Read, *et al.*, NMI-1178 (1954).
 A gravimetric method for niobium involves separation of niobium from uranium. The alloy is first dissolved in six molar hydrochloric acid and nitric-hydrofluoric acid added. The pH is adjusted to 7.0 with 1:1 ammonium hydroxide after addition of ammonium oxalate and chloride. Finally, four percent tannic acid solution is added to precipitate the niobium, and the precipitate is washed free of uranium with a solution of ammonium oxalate and chloride.
- 16**
THE GRAVIMETRIC DETERMINATION OF Nb IN U-Nb ALLOYS, O.H. Kriege, LA-2049 (1956).
 This determination involves the dissolution of the niobium-uranium alloy (8-12% niobium) in a hydrochloric acid-hydrogen peroxide mixture followed by hydrolyzing niobium in an acid-sulfur dioxide medium. The final niobium pentoxide contained <0.5% uranium.
- 17**
EVALUATION OF THE REVERSE STRIKE HEAD-END PROCEDURE, D.G. Karraker and S.G. Parker, DP-259 (1957).
 Presented are details of laboratory investigations involving the removal of ruthenium along with niobium from nitric acid solutions containing fission products and uranium. A precipitate of manganese dioxide is employed in the separation.
- 18**
THE DETERMINATION OF NIOBIUM IN URANIUM NIOBIUM ALLOYS, G.W. Goward, T.M. Reinhold, and V.R. Wiederkehr, WAPD-543 (1958).
 This analytical method for 8 to 12% niobium-uranium alloys utilizes hydrolysis separation of the niobium from uranium. The alloy is dissolved in hydrofluoric-nitric acid and the fluoride removed by fuming sulfuric acid. Then the solution is hydrolyzed in a sulfuric acid-sulfur dioxide medium and the niobium removed as the insoluble niobic oxide. Sulfur dioxide solution is used to wash the precipitate.
- 19**
PRECIPITATION OF MANGANESE DIOXIDE, J.T. Grace, H.E. Henry, and D.G. Karraker, DP-346 (1958).
 Addition of a 3% solution of potassium permanganate to a nitric acid solution containing uranium, with niobium as a fission product, and manganese nitrate produces a precipitate which decontaminated the niobium by a factor 5.1 to 6.2. Precipitation variables were also investigated.
- 20**
REMOVAL OF SILICA FROM SOLUTIONS OF NUCLEAR FUELS, H.J. Groh, DP-293 (1958).
 A one percent aqueous solution of gelatin precipitates silica from niobium-uranium containing solutions. The silica-gelatin precipitate scavenges up to 50% of the niobium, and the uranium can be quantitatively removed from the precipitate by washing with dilute acid.
- 21**
PRECIPITATION OF ZIRCONIUM, NIOBIUM, AND RUTHENIUM FROM AQUEOUS FISSION-PRODUCT SOLUTIONS, A.S. Wilson, U.S. Patent 2,847,278 (1958).
 Niobium can be separated from a fission product solution containing uranium by treatment with manganese dioxide and digesting under the boiling point of the solution. The niobium is incorporated in the manganese dioxide.

22

THE GRAVIMETRIC DETERMINATION OF NIOBIUM IN URANIUM-NIOBIUM ALLOYS, IGO-Am/S-142 (1959).

In a sulfate solution, the bulk of the niobium from niobium-uranium alloys (0.2-0.4% niobium) can be precipitated by hydrolysis. The remaining niobium can be precipitated by tannic acid and cinchonine.

23

DECONTAMINATION OF URANIUM, J.S. Buckingham and J.L. Carroll, U.S. Patent 2,918,350 (1959).

The bulk of the niobium can be removed from uranium solution by adding sodium, potassium and ammonium thiosulfates. The precipitation is performed in 0.1 to 0.3 molar acid solutions at 55 to 70°.

24

GRAVIMETRIC PROCEDURE FOR THE QUANTITATIVE DETERMINATION OF ZIRCONIUM AND OF NIOBIUM AS PHOSPHATE, C.G. Wallace, AERE-R-5999 (1969).

The described analytical method was also applied to the gravimetric determination of niobium in the presence of uranium. The determination gave a positive bias of approximately 2% after a single phosphate precipitation and less than 1% after a double precipitation.

Ion Exchange Techniques

25

NEPTUNIUM IN RADIOACTIVE RAINWATER, Y. Yokoyama, Proc. Symposium Chem. Radioactive Rain Meteorol. Research Inst., Tokyo (1955).

Fission products in one molar nitric acid are passed through an Amberlite IR-120 column. The uranium is eluted with two molar nitric acid and the niobium with 0.5% oxalic acid.

26

ANALYTICAL APPLICATION OF THE NITRIC ACID ANION-EXCHANGE SYSTEM TO PLUTONIUM-FISSION AND PLUTONIUM-BINARY ALLOYS USED IN METALLURGICAL STUDIES, R.F. Buchanan, J.P. Faris, K.A. Orlandini, and J.P. Hughes, TID-7560 (1958).

Radioactive tracers were used to study the elution characteristics of 11 elements in nitric acid on Dowex 1-X10 anion exchange resin. In 14 molar nitric acid, 50% of the niobium was adsorbed, whereas the uranium did not adsorb.

27

SEPARATION OF LONG-LIVED RADIOACTIVE ISOTOPES FROM A MIXTURE OF FISSION PRO-

DUCTS AND CORROSION ELEMENTS, M.V. Susic and T.S. Selenic, Proc. U.N. Intern. Conf. Peaceful Uses At. Energy, 2nd., Geneva 18, 82 (1958).

Fission product mixtures were loaded onto a column of Amberlite IR-120 using 0.1 molar hydrochloric acid. The uranium and part of the niobium were eluted with 1.5 molar hydrochloric acid while the major part of the niobium was eluted with six molar hydrochloric acid.

28

QUANTITATIVE RADIOCHEMICAL ANALYSIS BY ION EXCHANGE, L.R. Bunney, N.E. Ballou, J. Pascual, and S. Foti, Anal. Chem., 31, 324 (1959).

On Dowex 2-X8 resin, the equilibrium distribution coefficient (Kd) was measured for a number of elements including niobium and uranium in various concentrations of hydrochloric acid, nitric acid, and sulfuric acid. The optimum Kd values for niobium and uranium are as follows:

	3M HCl	8M HNO ₃	Conc. H ₂ SO ₄
Uranium	~10	8	<0.1
Niobium	~0.1	Not adsorbed	1.0

29

QUANTITATIVE RADIOCHEMICAL ANALYSIS BY ION EXCHANGE, E.C. Freiling, J. Pascual, and A.A. Delucchi, Anal. Chem., 31, 330 (1959).

In low concentrations (<0.5 molar) of phosphoric acid, niobium (V) has an equilibrium distribution coefficient (Kd) of the order of 10^6 while uranium (VI) has a Kd value of 5×10^3 on Dowex 2-X8 resin.

30

QUANTITATIVE RADIOCHEMICAL ANALYSIS BY ION EXCHANGE, L. Wish, Anal. Chem., 31, 326 (1959).

Niobium (V) is reported to adsorb strongly on Dowex 2-X8 resin with a mixture of 0.1 molar hydrochloric acid and 0.3 molar hydrofluoric acid yielding an equilibrium distribution coefficient (Kd) of 275. Uranium has a Kd value of 8.3 in this system. In 12 molar hydrochloric-0.3 molar hydrofluoric acid, niobium has a Kd of 15.5 and uranium has a Kd of 830.

31

SEPARATION OF URANIUM FROM SCANDIUM, YTTRIUM, RARE-EARTH ELEMENTS, THORIUM, BERYLLIUM, MAGNESIUM, COPPER, MANGANESE, IRON, ALUMINUM AND OTHER ELEMENTS BY CATION-EXCHANGE CHROMATOGRAPHY, F.W.E. Strelow, J.S. African Chem. Inst., 16, 38 (1963).

Using a column of AG 50W-X8 resin, niobium (V) can be separated from uranium (VI) by elution with 0.5 normal sulfuric acid containing hydrogen peroxide. Uranium is retained by the resin under these conditions.

32

ANION EXCHANGE CHARACTERISTICS OF ELEMENTS IN NITRIC ACID MEDIUM, M.A. Essling, J.W. Warton, K.A. Orlandini, G. Jewett, and F. Lawless, *Anal. Chem.*, **36**, 1157 (1964).

The absorption behavior of 70 elements on Dowex 1-X10 basic anion exchanger in 1 to 10 molar nitric acid concentrations was studied. Niobium (V) has slight adsorption while uranium (VI) has a distribution coefficient of 10 in 5 to 10 molar nitric acid.

33

ANION EXCHANGE STUDY OF A NUMBER OF ELEMENTS IN NITRIC-HYDROFLUORIC ACID MIXTURES, E.A. Huff, *Anal. Chem.*, **36**, 1921 (1964).

On Dowex 1-X4, the adsorption of 19 elements was studied as a function of nitric and hydrofluoric acid concentration. In six molar nitric acid - five molar hydrofluoric acid, uranium (VI) is not adsorbed while niobium (V) has a volume distribution coefficient of 10. In lower concentrations of hydrofluoric acid, the uranium (VI) starts adsorbing on the exchanger.

34

SYNTHETIC INORGANIC ION EXCHANGER. IV. THE SEPARATION OF THE FISSION PRODUCTS AND URANIUM BY MEANS OF A STANNIC PHOSPHATE CATION EXCHANGER, Y. Inoue, S. Suzuki, and H. Goto, *Bull. Chem. Soc. Japan*, **37**, 1547 (1964).

Uranium can be quantitatively eluted by nitric acid above one molar using stannic phosphate cation exchanger. Niobium is not eluted under these conditions.

Chromatographic Techniques

35

PREPARATION OF CARRIER-FREE RADIOZIRCONIUM FROM PILE-IRRADIATED URANIUM, W.E. Roake, AECD-3201 (1951).

The adsorption capacity and the effect of pH and temperature on the adsorption of niobium and fission products on Super Filtrol are presented. The niobium is removed from irradiated uranium in a 1.5 molar nitrate solution by adsorption on Super Filtrol after a one hour heating period at 100°. The Filtrol cake is washed with 0.01 molar nitric acid to remove any supernate containing uranium, and the Filtrol can be leached with 10 molar nitric acid for 30 minutes at 100° to remove the niobium.

36

ADSORPTION OF ZIRCONIUM-NIOBIUM ON SILICA GEL, D.G. Karraker and S.G. Parker, DP-228 (1957).

Adsorption characteristics on silica gel of radioactive niobium in uranyl nitrate solutions are presented. The

adsorption of niobium was increased by increasing the temperature of solution and increasing the contact time. Aging the solution before processing decreased adsorption.

37

SEPARATION PROCESS BY ADSORPTION, W.W. Schulz, *et al.*, U.S. Patent 2,796,424 (1957).

A process to remove irradiated elements in mineral acids including niobium from uranium is presented. The niobium is adsorbed on a column of silica gel whereas the uranium is not.

38

SEPARATION OF URANIUM (VI), PLUTONIUM (VI), AND PLUTONIUM (IV) FROM ZIRCONIUM AND NIOBIUM ON A COLUMN OF SILICA GEL, D. Cvjetcanin, *JENER Rept. No. 57* (1958).

A chromatographic method for separating uranium from irradiated niobium using a silica gel column is presented. The method involves extraction of uranyl nitrate from niobium using methyl isobutyl ketone in the presence of silica gel. The separation is also functional in an aqueous system.

39

SEPARATION OF URANIUM AND PLUTONIUM FROM ZIRCONIUM, NIOBIUM, RUTHENIUM, AND CESIUM ON A COLUMN OF MANGANESE DIOXIDE, D. Cvjetcanin and N. Cvjetcanin, *JENER Ann. Rept. No. 54* (1958).

A two milliliter column of 100-140 mesh manganese dioxide was used to separate uranium from niobium. Nitric acid (0.1 molar) eluted the uranium (VI) while 99.2% of the niobium was retained on the column.

40

PAPER CHROMATOGRAPHY OF PROTACTINIUM, J. Vernois, *J. Chromatog.*, **1**, 52 (1958).

A mixture containing niobium and uranium could be separated using Whatman No. 1 paper after a 12 hour development. The solvent used consisted of butanol, hydrofluoric and hydrochloric acids, and water. The concentrations of the hydrochloric and hydrofluoric acid were varied.

41

SEPARATION OF ZIRCONIUM AND NIOBIUM FROM ACTINIDE FISSION PRODUCTS BY ADSORPTION, C.S. Lowe and W.H. McVey, U.S. Patent 2,903,333 (1959).

Niobium as a fission product can be separated from neutron-irradiated uranyl nitrate by adsorption on silicate glass wool. After adsorption of the niobium from aqueous solution at a pH of 0.5 to 2, it can be removed from the glass using five molar nitric acid.

42

SEPARATION OF FISSION PRODUCTS AND CORROSION ELEMENTS ON ALUMINA, D.S. Nemoda, *Bull. Boris Kidrich Inst. Nucl. Sci.*, 9, 95 (1959).

Long-lived radioactive isotopes and elements can be separated into fractions using an aluminum oxide chromatographic column and different eluents. Uranium is eluted using one molar sodium carbonate and niobium with one molar ammonium carbonate.

43

ZIRCONIUM AND NIOBIUM ADSORPTION BY SILICA GEL, B.P. Nikol'skii, A.M. Trofimov, and G.G. Panfilova, *Radiokhimiya*, 1, 283 (1959).

The adsorption of niobium on silicon dioxide gel from nitric acid solutions of uranium was investigated. Maximum adsorption was attained using 0.5 to 1.0 molar nitric acid and various adsorption conditions and concentrations are given.

44

FEASIBILITY REPORT ON RECOVERY OF FISSION Zr AND Nb FROM REDOX PROCESS WASTE SOLUTIONS USING SILICA GEL, CF-49-9-178 (1960).

The feasibility of separating irradiated niobium from uranium on a silica gel column is described. The process provides yields of 80 to 85% which increase with operation temperature or slower flow rates. A 1.4 molar ammonium hydroxide solution is an effective washing agent for removing uranium supernate. About 70% of the niobium was eluted from silica using 0.4 molar oxalic acid.

45

RADIOCHEMICAL STUDY OF ADSORPTION BEHAVIOR OF INORGANIC IONS ON ZIRCONIUM PHOSPHATE, SILICA GEL, AND CHARCOAL, E. Akatsu, R. Ono, K. Tsukuechi, and H. Uchiyama, *J. Nucl. Sci. Technol.*, 2, 141 (1965).

The adsorption behavior of 53 inorganic ions was studied using zirconium phosphate, silica gel, and charcoal as adsorbants. The only suitable adsorbant for niobium was silica gel. In nitric acid, the uranyl (VI) is not adsorbed whereas the niobium (V) is strongly adsorbed.

46

CHROMATOGRAPHIC SEPARATION OF SOME URANIUM-FISSION PRODUCTS AND URANIUM WITH ION-EXCHANGE PAPER, N. Getoff and H. Bildstein, *Oesterr. Chem. Zeit.*, 66, 72 (1965).

On Whatman P20 cation exchange paper, the R_f values of uranium and some fission products were determined in a variety of mineral acids. Uranium can be separated from niobium with 0.5 to 1.0% hydrofluoric acid solution.

47

SEPARATION OF Pu, Np, Zr, Nb, Mo, Tc, Te, AND U FROM FISSION PRODUCTS, I. Markl and O. Bobleter, *Z. Anal. Chem.*, 219, 160 (1966).

Fission products are dissolved in 12 molar hydrochloric acid and chromatographed on a Lewatit M 500 ion-exchange column. Several elements pass through the column and the adsorbed metals can be eluted in the order Pu, Rh, Np, Zr, Nb, Te, Mo, U, and Tc by selecting the proper eluent.

48

RADIOCHEMICAL SEPARATIONS BY ADSORPTION ON MANGANESE DIOXIDE, C. Bigliocca, F. Girardi, J. Pauly, E. Sabbioni, S. Meloni, and A. Provasoli, *Anal. Chem.*, 39, 1634 (1967).

In 0.1 molar nitric acid, niobium was completely adsorbed ($D_v > 16$) while uranium was not adsorbed ($D_v < 4$) on a column of manganese dioxide. The manganese dioxide was prepared by heating a mixture of potassium permanganate and manganese sulfate at 90° .

49

PROCESS FOR REPROCESSING USED URANIUM FUEL, French Patent 1,446,640, Filed September 8, 1965.

Uranium as the metal, carbide, or oxide, which contains niobium as a fission product, is dissolved in a mixture of ammonium nitrate and alkali metal nitrate at $>210^\circ$. The molten mixture is then passed through a column packed with either quartz filter fiber, glass powder, calcium oxalate or $\gamma\text{-Al}_2\text{O}_3$. The niobium is retained on the column and can be recovered by subsequent described steps.

Solvent Extraction Techniques

50

THE ANALYSIS OF URANIUM-NIOBIUM ALLOYS, G.W.C. Milner, AERE-C/R-852 (1952).

In this analytical procedure, the 10% niobium-uranium alloy was dissolved in hydrofluoric-nitric acid and the niobium extracted as the niobium cupferron into chloroform. When double extractions were made, greater than 99% of the niobium was extracted. The uranium extracted was nil.

51

THE ANALYSIS OF URANIUM-TANTALUM AND URANIUM-NIOBIUM ALLOYS, G.W.C. Milner and A.J. Wood, AERE-C/R-895 (1952).

This analytical method for uranium-niobium alloy (up to 25% Nb) involves the extraction of niobium fluoride into methyl ethyl ketone and drying and igniting the residue to niobium pentoxide. Greater than 99% separation of niobium from uranium was achieved.

52

THE DETERMINATION OF NIOBIUM OR TANTALUM IN URANIUM AND ZIRCONIUM-BASE ALLOYS, G.W.C. Milner, G.A. Barnett, and A.A. Smales, *Analyst*, 80, 380 (1955).

Separation of niobium from niobium-uranium alloy (5 to 50% niobium) was greater than 99.9% effective in this analytical method. Several extraction reagents were evaluated. The recommended separation involved extraction of niobium from a 10 molar hydrofluoric acid - 6 molar sulfuric acid - 2.2 molar ammonium fluoride solution using *iso* butyl methyl ketone. The niobium was recovered from the solvent layers with hydrogen peroxide and precipitated with tannic acid. Several factors were studied in the various steps of the separation procedure.

53

THE DETERMINATION OF NIOBIUM AND MOLYBDENUM IN URANIUM TERNARY ALLOYS, G.W.C. Milner, G.A. Barnett, and A. Bacon, AERE-C/R-1805 (1955).

Niobium is extracted into hexone to separate 5 to 20% niobium from niobium-molybdenum-uranium alloy. The extraction is taken from a 10 molar hydrofluoric acid-6 molar sulfuric acid - 2.2 molar ammonium fluoride solution, and the niobium is removed from the hexone with hydrogen peroxide.

54

A PROCEDURE FOR THE DETERMINATION OF RADIOACTIVITY DUE TO FISSION PRODUCT NIOBIUM, D.F.C. Morris and D. Scargill, *Anal. Chim. Acta*, 14, 57 (1956).

A solvent extraction procedure for separating fission niobium from irradiated uranium is described. Tributyl phosphate (TBP) was used as the extraction solvent.

55

EXTRACTION OF METAL COMPLEXES. XXVIII. THE DISTRIBUTION OF SOME ACTINIDES AND FISSION PRODUCTS BETWEEN TRIBUTYL PHOSPHATE (TBP) AND AQUEOUS SOLUTIONS OF HNO_3 AND $\text{Ca}(\text{NO}_3)_2$, B.H. Bernstrom and J. Rydberg, *Acta. Chem. Scand.*, 11, 1173 (1957).

Distribution ratios were determined for ten elements including uranium (VI) and niobium (V) between tributyl phosphate and nitric acid. Optimum conditions for removing uranium (VI) via TBP from niobium (V) are the use of one to three molar nitric acid, adding calcium nitrate in the aqueous phase, and employment of three extraction and stripping stages. This procedure removes 99.9% of the uranium (VI).

56

SIGNIFICANCE OF CERTAIN COMPLEXES OF RUTHENIUM, NIOBIUM, ZIRCONIUM, AND URANIUM

IN PLANT PROCESSES, P.G.M. Brown, J.M. Fletcher, C.J. Hardy, J. Kennedy, D. Scargill, A.G. Wain, and J.L. Woodhead, *Proc. U.N. Intern. Conf. Peaceful Uses At. Energy*, 2nd, Geneva, 17, 118 (1958).

Based on laboratory investigations, the solvent extraction of uranium (VI) from niobium (V) is discussed and evaluated. Several factors are presented for nitric acid systems including the use of tributyl phosphate and dibutyl carbitol as the extraction solvent.

57

SEPARATION OF URANIUM FROM ZIRCONIUM AND NIOBIUM BY SOLVENT EXTRACTION, E.E. Voiland, U.S. Patent 2,833,616 (1958).

The separation of uranium from niobium in three to seven molar nitric acid containing 0.05 to 0.2 molar phosphoric acid is described. The uranium is taken up in an organic, water - immiscible solvent such as methyl ethyl ketone while leaving the niobium in the aqueous phase.

58

ANOMALOUS EXTRACTION OF ZIRCONIUM, NIOBIUM, AND RUTHENIUM BY TRIBUTYL PHOSPHATE, R.M. Wallace and H. Pollock, DP-308 (1958).

Improvement of the tributyl phosphate extraction of uranyl nitrate from fission product solutions is presented. The cause of partial extraction of niobium (V) into the TBP phase is explained and remedies are examined.

59

SEPARATION AND SPECTROPHOTOMETRIC DETERMINATION OF MICROGRAM AMOUNTS OF NIOBIUM, G.R. Waterbury and C.E. Bricker, *Anal. Chem.*, 30, 1007 (1958).

An analytical procedure utilizing hexone (4-methyl-2-pentanone) extraction of uranium to separate microgram amounts of niobium from niobium-uranium alloy is presented. The niobium (99.9%) remains in the 6.3 sulfuric - 1.6 molar hydrofluoric acid layer.

60

THE USE OF TRIOCTYLPHOSPHINE OXIDE IN ANALYTICAL CHEMISTRY, J.C. White, *Am. Soc. Testing Materials, Spec. Tech. Publ. No. 238*, 27 (1958).

Extraction data for niobium and uranium using trioctylphosphine oxide in solutions of hydrochloric, nitric, and sulfuric acid are given. Uranium has been found to extract better using low polar solvents.

61

SEPARATION OF URANIUM FROM OTHER METALS, H.H. Hyman, U.S. Patent 2,893,822 (1959).

Uranium can be separated from an acid solution containing niobium by adding hydrazine and a water immiscible ketone, e.g., hexone. A ketazine complex is formed and has a greater extraction for uranium than the ketone alone. The niobium remains in the aqueous phase.

62

ISOLATION OF ZIRCONIUM AND NIOBIUM FROM FISSION PRODUCTS BY SOLVENT EXTRACTION WITH DILUTE TRIBUTYL PHOSPHATE (TBP), T. Ishimori, H. Umesawa, and K. Watanabe, *Nippon Genshiryoku Gakkaishi*, 1, 299 (1959).

The recovery of niobium from uranium fission products using 1% tributyl phosphate in toluene is described as good. The radiochemically pure niobium was obtained from 8 to 10 molar hydrochloric acid solutions.

63

LIQUID-LIQUID EXTRACTION OF URANIUM AND PLUTONIUM FROM ACETATE SOLUTION WITH TRIISOOCTYLAMINE, F.L. Moore, *Anal. Chem.*, 32, 1075 (1960).

Several analytical applications for the liquid-liquid extraction of uranium from niobium are given. The method utilizes triisooctylamine extraction in acetate containing solutions of hydrochloric, nitric, and sulfuric acid.

64

SOME EXPERIMENTS WITH IMPORTANT FISSION PRODUCTS, G. Rudstam, *Atompraxis*, 6, 124 (1960).

Uranium was successfully extracted in dibutyl carbitol, diethyl ether, and tributyl phosphate when a salting out agent such as ammonium, magnesium, sodium, or calcium nitrate was used. Niobium was successfully coprecipitated with ferric hydroxide.

65

SPECTROPHOTOMETRIC DETERMINATION OF NIOBIUM AND MOLYBDENUM WITH 8-QUINOLINOL IN URANIUM-BASE ALLOYS, K. Motojima and H. Hashitani, *Anal. Chem.*, 33, 48 (1961).

An analytical method for niobium determination of 5% niobium-uranium alloy is based on the extraction of niobium quinolate with chloroform from uranium masked with fluoride. The average separation was found to be greater than 98% efficient.

66

ANALYSIS OF Al, Cr, Fe, Mo, Nb, Ni, Ti, V, AND Zr-U ALLOYS. 8-QUINOLINOL, 2-METHYL-8-QUINOLINOL, AND DIMETHYLGLYOXIME EXTRACTION-PHOTOMETRIC METHOD, H. Hashitani, *Nippon Genshiryoku Gakkaishi*, 4, 287 (1962).

A satisfactory analytical method utilizing 8-quinolinol extraction of niobium (0.1-5%) from uranium is described. The following extraction parameters were studied: fluoride as a masking agent, optimum pH, and washing.

67

EXTRACTION OF PLUTONIUM AND OTHER ELEMENTS WITH N-BENZOYLPHENYLHDROXYLAMINE, M.K. Chmutova, O.M. Petrukhin, and Yu. A. Zolotov, *Zh. Anal. Khim.*, 18, 588 (1963).

The extraction of plutonium by 0.4 molar BPHA into chloroform from one to six molar nitric acid solutions was studied. Several parameters were evaluated, and niobium as a fission product was found to accompany the plutonium while the uranium remained in the aqueous phase.

68

CHEMICAL FLOWSHEET FOR TRIBUTYL PHOSPHATE EXTRACTION AT THE EUROCHEMIC PLANT, E. Detilleux, A. Redon, J. Centeno, W. Drest, and R. de Witte, *Aqueous Reprocess. Chem. Irradiated Fuels, Symp., Brussels*, 219 (1963).

A chemical process involving the tributyl phosphate extraction of niobium from uranium is presented along with the effect of temperature and acidity.

69

ANALYSIS OF VARIOUS KINDS OF URANIUM-BASE ALLOYS USING EXTRACTION PHOTOMETRIC METHOD. 8-QUINOLINOL, 2-METHYL-8-QUINOLINOL AND DIMETHYLGLYOXIME EXTRACTION-PHOTOMETRIC METHOD, H. Hashitani, *JAERI-1053* (1963).

An analytical method utilizing the 8-quinolinol extraction of niobium from uranium is described. The selection of masking agent, optimum pH range, and extract washing agent was evaluated.

70

RECOVERY OF FISSION PRODUCTS FROM WASTE SOLUTIONS BY SOLVENT EXTRACTION, D.E. Horner, K.B. Brown, D.J. Crouse, and B. Weaver, *ORNL-3518* (1963).

From an acidic uranium solution, 90% of the niobium was extracted using 0.1 molar tributyl phosphate in a petroleum solvent. The extraction time and pH are given as functions of distribution coefficients.

71

RECOVERY OF IRRADIATED URANIUM BY A TWO-STAGE DIFFERENTIAL EXTRACTION PROCEDURE, R.E. McHenry, J.C. Posey, and P.S. Baker, *ORNL-3312* (1963).

Uranium is extracted from a nitric acid solution containing niobium as a fission product by use of 30% tributyl phosphate and Amsco 125-82 petroleum distillate. A separation factor greater than 1200 was obtained between the uranium and niobium, and during production runs, greater than 99.5% efficiency was reached in recovering 15 kilograms of uranium.

72

EXTRACTION OF (95 Zr, 95 Nb) FROM OXALIC ACID SOLUTIONS BY MEANS OF TRI-ISOOCTYL AMINE, M.V. Susic and Z.B. Maksimovic, *Bull. Boris Kidrich Inst. Nucl. Sci.*, 14, 135 (1963).

The extraction of niobium from oxalic acid solutions using tri-isooctylamine in xylene was studied. Several parameters were investigated including the effect of acid and amine concentrations. The presence of uranium was also observed, and the possibility of separating the niobium from uranium using this system was considered.

73

BEHAVIOR OF SOME RADIOACTIVE ISOTOPES IN THE PROCESS OF EXTRACTION OF URANYL SULFATE WITH TRI-ALKYL AMINES, V.A. Minaer, V.I. Savel'eva, V.P. Selezner, and B.U. Gromov, *Tr. Mosk. Khim.-Tekhnol. Inst. No. 47*, 151 (1964).

The tri-alkyl amine extraction of niobium 95 in xylene was investigated to separate the niobium from uranium sulfate. Sodium fluoride solution was added to extract macroamounts of niobium. In one-tenth to two normal sulfuric acid, the distribution coefficient of niobium was about 340.

74

EXTRACTION SEPARATION OF NIOBIUM FROM ZIRCONIUM, TUNGSTEN, URANIUM, AND IRON WITH AMMONIUM TETRAMETHYLENEDITHIOCARBAMATE, I.M. Gibalo, I.P. Alimarin, and P. Davaadorzh, *Vestn. Moskov. Univ., Ser. II: Khim.*, No. 4, 73 (1965).

Niobium can be satisfactorily separated from uranium by complexing the niobium with ammonium tetramethylenedithiocarbamate in nine molar hydrochloric acid and extracting the complex into chloroform.

75

EXTRACTION OF NIOBIUM (V) BY TRI-n-BUTYL PHOSPHATE FROM STRONGLY ACID SOLUTIONS IN THE PRESENCE OF MASKING AGENTS, C. Konecny, *Radiochim. Acta*, 6, 192 (1966).

The tri-n-butyl phosphate extraction of niobium from hydrochloric and nitric acid solutions was studied in the presence of several masking agents. The separation of microamounts of niobium from uranium (VI) was also investigated, and the following chelating agents were found most convenient: EDTA, NTA, oxalic acid, citric acid, Tiron, pyrogallol-sulfonic acid, and chromotropic salt.

76

FEED ADDITIVES: AN ATTEMPT TO IMPROVE THE DECONTAMINATION OF U BY COMPLEXATION OF FISSION Zr, Nb, AND Ru IN NITRIC ACID SOLUTIONS OF SPENT REACTOR FUEL, K. Joon and P.A. Deurloo, *KR-115* (1966).

Several complexing agents were studied which would reduce niobium extraction from nitric acid solutions of uranium into the tributyl phosphate extraction phase. The following agents were found suitable: EDTA, DTPA, oxalic acid, sulfosalicylic acid, Arsenazo I, chromotropic acid, Tiron, thiourea, tartronic acid, citric acid, and barbituric acid. These reagents were evaluated under various reagent and acid concentrations as well as various temperature and contact times.

77

SEPARATION OF 95, 97 Zr AND 95, 97 Nb FROM FISSION PRODUCT-MIXTURES AND IRRADIATED URANIUM BY AN EXTRACTION METHOD USING 8-QUINOLINOL, K. Motojima, and H. Hashitani, *J. Nucl. Sci. Technol.*, 3, 326 (1966).

Niobium existing as a fission product can be separated from irradiated uranium by 8-quinolinol extraction; 95% of the niobium is extracted from a solution of pH 4.8 to 5.2 containing fluoride as a masking agent.

78

THE SEPARATION AND MICROANALYSIS OF NIOBIUM, TANTALUM, TITANIUM, VANADIUM, AND ZIRCONIUM; APPLICATION TO URANIUM COMPOUNDS, O.A. Vita, *GAT-524* (1966).

Niobium is separated from uranium by complexing with N-benzoylphenylhydroxylamine (BPHA) and extracting into chloroform from 10 molar hydrochloric acid solutions. Spectrographic analysis found that microgram quantities of niobium are effectively separated from 10 grams of uranium using this separation technique.

79

SOLVENT EXTRACTION SEPARATIONS WITH BPHA (N-BENZOYL-N-PHENYLHYDROXYLAMINE). APPLICATIONS TO THE MICROANALYSIS OF NIOBIUM AND ZIRCONIUM IN URANIUM, O.A. Vita, W.A. Levier, and E. Litteral, *Anal. Chim. Acta*, 42, 87 (1968).

Niobium is separated from uranium, in this analytical method, by extraction into BPHA-CHCl₃ from 10 molar hydrochloric acid.

80

SEPARATION AND SPECTROCHEMICAL DETERMINATION OF 0.5 TO 5 PPM NIOBIUM, TANTALUM, AND TITANIUM IN URANIUM, H.M. Burnett, J.V. Pena, C.J. Martell, and R.T. Phelps, *LA-3985*, May 1968.

Niobium in microgram amounts is separated from uranium by a solvent extraction method. Niobium is

extracted into chloroform as the N-benzoylphenyl-hydroxylamine (BPHA) complex from a 10 N-hydrochloric acid solution of the uranium.

Volatilization Techniques

81

RECOVERY OF URANIUM, THORIUM, NIOBIUM, TANTALUM, AND RARE-EARTH ELEMENTS FROM ORES, Y. Shiraishi, *Japanese Patent 5902 (1957)*.

Pegmatite ore containing 0.01% U_3O_8 is fluorinated at 400° and the fluoride distillates (UF_6 , b. 65° and NbF_5 , b. 218°) are fractionally separated. Uranium of 99.9% purity is obtained in 86.6% yield, and the niobium pentafluoride is recovered as a hydrolysis precipitate.

82

RECOVERY OF URANIUM FROM HIGHLY IRRADIATED REACTOR FUEL BY A FUSED-SALT FLUORIDE-VOLATILITY PROCESS, G.I. Cathers, W.H. Carr, R.B. Lindauer, R.P. Milford, and M.E. Whatley, *Proc. U.N. Intern. Conf. Peaceful Uses At. Energy, 2nd, Geneva, 17, 473 (1958)*.

Volatilization of uranium and niobium from two types of fluoride melts is described. The volatile fluorides are passed through a sodium fluoride trap heated to 100-400° using excess of fluorine. The uranium hexafluoride passes through the trap and the niobium remains adsorbed.

83

CHLORINATION OF EUXENITE CONCENTRATES, A.W. Henderson, S.L. May, and K.B. Higbie, *Ind. Eng. Chem., 50, 611 (1958)*.

Separation of niobium from uranium in euxenite ores is accomplished by adding carbon and sodium chloride to the ore and passing chlorine through the heated (500°) mixture. The niobium is volatilized as niobium pentachloride and the uranium is retained in the nonvolatile residue.

84

PROCESSING OF IRRADIATED URANIUM ALLOYS BY FRACTIONAL SUBLIMATION OF THE CHLORIDES, Ph. Speeckaert, *AEC-tr-4553 (1960)*.

Described is the separation of niobium from niobium-uranium alloy by treatment with dry hydrogen chloride and fractional volatilization of their chlorides. Separation was found, however, to be less than 99% effective.

85

CHLORIDE VOLATILITY PROCESSING OF NUCLEAR FUELS, T.A. Gens, *Chem. Eng. Progr., Symp. Ser., 60, 37 (1964)*.

Description of the Zircex process is presented. The uranium alloy fuels are hydrochlorinated between 200°

and 300° to remove the volatile niobium, zirconium, and aluminum chlorides. The nonvolatile residue contains uranium trichloride and oxide impurities.

86

EXTRACTION OF TANTALUM AND COLUMBIUM FROM ORES AND CONCENTRATES BY CHLORINATION, S.L. May and G.T. Engel, *RI 6635 (1965)*.

Chlorination of euxenite ores (containing approximately 10% U_2O_3 and 27% Nb_2O_5) is reviewed. The niobium can be volatilized from the euxenite at 500° using Cl_2 , CCl_4 , $SOCl_2$, $COCl_2$, or S_2Cl . Uranium is retained by either adding sodium chloride or by passing the volatile niobium pentachloride through a sodium chloride trap.

87

DETERMINATION OF MICRO QUANTITIES OF NIOBIUM IN URANIUM, S.K. Shrimal and M.S. Varde, *Anal. Chim. Acta, 33, 683 (1965)*.

This analytical method for the determination of niobium in niobium-uranium alloys employs a volatile separation scheme. As little as five part-per-million of niobium can be separated by fuming with perchloric acid using tantalum as a carrier.

88

ENGINEERING DEVELOPMENT OF FLUID-BED FLUORIDE VOLATILITY PROCESSES. PART 10. BENCH-SCALE STUDIES ON IRRADIATED, HIGHLY ENRICHED, URANIUM-ALLOY FUELS, A.A. Chilenskas, K.S. Turner, J.E. Kincinas, and G.L. Potts, *ANL-6994 (1966)*.

Hydrochlorination of uranium-Zircaloy alloy containing niobium as a fission product resulted in decontamination of uranium from niobium and other fission products by a factor of 3.5×10^5 to 1×10^8 . Fluorination of the alloy resulted in the niobium and uranium volatilizing.

89

ENGINEERING DEVELOPMENTS OF A FLUIDIZED-BED FLUORIDE VOLATILITY PROCESS. PART III. BENCH-SCALE STUDIES WITH IRRADIATED URANIUM ALLOY FUELS, A.A. Chilenskas and K.S. Turner, *Nucl. Appl., 4, 6 (1968)*.

Irradiated uranium was hydrochlorinated and fluorinated. Fission product niobium was removed in the hydrochlorination step as a volatile chloride.

90

LABORATORY INVESTIGATIONS ON DIRECT CHLORINATION VOLATILITY PROCESSING OF NUCLEAR FUELS. PART III. SEPARATION OF THORIUM AND URANIUM FROM ThO_2-UO_2 AND PROCESSING IRRADIATED FUEL MATERIALS, S.P. Sood, D.D. Sood, K. Rengan, and A.V. Hariharan, *BARC-405 (1969)*.

Chlorination of thorium oxide-uranium oxide with chlorine and carbon tetrachloride was studied under a variety of conditions to separate the two and decontaminate fission products such as niobium. Uranium chloride fractions are adsorbed onto a sodium chloride bed at 250-300°. Then the fraction was pyrohydrolyzed, oxidized, and rechlorinated at 500° to yield a niobium decontamination factor of $>10^3$ for the purified uranium.

Miscellaneous Separation Techniques

91

BEHAVIOR OF REPRESENTATIVE FISSION PRODUCTS AND Pu IN THE KAPL ELECTROREFINING PROCESS AND REVIEW OF THE STATUS OF THE PROCESS, L.W. Needrach, A.C. Glanum and G.R. Fountain, KAPL-1692 (1957).

Decontamination results of removing niobium from uranium by miltithroughput electrorefining runs are presented along with a general review of electrorefining methods.

92

DECOMPOSING COMPLEX URANIUM-RARE EARTH TANTALONILOBATES, J.R. Ruhoff, C.O. Gerfen, and G.B. Wills, U.S. Patent 2,956,857 (1960).

Processing of euxenite ore involves digestion with 50% sodium hydroxide solution at 140-170°, hydrochloric acid added to maintain a pH of one, and sulfuric acid added for a final sulfate concentration of 0.15 molar. This treatment dissolves the uranium from the insoluble niobium.

93

SEPARATION OF VARIOUS COMPONENTS FROM URANIUM BY OXIDATION-REDUCTION REACTIONS IN A LIQUID KCl-LiCl/ZINC SYSTEM, P. Chiotti and S.J.S. Parry, IS-286 (1961).

The separation of niobium and several other elements from uranium by oxidation-reduction reactions in a fused lithium, potassium chloride-zinc system was studied. The addition of $ZnCl_2$ to the system at 500° oxidizes the uranium to the salt phase leaving the niobium in the zinc phase; this sequence is reversed upon reduction with magnesium.

94

SEPARATION OF FISSION PRODUCTS FROM IRRADIATED URANIUM DIOXIDE IN FUSED NITRATE SYSTEMS, S. Yamagishi and Y. Kamemoto, *J. Nucl. Sci. Technol.*, 2, 457 (1965).

Irradiated niobium is completely removed from uranium by chromatographic separation on an aluminum oxide column. The niobium-uranium is fused in ammonium nitrate or ammonium nitrate-lithium nitrate medium at 150-180° and then the niobium is adsorbed on aluminum oxide.

95

EXTRACTION OF METALS BY FATTY ACIDS, I.M. Novak and A. Havel, *J. Inorg. Nucl. Chem.*, 29, 531 (1967).

When the degree of alkalization (mole ratio of base to metal salt) is 0.04, niobium is extracted with a distribution ratio of approximately 10 in a melt of palmitic acid and paraffin. Uranium has a distribution ratio lower by at least two orders; thus good separation of the two elements is feasible.

96

ELECTROLYTIC RECOVERY OF METALLIC URANIUM FROM U-Nb ALLOY BY MOLTEN SALT ELECTROLYSIS, K. Cho and J. Mochinaga, *Denki Kagaku*, 37, 791 (1969).

A molten-salt electrolysis method is described for recovering uranium from uranium-niobium alloys. The alloy is used as the anode, the electrolyte is an equimolar UCl_3 -KCl-NaCl mixture and electrolysis conditions consist of an atmosphere of argon and bath temperatures of 600 to 750°. Niobium decontamination factors from the alloy were about 70 to 700.

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SECTION III

Niobium Metal Preparation

Aluminum Reductant

97

THERMAL PROCESS FOR PRODUCTION OF PURE TANTALUM AND NIOBIUM COMPOUNDS, D. Gardner, U.S. Patent 2,556,912 (1951).

Niobium sulfide is made from the pentachlorides, pentafluorides, and pentoxides of niobium and then reduced to the metal with aluminum. Aluminum sulfide can be distilled off at 1550° to produce pure niobium.

98

RECOVERING REFRACTORY METALS FROM THEIR OXIDES OR DOUBLE FLUORIDES, D.F. Taylor, R.L. Baughman, and L.F. Yntema, U.S. Patent 2,905,548 (1959).

Described is a process involving the aluminum reduction of niobium pentoxide or potassium niobium oxyfluoride using copper or silver as a dispersing agent. The niobium ingot containing the copper or silver and aluminum is leached in nitric acid to leave a residue of niobium powder.

99

NIOBIUM AND TANTALUM, A. Mayer and W.D. Hubler, Canadian Patent 620,031 (1961).

Described is the thermite reduction of niobium pentoxide using aluminum as the reductant and copper oxide as a wetting agent. The copper oxide allows the reduced metal to form in a molten mass and not in the easily separable slag. The copper is removed by nitric acid leaching. Vacuum sintering of the sponge product removes almost all of the copper and aluminum.

100

PREPARATION OF REFRACTORY METALS, Imperial Metal Industries Ltd., Netherland Appl. 6,401,240 (1963).

Aluminum, calcium, cerium, magnesium, and boron reduction of niobium pentoxide is described. An alloy of aluminum and niobium resulted when niobium pentoxide and aluminum powder was heated in a water-cooled copper reactor by passing current through an imbedded aluminum wire. Electron-beam melting under 10^{-4} torr produced ductile niobium containing <50 ppm aluminum, <50 ppm oxygen, <30 ppm nitrogen, and <50 ppm carbon.

101

COLUMBIUM METAL BY THE ALUMINOTHERMIC REDUCTION OF Cb_2O_5 , H.A. Wilhelm, F.A. Schmidt, and T.G. Ellis, *J. Metals*, 18, 1303 (1966).

Niobium metal yields of at least 95% were obtained by the bomb-aluminum powder reduction of niobium pentoxide at 1000°. The major impurities of the niobium metal were two w/o aluminum and 0.8 w/o oxygen. High purity niobium metal was obtained via a vacuum inductive furnace at 2000° for eight hours. The resultant impurity concentration was 0.12 w/o aluminum and <0.01 w/o oxygen. Electron beam melting reduced the aluminum concentration to <0.002 w/o and reduced other impurities to very low concentrations.

102

HIGH-PURITY NIOBIUM THROUGH ALUMINOTHERMIC REDUCTION OF NIOBIUM PENTOXIDE, C.K. Gupta and P.K. Jena, BARC-328 (1968).

Niobium pentoxide was reduced in a magnesium oxide lined steel bomb with aluminum. A small amount of sulfur and calcium was used to help initiate the reaction at 450°. The 88% pure niobium produced was brittle and contained 2.82% aluminum. Non-consumable electrode arc melting under argon and electron-beam refining removed the aluminum and other impurities and gave easily workable metal with a diamond pyramid hardness of 100.

Calcium Reductant

103

REDUCTION OF OXIDES OF METALS OF HIGH MELTING POINTS WITH CALCIUM HYDRIDE, G.A. Meerson, G.A. Kats, and A.V. Khokhlova, *J. Applied Chem.*, (U.S.S.R.), 13, 1770 (1940).

Niobium pentoxide is reacted with calcium hydride in a hydrogen atmosphere at 950 to 1075° to produce a crystalline powder. Residual calcium oxide is removed by hydrochloric acid leaching and the final metal contains 1.0 to 1.4% hydrogen.

104

REDUCING Ta AND Nb OXIDES, D. Gardner, U.S. Patent 2,516,863 (1950).

Niobium hydride is prepared by heating calcium hydride with niobium pentoxide at 675°. The niobium hydride can then be decomposed at 1850° to yield niobium metal. A direct aluminothermic reduction of niobium pentoxide and the decomposition of niobium silicate are also claimed.

105

REDUCTION OF NIOBIUM COMPOUNDS BY METAL REDUCTANTS, W.E. Dennis and A.F. Adamson, RDB(C) /TN-92 (1954).

Producing ductile niobium was achieved when calcium was used to reduce pure niobium pentoxide or niobium trichloride. Magnesium was also studied as a reductant on a variety of sources of niobium pentoxide and pentachloride. The oxygen content of the final niobium powder was less when calcium was used as the reductant.

106

METAL POWDERS AND METAL HYDRIDES, P.P. Alexander and R.C. Wade, U.S. Patent 2,753,255 (1956).

Niobium pentachloride is reduced to metal powder with calcium hydride at 100 to 140° in an inert atmosphere. The calcium hydride is prepared from the reaction of calcium chloride and sodium hydride.

107

THE PREPARATION OF NIOBIUM METAL BY THE OXIDE REDUCTION ROUTE, N.R. Williams, J.H. Buddery, and J.D. Reeve, AERE-M/M-97 (1957).

Niobium pentoxide reduction to the metal using calcium was found inferior to the calcium reduction of $\text{NbO}_{1.4}$. The lower oxide was prepared by reducing K_2NbOF_5 with sodium. Niobium powder (98% purity) was produced by reducing $\text{NbO}_{1.4}$ with calcium, and vacuum treatment at 2200° yielded a soft, ductile metal.

108

PRODUCTION OF DUCTILE VANADIUM AND NIOBIUM FROM THEIR OXIDES, M. Joly, *Proc. U.N. Intern. Conf. Peaceful Uses At. Energy, 2nd, Geneva, 4, 309 (1958).*

The reduction of niobium pentoxide with calcium was studied. The product was either niobium sponge or metal. The sponge was hard to recover, whereas the metal was hazardous to prepare. The reduction of niobium carbide with carbon was found more practical than the reduction of niobium pentoxide with calcium.

109

SOME DEVELOPMENTS IN THE EXTRACTIVE METALLURGY OF COLUMBIUM (NIOBIUM), M.L. Wright and F.E. Block, A/Conf. 15/P/697, *Proc. U.N. Intern. Conf. Peaceful Uses At. Energy, 2nd Geneva (1958).*

Niobium metal was prepared directly by the bomb reduction of niobium pentoxide using calcium. Niobium sponge was prepared by reacting liquid magnesium and gaseous niobium pentachloride in an inert atmosphere. Both reduction products were electron-beam melted to remove oxygen and yield soft, ductile niobium.

110

METHOD FOR PRODUCING THE REFRACTORY METALS HAFNIUM, TITANIUM, VANADIUM, SILICON, ZIRCONIUM, THORIUM, COLUMBIUM, AND CHROMIUM, K.F. Griffiths, U.S. Patent 3,085,871 and U.S. Patent 3,085,872 (1963).

Liquid phase production of niobium metal is described as well as the apparatus used. Calcium and magnesium were found preferable reductants for the niobium halides although the alkali metals are included as suitable reductants in this claim.

111

REDUCTION OF TITANIUM AND NIOBIUM OXIDES BY CALCIUM CARBIDE AND CYANAMIDE, G.A. Meerson, L.M. Yakeshova, and T.A. Shvedova, *Izv. Akad. Nauk. SSSR, Otd. Tekhn. Nauk, Met. i Gorn. Delo No. 1, 69 (1963).*

This paper considers the thermodynamic and experimental aspects of reducing niobium pentoxide with calcium carbide or calcium cyanamide. The study describes the preparation of niobium metal at 1000° and its reaction mechanism.

112

CALCIOTHERMIC REDUCTION OF NIOBIUM (COLUMBIUM) PENTOXIDE, C.K. Gupta and P.K. Jena, *Trans. Met. Soc. AIME, 230, 1433 (1964).*

Several parameters on the bomb reduction of niobium pentoxide at 600° to the metal using calcium reductant and sulfur as a heat booster have been studied. The maximum yield was 82 to 84%, and the resultant metal was electron-beam melted to reduce the major impurities. Calcium impurity was reduced from 7000 to 400 ppm and sulfur was reduced from 800 to 200 ppm.

113

PREPARATION OF COLUMBIUM METAL BY CALCIUM HYDRIDE REDUCTION OF COLUMBIUM PENTOXIDE, T.K. Mukherjee, G.R. Kamat, and C.K. Gupta, *J. Metals, 22 (2), 50 (1970).*

A process is described for reducing niobium pentoxide with calcium hydride under a vacuum or in an argon atmosphere. The resultant niobium hydride is then reduced to high-purity ductile metal by either electron-beam melting or vacuum sintering.

Carbon Reductant

114

REDUCTION OF COLUMBIUM PENTOXIDE, H. Kh. Abrikosov, *Metallurg., 13, No. 4, 9 (1938).*

A comparison between hydrogen, carbon monoxide, and carbon for reducing niobium pentoxide is described. The reduction conditions are given for the most promising agent - carbon.

115

PURE COLUMBIUM, W. Balke, *Trans. Electrochem. Soc.*, 85 (1944).

A process is described for preparing pure, ductile niobium by reaction of a mixture of pure niobium carbide and oxide under vacuum and heating conditions.

116

REDUCTION OF DIFFICULTLY REDUCIBLE OXIDES, H. Schlecht and M. Jahrstorfer, U.S. Patent 2,242,759 (1947).

A process is claimed for reducing niobium oxide with carbon under an atmosphere of hydrogen at 800 to 1600°.

117

REACTIONS OF CARBON AND METAL OXIDES IN VACUUM, W.J. Kroll and A.W. Schlechten, *J. Electrochem. Soc.*, 93, 247 (1948).

A literature review and the results of an experimental study on the carbon reduction of metal oxides in a vacuum are presented. Carbon reduction of niobium oxide produced suitable results.

118

ON CERTAIN PROPERTIES OF CARBON THERMAL REDUCTION OF PENTAVALENT NIOBIUM OXIDE, P.V. Gel'd and G.P. Shveikin, *Izvest. Akad. Nauk S.S.S.R., Otdel. Tekh. Nauk, Met. Topl., No. 1*, 44 (1959).

The reduction kinetics were studied for reducing niobium pentoxide to niobium. The effects of structure in the low-temperature phase were studied, and the phase composition from 550 to 1200° was tabulated. The reduction of a niobium pentoxide and niobium carbide mixture was technically found to be the most convenient method to prepare niobium.

119

KINETICS OF NIOBIUM PENTOXIDE REDUCTION BY MEANS OF CARBON IN VACUO, P.V. Gel'd and G.P. Shveikin, *Primenen. Vakuuma v Met., Akad. Nauk S.S.S.R., Inst. Met. im A.A. Baikova*, 101 (1960).

Reaction kinetics of various carbon materials and niobium pentoxide were studied. For preparing 99.7 to 99.8% niobium containing a maximum of 0.3% carbon, 0.03% nitrogen, and 0.5% oxygen, one of the following methods was employed: (1) Coal-tar pitch reduction of niobium oxide, (2) Preliminary preparation of a niobium oxide-carbide mixture and its subsequent reduction to metal, (3) Preparation of niobium carbide and its reaction with niobium oxide. The second method is most useful since it has a low reaction temperature (1400°).

120

EFFECT OF RECRYSTALLIZATION OF NIOBIUM PENTOXIDE ON ITS REDUCTION RATE, G.P. Shveikin, P.V. Gel'd, and V.D. Lyubimov, *Izv. Vyssh. Ucheb. Zaved., Tsvetn. Met.*, 3, 120 (1960).

The kinetic impairment of hydrogen and carbon reduction of recrystallized niobium pentoxide is explained. The recrystallization process and reduction conditions are described.

121

METHOD OF PRODUCING NIOBIUM METAL, H.A. Wilhelm and E.R. Stevens, U.S. Patent 2,937,939 (1960).

Low carbon and oxygen content niobium sponge is produced by the graphite reduction of niobium pentoxide when titanium (0.05 to 0.4 atom per mole of niobium pentoxide) is added. The reduction conditions are below atmospheric pressure and above 1300°.

122

PRODUCTION OF MIXED TANTALUM AND NIOBIUM PENTOXIDES ON DECOMPOSITION OF TANTALITE-COLUMBITE BY FUSION WITH CAUSTIC POTASH, V.I. Konstantinov, *Tsvetnye Metally*, 34, No. 4, 35 (1961).

The acetylene black reduction of niobium pentoxide to niobium was studied in a two-stage process. Stage one produced a niobium oxide-carbide mixture at 1200 to 1300° which was, in stage two, pressed into rods and electrically heated to 1500° under 2.3×10^{-3} torr. The 99.4 to 99.8% niobium product contained 0.1 to 0.5% carbon and <0.1% oxygen.

123

EQUILIBRIA IN THE Nb-C-O SYSTEM, F.G. Kusenko and P.V. Gel'd, AEC-tr-4867. Translated from *Izv. Vyssh. Ucheb. Zaved. Tsvet. Met.*, No. 2, 43 (1961).

The reduction steps of reducing niobium pentoxide to metal with carbon are examined from a thermodynamic viewpoint. The reduction steps involving lower oxides of niobium are less favorable than forming carbides as intermediate steps in producing the metal.

124

THE PRODUCTION OF METALLIC NIOBIUM FROM AN OXIDE-CARBIDE MIXTURE, G.P. Shveikin and P.V. Gel'd, *Tsvetnye Metally*, 34, No. 4, 39 (1961).

The reaction kinetics of niobium pentoxide and acetylene black were studied by weight loss in a resistance furnace. The preparation of 99.4 to 99.8% niobium containing 0.1 to 0.5% carbon and <0.1% oxygen was achieved by preparing a mold of niobium pentoxide and acetylene black under vacuum for 30 minutes at 1400°, then increasing the temperature to 1750°.

125

REDUCTION OF NIOBIUM OXIDES BY CARBON, N.P. Sazhin, O.P. Kolchin, and N.V. Sumarokova, *Izv. Akad. Nauk. SSSR, Otd. Tekh. Nauk, Met. Topl.*, No. 6, 8 (1961).

The reduction of niobium pentoxide with carbon was studied between 1600 and 2000°. The preparation of lower oxides of niobium was achieved using an inert or hydrogen atmosphere, but the preparation of metal requires a high vacuum.

126

REDUCTION OF NIOBIUM PENTOXIDE BY CARBON, E.R. Stevens and H.A. Wilhelm, *IS-339* (1961).

Small-scale experiments on the direct reduction of niobium pentoxide to niobium using carbon black are described. The data indicate that reduction at 1800° under one micron followed by increasing the temperature to 2000° via arc melting could eliminate residual oxygen. Thus, on a larger scale, intermediate steps would be eliminated during usual industrial production.

127

ON THE PRODUCTION PROCESS OF CRUDE NIOBIUM BY ARC REDUCTION, H. Kimura and Y. Sasaki, *Nippon Kinzoku Gakkaishi*, 26, 699 (1962).

Crude niobium metal (99% niobium) was produced from reducing niobium carbide and oxide in an arc-furnace. The effect of remelting, pressure, and temperature on the following reaction was studied:

128

ON THE VACUUM REDUCTION OF NIOBIUM PENTOXIDE WITH NIOBIUM CARBIDE, H. Kimura and Y. Sasaki, *Nippon Kinzoku Gakkaishi*, 26, 695 (1962).

The arc-melting of niobium pentoxide/niobium carbide mixtures in ratios of 33/67 to 45/55 was studied and 97% niobium was produced above 1700°. The crude niobium was vacuum-melted above 2150° at 10⁻⁵ torr pressure to produce 99.7% niobium with a Vickers hardness <200. The removal of carbon was found to be dependent on oxygen content and temperature.

129

REDUCTION OF OXIDES OF NIOBIUM OR TANTALUM TO METAL WITH CARBON, J.H. Downing, N.B. Colton, and C.G. Chadwick, U.S. Patent 3,114,629 (1963).

A process is described where, for example, 2.42 lb. of 99.8% niobium pentoxide was reduced with 0.53 lb. graphite after heating to 2065° under 0.6 micron of argon. The 99.76% niobium, in 99.8% yield, contained 0.05% carbon, 0.14% oxygen, 0.001% hydrogen, and 0.05% nitrogen. Addition of small amounts of either barium oxide, calcium oxide, strontium oxide, or aluminum oxide in the charge increased

the rate of carbon and oxygen removal, but an excess may cause an explosion.

130

ON THE VACUUM REDUCTION PROCESS FOR THE PRODUCTION OF NIOBIUM METAL, H. Kimura and Y. Sasaki, *Trans. Natl. Res. Inst. Metals*, 5, 213 (1963).

Niobium carbide reduction of niobium pentoxide was studied. Niobium metal (96 to 97%) was obtained easily using this vacuum process at 1700°. Impurities such as carbon, oxygen, and nitrogen were removed by vacuum refining at 2000° and pressure of 10⁻³ torr for one-half hour. The purified niobium was 99.6 to 99.7%.

131

ON THE STUDY OF ELECTRIC ARC REDUCTION PROCESS FOR MAKING RAW METALLIC NIOBIUM, H. Kimura and Y. Sasaki, *Kinzoku Zairyo Gijutsu Kenkyusho Kenkyu Hokoku*, 6, 112 (1963).

Crude niobium (99%) was produced using an electric arc furnace from a mixture of niobium pentoxide and niobium carbide. The optimum niobium pentoxide/niobium carbide ratio was found to be 35/65.

132

REDUCTION OF NIOBIUM OXIDES BY CARBON, V. Caslavská and A. Blazek, *Sb. Práci Vyzkum. Ustavu ZDHE*, 6, 90 (1965).

Reduction of niobium pentoxide by carbon was investigated as a function of reductant concentration, particle size, temperature, and pressure. The reaction rate was found to increase with temperature increase, lower vacuum, finer homogeneous mixture, and increased component contact.

133

KINETICS OF REDUCTION OF LOW-OXIDE NIOBIUM BY CARBON, V.D. Lyubimov, P.V. Gel'd, G.P. Schweikin, and S.I. Alyamovskii, *Zh. Prikl. Khim.*, 38, 2174 (1965).

An investigation of the kinetic characteristics involved in reducing niobium dioxide and niobium oxide to metal is presented. Various carbon materials and briquetting compositions were tried under various temperatures and pressures. The degree of reduction and resultant composition of the products are also presented.

134

NIOBIUM OXIDES REDUCTION BY CARBON, V. Caslavská and A. Blazek, *Proc. Conf. Appl. Phys.-Chem. Methods Chem. Anal.*, 3, 387 (1966).

Niobium oxides were reduced by carbon and niobium carbide on a thermobalance at 1200 to 1700° and 10⁻⁵ to 10⁻⁶ torr. X-ray techniques were used to determine the reaction components. Absorption-catalytic phenomena predominate in the initial stage of the reduction process, but later diffusion processes

take precedence. The rate was found to be dependent on temperature, pressure, and grain of the reactants.

135

SEMI-INDUSTRIAL PREPARATION OF HIGH-PURITY NIOBIUM AND ITS ANALYTICAL CONTROL, F. Joly and J. Saintcriq, *Ann. Mines*, 827 (1966).

A pilot-plant process is described for producing 99.98% pure niobium in 50 lb. lots. Niobium pentachloride is distilled to remove impurities and is hydrolyzed to niobium pentoxide. Niobium pentoxide is reduced with carbon and the product purified by repeated electron-beam melting.

136

OBTAINING PURE METALS BY CARBOTHERMIC REDUCTION, J. Coursier and M. Moutach, French Patent 1,518,560 (1968).

The reduction of niobium pentoxide is accomplished by use of carbon black and conditions of high vacuum and temperatures of 2150°. Preparation of metal powders via a hydriding step is also described.

137

NIOBIUM AND TANTALUM OF HIGH PURITY, A. Restelli, German Patent 1,928,149 (1969).

A process for producing high-purity niobium is described. The process consists of reducing niobium oxide with carbon at <2000° to yield a controlled amount of carbon. The resultant carbon is then used to remove oxygen impurities in a final melting step.

Hydrogen Reductant

138

REDUCTION OF DIFFICULTLY REDUCIBLE METAL OXIDES WITH HYDROGEN, H.V. Wartenberg, J. Broy, and R. Reinicke, *Z. Elektrochem.*, 29, 214 (1923).

Description of a tungsten furnace for reducing niobium pentoxide to the metal with hydrogen is given. The reduction conditions are 2500° and five atmospheres of pressure.

139

METALLIC NIOBIUM AND TANTALUM FROM THEIR CHLORIDES, H. Schafer and H. Speidel, German Patent 903,034 (1954).

Reduction of niobium pentachloride with hydrogen produces niobium tetrachloride above 300° and produces metal at 800 to 1000°. The residual chlorine is removed from the metal by heating to 1400°.

140

NIOBIUM POWDER, F. von Bichowsky, U.S. Patent 2,761,776 (1956).

Niobium pentoxide was reduced with hydrogen at 1150° in the presence of nickel oxide to produce niobium sponge. The sponge was heated to 50° with a carbon monoxide purge to remove the nickel and leave niobium powder.

141

PURE NIOBIUM, TANTALUM, TUNGSTEN, VANADIUM OR RHENIUM, W.A. Jenkins and H.W. Jacobson, German Patent 1,111,835 (1956).

Niobium halides are reduced in a silicon dioxide reactor with a stream of hydrogen containing niobium metal particles. The reaction temperature is 800° and a 20-200 mole excess of hydrogen per mole of halide is required for continuous reduction.

142

EXTRACTION OF PURE NIOBIUM BY A CHLORINATION PROCESS, A.B. McIntosh and J.S. Broadley, *Extr. Refining Rarer Metals, Proc. Symposium, London*, 272 (1957).

A purification process is described where niobium pentachloride is reduced with hydrogen first to niobium trichloride at 500° and then to the metal powder at temperatures between 600 and 1000°.

143

REDUCING NIOBIUM OXIDES TO THE METALLIC STATE, F. von Bichowsky, U.S. Patent 2,861,882 (1958).

An 11.5 gram niobium pentoxide mixture containing 0.7 grams sodium carbonate and 0.8 grams calcium carbonate is reduced with hydrogen at 1050° in a nickel pipe. The nickel acts as a catalyst and must be present. The niobium powder is leached free of calcium and sodium.

144

REDUCTION OF REFRACTORY-METAL CHLORIDES TO METAL WITH HYDROGEN, R.B. Eaton and H.W. Jacobson, U.S. Patent 3,012,876 (1960).

Niobium pentachloride vapors and hydrogen are passed through a fluidized bed of either sodium chloride or barium chloride at 650 to 950°. The niobium metal is deposited on the salt particles and leached to yield 97.2% niobium. The product is arc-melted to produce metal of high ductility, a Brinell hardness of 95, and a chloride content of 0.05%.

145

NIOBIUM AND RELATED METALS BY HYDROGEN REDUCTION OF THEIR HALIDES, E.I. duPont de Nemours & Co., British Patent 863,310 (1961).

Niobium pentachloride was reduced with hydrogen in a 9 ft. high, 18 in. diameter reactor containing 125 lb. of niobium particles. Niobium pentachloride vapor at 0.35 mole/min. was charged through the 800° bed with 120 moles hydrogen/mole of niobium pentachloride. The coarser niobium particles were intermittently removed for a 90% yield.

146

FLUIDIZED-BED PROCESS FOR PRODUCTION OF Nb, J.H. Oxley, G.H. Kesler, and I.E. Campbell, *Chem. Eng. Progr., Symp. Ser.*, 58, 60 (1962).

Description of a fluid bed process to reduce niobium pentachloride with hydrogen at approximately 800° is given. Using an excess hydrogen ratio of 90:1, essentially complete conversion of niobium pentachloride was obtained in a two-inch diameter reactor. Bed height, hydrogen pretreatment, and particle size had no significant effect on reduction.

147

NIOBIUM, TANTALUM, AND THEIR HYDRIDES, F.A. Schaufelberger and W. Scheller, German Patent 1,130,186 (1962).

Niobium pentachloride is reduced with hydrogen in a reactor and the metal is deposited on a hot surface (>700°). The metal is cooled under hydrogen to form the hydride which in turn flakes off. The hydride is heated and cooled in an inert atmosphere to produce the metal.

148

ULTRAFINE METAL POWDERS, D.R. Brutvan, R.L. Ripley, and H.V. Seklemian, British Patent 919,954 (1963), and British Patent 950,148 (1964).

A reactor is described to reduce niobium pentachloride with hydrogen. The reaction conditions are given.

149

ULTRAFINE PARTICLES OF TUNGSTEN AND RELATED METALS, R.L. Ripley, U.S. Patent 3,104,165 (1963).

The description and operation of a reactor to continuously prepare niobium powder of <0.1 micron particle size by the reaction of hydrogen and a niobium halide is presented.

150

NIOBIUM AND TANTALUM, W. Scheller and J. Renard, German Patent 1,144,489 (1963).

Gaseous niobium pentachloride reduced above 650° with hydrogen in the presence of alkali metal chlorides or fluorides produces crystalline niobium in and over the melt.

151

PREPARATION OF NIOBIUM BY REDUCTION OF NbCl₅ BY HYDROGEN, L.A. Niel'son, Ya. M. Polyakov,

and A.N. Krestovnikov, *Zh. Prikl. Khim.*, 37, 669 (1964).

The hydrogen reduction of niobium pentachloride onto a niobium band was studied as functions of niobium pentachloride concentration and temperature. The rate of niobium precipitation onto the 0.2 × 5 × 100 mm band at 820 to 1600° was 0.7 to 1.5 g/cm²/hr when one mole of niobium pentachloride-water mixture and 0.1 to 0.2 mole of niobium pentachloride were heated to 1000 to 1300°. The yield was 1.5 to 3.2 g Nb/mole of mixture and 15 to 30% conversion of NbCl₅ to the metal.

152

COARSE REFRACTORY-METAL POWDERS FROM THE GASEOUS REDUCTION OF THE METAL CHLORIDE VAPORS, N.S. Nichols, U.S. Patent 3,177,067 (1965).

A two-chamber reactor is described to prepare pure niobium powder by reacting niobium pentachloride vapor with hydrogen or zinc vapor. A niobium pentachloride to hydrogen mole ratio of 20-200 to 1 and temperatures of 700 to 1200° should be used.

153

HYDROGEN REDUCTION OF NIOBIUM PENTACHLORIDE, Y. Saeki and T. Suzuki, *Denki Kagaku*, 33, (2), 159 (1965).

The reaction conditions and products were studied during the gas phase reduction of niobium pentachloride with hydrogen. The niobium yield was maximum at 1200° when the hydrogen flow was 350 cc/min. and niobium pentachloride flow was 10 cc/min.

154

PROCESS FOR THE PRODUCTION OF TANTALUM OR NIOBIUM IN A HYDROGEN PLASMA JET, W. Scheller, C. Beguin, and K. Schuett, U.S. Patent 3,211,548 (1965).

A process for reducing niobium pentachloride in a hydrogen jet reactor at 2000 to 5000° is described. Dispersed material or niobium particles in the reactor are coated with the reduced metal during the operation.

155

REDUCING REFRACTORY-METAL CHLORIDE VAPOR TO METAL WITH HYDROGEN, R.L. McBrayer, U.S. Patent 3,282,581 (1966).

Description and operation details are given for an apparatus which reduces volatilized niobium pentachloride onto powdered niobium metal using heated hydrogen. In the 20 × 6-inch-diameter reactor, 5.5 kg of pure niobium powder was agitated at 800° while niobium pentachloride at 275° and 9.4 g/min. and hydrogen at 0.9 ft³/min. was added. The niobium increased in weight by 792 grams in 4.5 hours, which represents 91% recovery of the niobium pentachloride.

156

REDUCTION OF REFRACTORY METALS FROM THEIR VOLATILE HALIDES WITH HYDROGEN, R.L. McBrayer, U.S. Patent 3,245,673 (1966).

An agitated-bed reactor to reduce niobium pentoxide vapor with hydrogen is described. Using >300% excess of hydrogen, 90% recovery of niobium was achieved.

157

REDUCTION OF NIOBIUM PENTACHLORIDE BY HYDROGEN, B.N. Babich, G.V. Kurganov, and Yu. D. Stroganov, *Izv. Akad. Nauk SSSR, Metal (1)*, 226 (1967).

The reaction conditions and reactor design are given for reduction of niobium pentachloride to metal using hydrogen. The reaction temperature range was 925 to 1265° and the reaction rate was found to increase with increasing niobium pentachloride vapor pressures between approximately 0 and 30 torr.

158

STUDIES OF NIOBIUM PENTACHLORIDE REDUCTION BY HYDROGEN, V.P. Elyutin, B.S. Lysov, and G.I. Pepekina, *Izv. Vyssh. Ucheb. Zaved., Tsvet. Met.*, No. 3, 67 (1967).

The rate of niobium pentachloride reduction on metal using hydrogen was found to increase with temperature. Maximum metal precipitation occurred at 1470°. Suggested reducing conditions are given.

159

REDUCTION OF METALLIC HALIDES, A. van den Steen, French Patent 1,471,183 (1967).

A process and reactor for reducing niobium halide with hydrogen are described. A 99.7% yield of niobium was obtained from reducing 0.348 gram of niobium pentachloride, but the 50 grams of niobium metal obtained contained 6.74% chlorine.

160

PROCESS AND APPARATUS FOR DEPOSITING REFRACTORY METALS, Matsushita Electronics Corp., French Patent 1,527,560 (1968).

The method and apparatus for the vapor deposition and reduction of niobium are described. Vaporized niobium pentachloride is reduced in an atmosphere of hydrogen and subsequently deposited on a substrate of ceramic, metal, or quartz heated to 350-700°.

161

DENDRITIC GROWTH OF Mo, Nb, AND Ta ON DEPOSITION FROM THE GAS PHASE, P. Koss and K. Rotter, SGAE-M-22 (1968), From CONF-680603-5.

The hydrogen reduction of niobium pentachloride in the gas phase to metallic niobium was investigated. The reduction temperature was above 500°. The deposition rate dependence and growth form on the deposition parameters were studied.

Magnesium Reductant

162

REFRACTORY METALS BY HALIDE REDUCTION, C.H. Winter, Jr. and E.L. Anderson, U.S. Patent 2,744,006 (1956).

A reactor is described where niobium sponge is continuously produced by the reaction of niobium halide and a rod of magnesium at 750 to 1400°.

163

REDUCING METAL CHLORIDES INDIRECTLY WITH CALCIUM CARBIDE, H.D. Erasmus, U.S. Patent 2,814,561 (1957).

Niobium sponge is prepared by the reduction of the pentachloride with magnesium or sodium vapor. A description of the reactors for both reduction and producing the reducing vapor by reacting calcium carbide with magnesium or chloride is given.

164

METALS FROM HALIDES, G.R. Findlay, U.S. Patent 2,828,199 (1958).

The claim involves producing niobium metal by the reduction of its halides with either lithium, sodium, potassium, magnesium, or calcium above the melting point of both niobium and the vapor temperature of the halide.

165

REFRACTORY METALS, R.B. Eaton, U.S. Patent 2,891,857 (1959).

Reduced chlorides of niobium are produced by reacting niobium pentachloride and hydrogen in the presence of a sodium chloride-potassium chloride mixture at 850°. The lower niobium chloride in the salt mixture is reduced to metal sponge with magnesium or sodium at 700-1100°. The sponge is crushed, leached with six molar hydrochloric acid, and vacuum refined at 1300° for four hours to produce 99.96% niobium.

166

REFRACTORY METALS, ESPECIALLY TITANIUM, R.L. Yamartino, U.S. Patent 2,915,383 (1959).

A molten-salt reactor is described in which a niobium halide is reduced with magnesium, sodium, lithium, potassium or calcium to produce the crystallized form of the metal.

167

REFRACTORY-METAL-POWDER PRODUCTION BY VAPOR-PHASE REDUCTION OF CHLORIDES, C.H. Winter, Jr., U.S. Patent 2,997,385 (1961).

A baffled reactor is described for producing niobium powder by reducing purified niobium pentachloride with either sodium or magnesium vapor at 750 to 1200°.

168

THE AFFINITY OF NIOBIUM TO OXYGEN, G.A. Meerson and T. Segorceanu, *At. Energ. (USSR)*, 13, 597 (1962).

Niobium powder containing 5 w/o oxygen was prepared by reducing niobium pentoxide with powdered magnesium. This material was heated to 1000° for six hours with powdered magnesium or calcium shavings to remove oxygen. The resultant oxygen content was 0.2 w/o using magnesium and 0.1 to 0.2 w/o using calcium. The reduction mechanism was also studied.

169

NIOBIUM AND TANTALUM, W. Scheller and M. Blumer, German Patent 1,139,982 (1962).

The patent describes the preparation of niobium from the reduction of KNbCl_6 using magnesium. The resultant metal is separated from the reaction products using known techniques.

170

POWDERED METALS, A. Hivert and S. Tacvorian, French Addn. 79,879 (1963).

A process which was adapted from the main patent (Fr. 1,123,326) is described to prepare powdered niobium. Niobium pentoxide is layered between magnesium and magnesium oxide and heated to 1000° for 6 hours. The final niobium product can be separated from slag impurities either by mechanical or leaching methods.

171

REDUCTION OF NIOBIUM PENTOXIDE WITH MAGNESIUM, R. Baba and K. Imagawa, *Nippon Kogyo Kaishi*, 85, 351 (1969).

Niobium pentoxide previously calcined at 780° was reduced with magnesium in a closed vessel at 600 to 650°. The addition of potassium chlorate or perchlorate lowered the reaction temperature. After nonconsumable arc melting and electron-beam melting, cold reduction of over 95% was possible.

Sodium Reductant

172

METAL POWDERS, K. Kanbayashi, Japanese Patent 6613 (1957).

A process for producing niobium metal powder by reacting niobium pentachloride with a sodium emulsion in an inert atmosphere at 220° is described.

173

CHROMIUM AND NIOBIUM, W.H. Keller and I.S. Zonis, U.S. Patent 2,848,320 (1958).

A process for the reduction of niobium halides to the metal is presented. Niobium pentachloride, for example, is first dissolved in a fused salt mixture of sodium metal and sodium chloride. Then the salt mixture is reduced by addition of sodium metal. Excess of sodium and of salts are removed from the product by alcohol and water washes.

174

NIOBIUM, A.E. Williams, P.B. Eyre, and J. Jackson, British Patent 791,121 (1958).

Niobium as potassium niobium fluoride is reduced to the metal using sodium metal. The KNbF_6 is mixed with a stoichiometric excess of sodium (10%) and heated in an inert atmosphere. After the reaction, the products are heated to 1100° to boil off the excess of sodium. Soluble salts are removed by leaching the products in water, and niobium metal particles (5-66 micron range) are produced.

175

ZIRCONIUM SPONGE, R.E. Hulse, U.S. Patent 2,916,374 (1959).

The preparation of lower chlorides of niobium is accomplished by sodium metal reduction of niobium pentachloride on a bed of sodium chloride at 150 to 575°. Distillation of the reduction product at 500 to 1000° disproportionates it to sodium chloride, niobium chloride, and niobium. Niobium sponge can then be isolated by further distillation.

176

TITANIUM, R.M. Sarla and D.H. Barbour, British Patent 820,119 (1959).

A process for the molten sodium reduction of niobium chloride to niobium metal is presented.

177

NIOBIUM AND TANTALUM, R.A. Foos and R.L. Maddox, U.S. Patent 3,012,877 (1961).

A continuous process for reducing potassium niobium fluoride with molten sodium metal is described. Operating temperatures and argon pressures are 800 to 900° and 1 to 10 lb/sq. in., respectively.

178

NIOBIUM AND TANTALUM, H. Greenberg and R.A. Foos, U.S. Patent 2,994,603 (1961).

In the sodium reduction of K_2NbF_7 , addition of sodium chloride or potassium chloride can control the reaction temperature and metal particle size. For example, K_2NbF_7 is heated and stirred with sodium chloride at 350° for three hours, then cooled to 100° and sodium is added. The reaction temperature of 600° was maintained for six hours. After cooling, the mixture is crushed and leached with water. Reduction of K_2TaF_7 resulted in a 98% yield.

179

HIGH-PURITY METALLIC TANTALUM OR METALLIC NIOBIUM, T. Nakao, S. Osawa, S. Takeuchi, and M. Harada, *Japanese Patent 8*, (1961).

The metallic sodium reduction of potassium niobium fluoride to niobium metal is described. The sodium is prepurified, and the reduction takes place in an argon atmosphere at 830 to 850° . After reduction, the mixture is cooled to 300° and the excess of sodium and $KNbF_6$ is poured off.

180

LARGE FLAKY CRYSTALS OF REACTIVE REFRACTORY METALS FROM FUSED SALTS, M.L. Freedman, U.S. Patent 3,188,282 (1965).

Three arrangements of apparatus are described to reduce K_2NbF_7 with sodium. In one arrangement of the deep reactor, a molten sodium chloride-potassium chloride mixture is layered between the K_2NbF_7 at the bottom and fused sodium at the top. The sodium diffuses through the molten salt to produce crystalline niobium.

Zinc Reductant

181

REDUCING ALKALI METAL-NIOBIUM FLUORIDE WITH ZINC TO A NIOBIUM-ZINC COMPOUND, J.N. Robinson, U.S. Patent 3,157,493 (1964).

Niobium pentachloride is reacted in a molten bath of potassium and sodium chloride and sodium fluoride at 850° . Zinc metal is then added to form $NbZn_3$, and the molten salt is poured off. The $NbZn_3$ is finally leached with sulfuric acid and zinc distilled at $>900^\circ$ to leave niobium sponge. In one run, 84% of the niobium was recovered.

182

PRODUCTION OF NIOBIUM BY VAPOR PHASE REDUCTION OF NIOBIUM PENTACHLORIDE, J.A. Brothers and W.F. Pesold, U.S. Patent 3,216,822 (1965).

Niobium pentachloride is reduced with zinc vapor at 750 to 1100° to yield a mixture of niobium metal and zinc halide. The metal is condensed while the zinc halide is vaporized under an inert gas purge.

183

REDUCTION OF NIOBIUM FROM POTASSIUM NIOBIUM FLUORIDE WITH ZINC, C.A. Sutherland, Canadian Patent 711,673 (1965).

Niobium pentachloride is fed into a molten (700 to 800°) bath of potassium chloride, sodium chloride, and sodium fluoride of which there is a zinc layer on the bottom. When 40% potassium chloride, 40% sodium chloride, and 20% K_2NbF_7 is formed, an argon purge circulates the molten zinc in the salt mixture to form $NbZn_3$. The $NbZn_3$ is scraped off the graphite reactor walls, and finally the zinc is vaporized to leave pure niobium. One run gave 81% recovery of niobium.

184

PREPARATION OF METALLIC NIOBIUM AND TANTALUM BY REDUCTION OF THEIR PENTACHLORIDES USING A LARGE EXCESS OF ZINC, G. Jangg, R. Kieffer and P. Topic, *Monatsh. Chem.*, 100 (1), 379 (1969).

Niobium pentachloride is reduced under an atmosphere of argon and at 520° with an excess of zinc. The zinc forms an alloy with the niobium and the zinc chloride contained all the nonmetallic impurities. For the recovery of pure niobium, dilute hydrochloric acid leaching of the zinc was found unsatisfactory. However, pure metal containing about 1% oxygen was obtained by zinc distillation at 10^{-3} torr.

Electrolytic Reduction

185

POROUS TRANSITION METAL ANODES, B.C. Raynes, M.E. Sibert, and J.T. Burwell, Jr., U.S. Patent 2,813,069 (1957).

The preparation of niobium carbide anodes for the electrolytic production of niobium is described. The prepared anodes were used in a K_2NbF_7 - sodium chloride bath to deposit niobium at the cathode.

186

APPARATUS FOR CONTINUOUS PRODUCTION OF REFRACTORY METAL BY ELECTROLYSIS OF FUSED SALTS, G. Ervin, Jr., and H.F.G. Ueltz, U.S. Patent 2,837,478 (1958).

The described apparatus can continually produce niobium at the metallic cathode. The anode is niobium carbide, the electrolyte is molten potassium chloride and lithium chloride, and the furnace is graphite lined and heated with silicon carbide bars.

187

THE DECOMPOSITION POTENTIALS OF SOME NIOBIUM COMPOUNDS AND THE SEPARATION OF NIOBIUM BY THE ELECTROLYSIS OF MELTS, Ye. I. Gurovich, *Zhur. Neorg. Khim.*, 3, 450 (1958).

A method for the production of niobium from the electrolysis of molten salts of potassium chloride, potassium fluoride, K_2NbF_7 , and niobium pentachloride is described. The powdery niobium produced has high purity with only 0.01% niobium pentoxide present.

188

ELECTROLYTIC PRODUCTION OF TITANIUM, E. Wainer, U.S. Patent 2,833,706 (1958).

The preparation of $NbCl_2$ and $NbCl_3$ from $NbCl_4$ by reaction with lower niobium sulfides is described. The lower niobium chlorides and molten sodium chloride are electrolyzed with a carbon anode and a nickel cathode to produce niobium metal at the cathode.

189

MANUFACTURE OF SILICON, TANTALUM, AND NIOBIUM BY ELECTROLYTIC REDUCTION OF THE CHLORIDES, K. Kanbayashi, Japanese Patent 4803 (1959).

Niobium chloride is reduced with mercury to the metal by applying an electric arc from a mercury anode and a niobium cathode in an atmosphere of niobium chloride.

190

SEMICONINUOUS, FUSED HALIDE SALT BATH ELECTROLYTIC PROCESS FOR THE PRODUCTION OF HIGH-MELTING-POINT REACTIVE METALS, R.M. Sarla, British Patent 824,316 (1959).

Crystalline niobium is electrolytically produced from a molten bath containing <80% alkali metal chloride, >20% alkali metal fluoride, and niobium pentachloride operating at 750 to 800° and 4 to 7 volts.

191

ELECTROLYTIC PROCESSING, H.L. Slatin, U.S. Patent 2,899,369 (1959).

The electrolytic production of niobium is accomplished in a nickel lined crucible using a graphite anode. One part of niobium pentoxide is mixed with five parts of calcium oxide and 100 parts of fused calcium chloride and the mixture is electrolyzed at 850 to 1050° under an atmosphere of hydrogen chloride for 75 amp-hrs. The melt is cooled and acid leached to recover the metal.

192

CHLORINATION AND ELECTROLYSIS OF METAL OXIDES IN FUSED SALT BATHS, M.M. Sobel, M.J. Arnoff, and J.L. Sorkin, U.S. Patent 2,870,073 (1959)

Niobium chloride is produced by the chlorination of a molten sodium chloride bath (800°) containing niobium pentoxide with a variety of chlorinating agents. The molten mixture is then electrolyzed at 750° and 2.3 volts to produce niobium metal.

193

ELECTROLYTIC PREPARATION OF THORIUM, URANIUM, NIOBIUM, TANTALUM, VANADIUM, TUNGSTEN, MOLYBDENUM, AND CHROMIUM, G. Ervin, Jr., and H.F.G. Ueltz, German Patent 1,078,776 (1960).

An electrolyte of niobium carbide is suspended in molten alkali metal, alkaline earth, or aluminum halides at 500 to 600°. The anode is niobium carbide and the cathode niobium. The niobium carbide anode disintegrates and replenishes the niobium carbide in the molten suspension.

194

PREPARATION OF NIOBIUM AND TANTALUM BY MELT ELECTROLYSIS, K. Huber and E. Jost, German Patent 1,092,217 (1960).

Nb_3Cl_8 , sodium chloride and potassium chloride are added to a graphite crucible which acts as the anode. At the nickel cathode, very ductile niobium metal is produced when the cell is electrolyzed at 850° and 20 amp.

195

INVESTIGATION OF ELECTROLYTIC PROCESSES FOR PREPARATION OF HIGH PURITY NIOBIUM METAL, A.J. Kolk, Jr., M.E. Sibert, and M.A. Steinberg, TID-6101 (1960).

Two electrolytic processes were investigated for the production of niobium. Electrolyzing a melt of K_2NbF_7 and alkali halide mixture gave good, high purity, ductile niobium. Electro-refining impure niobium, prepared by carbon reduction of niobium pentoxide yielded niobium containing an excess of oxygen.

196

ELECTROLYTIC EXTRACTION OF REFRACTORY METALS OF GROUPS IV, V, AND VI FROM THEIR CARBIDES, H.F.G. Ueltz, U.S. Patent 2,910,021 (1960).

Niobium is electrolytically extracted from a niobium carbide anode in a molten bath of alkali metal or alkaline earth halide. The cathode is made of a suitable metal such as niobium. The fabrication of the niobium carbide anode is described.

197

METALLIC NIOBIUM AND TANTALUM BY AN ELECTROLYTIC METHOD, CIBA Ltd., British Patent 880,794 (1961).

Niobium pentachloride is bubbled through a molten mixture of sodium chloride, potassium chloride, and potassium fluoride at 750° in a graphite crucible. The molten mixture is then electrolyzed to produce niobium powder at the cathode.

198

PRODUCTION OF ELECTROLYTIC TANTALUM, NIOBIUM, AND THEIR ALLOYS, V.I. Konstantinov, S.I. Sklyarenko, and E.A. Kholobes, *Porosh. Met., Akad. Nauk Ukr. SSR* 1, No. 4, 47 (1961).

Niobium metal is electrolytically produced from molten mixtures of K_2NbOF_5 , potassium chloride, and potassium fluoride. The niobium powder produced was 97.6% pure, although the current yield was 31.7%.

199

DUCTILE NIOBIUM PRODUCTION FROM ELECTROLYTIC POWDERS, V.M. Amosov, *JPRS-16063*, Translated from *Izv. Vyssh. Uchebn. Zaved. 4: Tsvetn. Met.*, No. 4, 122 (1962).

The electrolytic reduction of niobium pentoxide in a molten bath of complex fluoride or chloride and fluorides of alkali metal is described. The preparation of ductile niobium was only possible when certain proportions of oxygen, carbon, and the admixtures were present.

200

ELECTRODEPOSITION OF NIOBIUM, CIBA Ltd., British Patent 897,138 (1962).

A fine, ductile niobium powder is produced in a molten bath containing sodium chloride, potassium chloride, and a chloride of niobium (valence of niobium <4) at 850° . The niobium is produced at the nickel cathode in a graphite crucible which acts as the anode under a current density of 1.3 amp./sq. dm.

201

ELECTROLYTIC PRODUCTION OF Ta AND Nb AND THEIR ALLOYS, V.I. Konstantinov and V.M. Amosov, *Tsvetn. Metal.*, No. 8, 72 (1962).

The electrolytic production of niobium from niobium pentoxide was found superior to the sodium thermal method. Results of experiments are given.

202

ELECTROLYTIC PROCESS FOR PRODUCING TANTALUM AND NIOBIUM, R. Monnier, J.L. Blanc, P. Grandjean, and J. Zahler, *Swiss Patent* 385,497 (1965).

An electrolytic method for producing pure niobium from a molten bath (1000°) of niobium pentoxide and cryolite is described. In the production of tantalum, yields of 50% were recovered at the cooled nickel cathode under a current density of 30 amp./sq. dm. for 1.5 hours.

203

PREPARATION OF NIOBIUM AND TANTALUM BY MELT ELECTROLYSIS, W. Rockenbauer, *Chem.-Ing.-Tech.*, 41, (4), 159 (1969).

Electrolytic production of niobium from niobium pentachloride in a potassium fluoride-potassium

chloride-sodium chloride carrier melt is described. Advantages over similar systems are also stated.

204

ON THE MELT ELECTROLYSIS OF NIOBIUM, G. Schenk, *NP-18096* (1968).

Optimum conditions are described for obtaining niobium from the electrolysis of $NbCl_5$ and K_2NbF_7 . Using a $NbCl_5$ melt gave a current yield of 77% and metal containing 410 H, 370 O, 96 N, 493 C, and <220 ppm total metallic impurities. Using K_2NbF_7 melts gave a current yield of 87% and metal containing 195 Cl and <1544 ppm total metallic impurities. Refining of the melts was accomplished under similar conditions. Two niobium melts containing 1.35% and 330 ppm C and 3092 and 1182 ppm metallic impurities were refined to 122 and 60 ppm C and <1178 and 495 ppm metallic impurities, respectively.

205

PRODUCTION OF METALLIC NIOBIUM FROM NIOBIUM CARBIDE BY MOLTEN SALT ELECTROLYSIS. III. ELECTROLYTIC REDUCTION OF POTASSIUM HEPTAFLUORO NIOBATE (V) IN A SODIUM CHLORIDE-POTASSIUM CHLORIDE EUTECTIC BATH, M. Sakawa and T. Kuroda, *Denki Kagaku Oyobi Kogyo Butsuri Kagaku*, 37 (2), 99 (1969).

The mechanism for the electrolytic reduction of K_2NbF_7 in an eutectic bath of sodium chloride-potassium chloride was studied. The results of the two step mechanism yielding niobium metal are described.

Miscellaneous Reduction Methods

206

PRODUCTION OF HIGH-MELTING METALS BY THERMAL DISSOCIATION OF THEIR COMPOUNDS, A.E. van Arkel, *Metallwirtschaft*, 13, 405 (1934).

The description of methods and results for decomposing niobium pentachloride on a heated niobium wire is given. The growth of the niobium wire is accomplished in an atmosphere of niobium pentachloride and the chlorine gas is removed in the process.

207

SILICON MONOXIDE, E. Zintl, W. Brauning, H.L. Gruber, W. Krings, and W.J. Morawietz, *Z. Anorg. Allg. Chem.*, 245, 1 (1940).

Niobium pentoxide is first reduced to niobium dioxide with silicon, then to niobium metal at 1800° . Niobium of 99% purity can be obtained by this method.

208

METALS AND ALLOYS SUCH AS TANTALUM AND COLUMBIUM, E. Zintl and W.J. Morawietz, U.S. Patent 2,301,663 (1942).

The reduction of niobium pentoxide to the metal using silicon is described. The materials are heated under reduced pressure and at a temperature where the silicon oxide will volatilize.

209

REFINING OF METALS BY THERMAL DECOMPOSITION OF THEIR HALIDES, S. Hata and M. Kuroda, Japanese Patent 10,452 (1957).

A vapor of a niobium halide is decomposed on a heated (1100-1300°) refractory such as quartz. When the refractory is cooled, the pure metal flakes off as a result of the difference in thermal expansion between the two materials.

210

REDUCTION OF REFRACTORY METALS FROM THEIR OXIDES WITH RARE-EARTH METALS, L.F. Yntema, U.S. Patent 3,232,749 (1966).

Niobium pentoxide is reduced to the metal with mixed rare-earth metals and a flux such as sodium fluoride or potassium fluoride in a closed evacuated vessel. The mixture is ignited by electrical resistance and magnesium, and the final metal-slag mixture is separated by mechanical means. The metal produced was 99.9% niobium, and 2 to 5 minutes of electron-beam melting removed all but 0.001% of the impurities.

211

THE PREPARATION OF HIGH-PURITY COLUMBIUM BY THERMAL DECOMPOSITION OF COLUMBIUM PENTACHLORIDE, L.V. McCarty and W.T. Bachmann, *Electrochem. Technol.*, 5, 299 (1967).

Niobium pentachloride was decomposed onto niobium wire at 1810-2000°. The wire grew from 2 mil to 200 mil in diameter. The grown wire contained 16 to 19 ppm oxygen, 1 ppm of hydrogen and nitrogen, and 10 to 190 ppm chloride.

212

APPARATUS FOR REDUCING CHLORIDES, H. Ishizuka, French Patent 1,531,946 (1968).

An externally heated, cylindrical apparatus is described for the reduction of niobium chlorides to metal. It consists of two zones separated by a grid; the lower for reducing agent and the upper for reduced products. The apparatus provides higher reaction efficiency and shorter reaction times.

213

MOLYBDENUM AND NIOBIUM REDUCTION FROM CHLORIDE MELTS WITH LEAD, B.D. Vasin, I.F. Nichkov, and S.P. Raspopin, *Izv. Vyssh. Ucheb. Zaved., Tsvet. Met.*, No. 1, 63 (1969).

Tests were conducted on the interaction of niobium containing chloride melts with lead. The results of the tests show that niobium is only partially reduced by the lead and that insignificant quantities of niobium pass over the lead.

214

PROCESS FOR THE PRODUCTION OF IV_a AND V_a METALS OF THE PERIODIC SYSTEM AND THEIR ALLOYS, Monte Amiata Societa Mineraria per Azioni, British Patent 1, 161,151 (1969).

A method is described for reduction of niobium halides to the metal with alkali or alkaline earth metal amalgams. It has the advantages of a lower reaction temperature, better control, and is a continuous operation. The only disadvantage concerns the final separation of the amalgam.

215

PRODUCTION OF PURE METALS AND ALLOYS, H.R. Smith and C.d' A. Hunt, British Patent 1,173,015 (1969).

The production method of reducing niobium from niobium pentoxide (92% pure-contains mostly silicon and iron oxides as impurities) is described. The oxide is reduced in a series of steps with silicon in an electron beam furnace under high vacuum. The resultant metal contained C 75, Fe 50, H 5, N 20, O <50, and Si 50 ppm.

SECTION IV

Refining of Niobium

Sintering

By increasing the particle size of niobium powders, the sintering behavior is improved.

216

REMOVAL OF ALUMINUM FROM REFRACTORY METAL POWDER DURING SINTERING, D.F. Taylor, R.L. Baughman, and L.F. Yntema, U.S. Patent 2,904,430 (1959).

If niobium is reduced to metal powder with aluminum, this sintering process can remove up to two percent of the alloyed aluminum. The sintering process consists of adding one to five percent of copper, gold, or silver and sintering at 1800-2000° under vacuum. The aluminum diffuses into the metal additive, and all the other metals are volatilized. The copper, gold, or silver-aluminum alloy is removed by a hydrochloric acid wash.

217

POWDER METALLURGY OF NIOBIUM, J. Vacek, *Neue Hutte*, 4, 493 (1959).

Niobium powder compacts were sintered three times with intermittent cold-working. Ideal sintering was achieved at 2100° for 90 minutes under less than 10^{-3} torr pressure. The following elements were removed by at least a factor of ten: aluminum, carbon, copper, hydrogen, manganese, nitrogen, oxygen, and titanium.

218

PRESSING AND HIGH-TEMPERATURE SINTERING OF TANTALUM AND NIOBIUM ELECTROLYTIC POWDERS, V.M. Amosov and V.V. Dianov, *Porosh. Met., Akad. Nauk Ukr. SSR I*, No. 3, 14 (1961).

After pressing niobium powders, they are sintered longer than four hours at 2300°. The niobium content went from 97.8 to 99.9% with sintering, and the following elements were significantly removed: iron, nickel, titanium, carbon, and oxygen.

219

MASS SPECTROMETRIC STUDIES OF THE PROCESS OF GAS EVOLUTION IN SINTERING TANTALUM AND NIOBIUM, V.M. Amosov and V.A. Lanis, *Zhur. Priklad. Khim.*, 34, 84 (1961).

Electrolytic powdered niobium was heated *in vacuo* up to 2800°. Temperature was plotted against the amount of evolved gas, and the gases were analyzed to detect impurity removal including carbon, hydrogen, nitrogen, and oxygen.

220

RELATION BETWEEN THE PROPERTIES OF TANTALUM AND NIOBIUM POWDERS AND THEIR SINTERING BEHAVIOR, J. Vacek, *Powder Met.*, 7, 156 (1961).

Electron Beam

221

ULTRA-PURE METALS PRODUCED IN ELECTRON BEAM FURNACE, T.C. DuMond, *Metal Progr.*, 74, 84 (1958).

The impurities in niobium are reduced to low levels when submitted to an electron beam furnace. A description of the equipment is given.

222

SOME EXPERIENCES WITH REACTIVE METALS MELTED IN HIGH VACUUM, D.W. Levinson, *Met. Soc. Conf.*, 2, 123 (1958).

A cleaner microstructure and improved mechanical properties result when niobium is purified by melting in an electron beam furnace under less than 10^{-4} torr of pressure.

223

ELECTRON-BOMBARDMENT MELTING – A HIGH-VACUUM TECHNIQUE APPLIED TO METALLURGY, H.R. Smith, Jr., C.d' A. Hunt, and C.W. Hanks, *Natl. Symposium Vacuum Technol. Trans.*, 146 (1958).

A process to purify niobium as the powder, sponge, or bars by electron bombardment is described. The vaporization of impurities is done under less than 5×10^{-4} torr of pressure, and longer molten times gave higher purity niobium.

224

MELTING IN THE ELECTRON-BEAM FURNACE, G. Ogiermann, *Vacuum*, 10, 454 (1960).

The Brinell hardness of niobium was reduced from 180 to 45 after melting twice under an electron beam. Operating conditions are described.

225

METAL FUSION BY ELECTRON BOMBARDMENT WITH MULTIPLE ELECTRON GUNS, H.L. Gruber and H. Stephan, *Vide*, 16, 317 (1961).

Advantages over powder metallurgy techniques are described when niobium is electronically melted at a large installation.

226

ELECTRON-BEAM MELTING, D. Peckner, *Materials in Design Eng.*, 53, 101 (1961).

The purity and properties of purification by electron-beam melting are presented for the following elements: beryllium, hafnium, vanadium, tungsten, tantalum, cobalt, and niobium. The techniques used and a diagram of the electron-beam furnace are given.

227

ELECTRON-BEAM MELTING NOW ON A PRODUCTION BASIS, M. Schussler, *Prod. Eng.*, 32, 78 (1961).

The described process yields niobium which has consistent physical and mechanical properties. The purity of the electron-beam melted metal is also improved over vacuum-arc melting. The concentration of carbon, hydrogen, and oxygen is 200 ppm compared to 500 ppm by vacuum-arc melting.

228

PROPERTIES OF ELECTRON-BEAM PURIFIED MATERIALS, A. Lawley, *Proc. Symp. Electron Beam Technol.*, 4th, Boston, Mass., 133 (1962).

The purity, workability, and mechanical properties of niobium after electron-beam refining are reviewed and compared to sintering, arc melting, and vacuum distillation techniques. In general, the electron-beam melted metal is more ductile and purer, but lower in yield, as compared to other refining methods.

229

PROPERTIES OF ELECTRON BEAM-MELTED METALS, E. Rexer, *Proc. Symp. Electron Beam Technol.*, 4th, Boston, Mass., 245 (1962).

Electron beam melting of niobium results in the metal containing less than 10 ppm of the following elements: aluminum, calcium, chromium, copper, hydrogen, magnesium, manganese, nitrogen, nickel, oxygen, lead, silicon, tin, titanium, and zinc.

230

DEOXIDATION OF REFRACTORY METALS, C. d' A. Hunt, U.S. Patent 3,091,525 (1963).

Niobium metal is deoxidized of its metal oxides by slowly feeding the ingot or billet into a high-energy electron beam. A high vacuum removes the volatilized oxides from the slowly melting metal.

231

PROPERTIES OF ELECTRON-BEAM-MELTED METALS, E. Zedler, D. Kraut, and K. Schlaubitz, *Freiberger Forschungsh.*, B76, 187 (1963).

A 15 kilowatt furnace is described for refining niobium and other metals. Preliminary results are reported.

232

METHOD AND APPARATUS FOR MELTING METALS BY INDUCTIVE HEATING AND ELECTRON BOMBARDMENT, A. Bussard, H.L. Gruber, and H. Stephan, U.S. Patent 3,226,223 (1965).

Niobium ingots of high purity were obtained by induction heating of the consumable electrode and electron bombardment of the molten metal. Details are given of the techniques and equipment used.

233

COMPOSITION OF THE GASES EVOLVED DURING ELECTRON-BEAM MELTING OF VANADIUM, NIOBIUM AND TANTALUM, E. Fromm, *Z. Metallk.*, 56, 493 (1965).

Niobium was electron-beam melted and the evolved gases analyzed chromatographically. A pretreatment of heating the metal for one hour at either 1000 or 2000° did not influence the melting stage. The major detected gases were hydrogen and carbon monoxide.

234

PURIFICATION OF REFRACTORY METALS, G. Gossee, P. Albert, L. Renucci, and P. Lehr, *Belgium Patent* 672,420 (1966).

Oxygen and carbon can be removed from niobium by adding powdered carbon, silicon, or aluminum and melting under electronic bombardment and vacuum. The improved performance of the refined metal was the only test used. No analytical data were given.

235

ELECTRON BEAM REFINING OF NIOBIUM, H. Kimura, Y. Sasaki, and S. Uehara, *Trans. Natl. Res. Inst. Metals*, 8, 15 (1966).

The removal of carbon, nitrogen, and oxygen from niobium was studied along with hardness tests on a laboratory scale electron-beam melting furnace. The carbon and oxygen are released as carbon monoxide and it was found that if an oxygen-carbon weight ratio is maintained at four to five by adding either element, maximum removal is obtained. The nitrogen is removed gradually in the process. Under optimum melting conditions, the resultant niobium had a hardness of 50 VHN and contained less than 200 ppm of gas impurities.

236

PURIFICATION OF METALS, H.R. Smith, Jr., and C. d' A. Hunt, U.S. Patent 3,288,593 (1966).

Vaporization of impurities by electron beam melting was found suitable for the purification of niobium. Pretreatment conditions and procedures are given along with product specifications.

237

ELECTRON-BEAM PURIFICATION OF METALS, H.R. Smith, Jr., U.S. Patent 3,288,594 (1966).

An electron beam furnace is used to produce pure metals and alloys. The desired metal is melted and contacted with a reactive gas under reduced pressure. The gas vaporized impurities from the molten metal, thus purifying the metal or alloy. In one run, the niobium product contained C 15, H 5, Fe 20, N 20, O <20, and Si 30 ppm.

238

200 kW ELECTRON BEAM MELTING FURNACE, Ind. Tech., No. 100, 31 (1967).

A 200 kW electron beam melting furnace for producing niobium of high purity is described.

239

APPLICATIONS OF ELECTRON BOMBARDMENT (IN REFINING AND WELDING), Met. Constr. Mec., 100, 506 (1968).

Two types of furnaces are described for electron bombardment refining of niobium. The elements almost eliminated by this technique are aluminum, antimony, iron, lead, manganese, sodium, and tin.

Zone Refining

240

FLOATING-ZONE MELTING BY ELECTRON BOMBARDMENT, A. Calverley. Colloq. Intern. Nouvelles Proprietes Phys. Chim. Metaux Tres Haute Purete, Paris, 243 (1959).

The electron bombardment apparatus is described along with purity and hardness data for niobium using the floating-zone melting technique.

241

PROCESSING MATERIALS WITH ELECTRON BOMBARDMENT, A. Lawley, Electronics, 32, 39 (1959).

Zone refining and crystal growing of niobium is accomplished by electron bombardment in a vacuum less than 10^{-4} torr. The resultant metal is free of gases, and technical details are presented.

242

ZONE MELTING OF NIOBIUM BY THE ARC METHOD, A.I. Evstyukhin, V.V. Nikishanov, and I.V. Milov, Met. Metalloved. Chistykh Metal., Sb. Nauch. Rabot, No. 3, 152 (1961).

The equipment, conditions, and techniques for zone refining technical niobium by the arc method are described. Iron, titanium, and silicon were not consolidated because of the fast zone movement; however, lead and carbon were consolidated at the ends.

Slower zone movement rates of 3-6 mm/min were found necessary for improved performance.

243

ZONE REFINING OF NIOBIUM BY THE ELECTRIC-ARC METHOD, A.I. Evstyukhin, V.V. Nikishanov, and I.V. Milov, JPRS-22336, Translated from Issled. po Zharoproch. Splavam, Akad. Nauk SSSR, Inst. Met., 9, 218 (1962).

The potential and advantages of zone refining niobium with an electric-arc furnace are described.

244

ON REDISTRIBUTION OF CARBON AND TUNGSTEN IN Nb DURING ZONAL RECRYSTALLIZATION, A.I. Evstyukhin, V.V. Nikishanov, and I.V. Milov, Izv. Akad. Nauk SSSR, Otd. Tekh. Nauk, Met. Top., No. 3, 98 (1962).

The feasibility of zonal recrystallization using an arc furnace to remove carbon and tungsten from niobium is described. The tests indicated this technique is an effective separation method.

245

HORIZONTAL ZONE MELTING OF REFRACTORY METALS BY REMOTE GUN ELECTRON BOMBARDMENT, T. Hikido, Proc. Symp. Electron Beam Technol., 4th, Boston, Mass., 276 (1962).

Electron bombardment in an ingot melting furnace is described for the zone melting of niobium. No purification data were given, and vaporization losses were lower than usual.

246

CONSOLIDATING REFRACTORY METAL POWDERS TO DENSE BARS, Westinghouse Electric Corp., British Patent 999,226 (1965).

Metals and alloys including niobium can be consolidated from their powders and purified by float-zone melting using an electron beam. Details are given of the conditions and techniques used.

247

ELECTRON BEAM FLOATING ZONE REFINING OF NIOBIUM, R.E. Reed, ORNL-P-2108 (1966).

Refining niobium by the electron beam floating zone method was evaluated using chemical analysis, mechanical properties, and resistance ratios. Except for tantalum and tungsten, metallic impurity levels in niobium were reduced by volatilization to less than one ppm using this technique.

248

MECHANICAL PROPERTIES OF ZONE REFINED COLUMBIUM AND TANTALUM, M.K. Thomas, E.S. Tankins, and J.F. Erthal, Met. Soc. Conf., 34, 451 (1966).

Comparisons between electron beam and induction heating floating zone refining techniques for niobium are described. Numerous physical tests as well as oxygen and nitrogen analyses were performed on the refined metal produced by the two techniques. The merits of the two techniques are critically compared.

249

THE PROPERTIES OF NIOBIUM CRYSTALS PRODUCED BY ZONE-MELTING, H.C. Kim and P.L. Pratt, *Mater. Res. Bull.*, 2, 323 (1967). From CONF-661126.

Several parameters were studied during the zone melting of niobium. Vacuum pressure in the furnace during melting was found to be the most important contribution to purification. With an increasing number of zone passes, the carbon and nitrogen contents of the resultant niobium were reduced.

250

EVALUATION OF NIOBIUM SOURCE MATERIALS SUITABLE FOR ELECTRON BEAM FLOAT ZONE MELTING, R.E. Reed, R.P. Tucker, and C.L. Brooks, ORNL-4246 (1968).

Three sources of niobium were purified by the electron beam float zone melting technique. Resistance ratios, chemical analysis, and flow stresses were used to characterize the resultant metal.

High Temperature-Vacuum

251

REMOVAL OF OXYGEN AND (OR) NITROGEN CONTAINED IN TITANIUM, ZIRCONIUM, OR THE LIKE, H. Ishizuka, *Japanese Patent* 15,203 (1960).

Removal of oxygen and nitrogen from niobium is accomplished by adding a reducing metal, such as barium, calcium, magnesium, or sodium and an alkali or alkaline earth halide, and melting under vacuum or in an inert atmosphere.

252

CONSUMABLE-ELECTRODE MELTING OF REACTIVE METALS, S.J. Noesen, *J. Metals*, 12, 842 (1960).

Consumable electrode melting was used to purify niobium by heating the metal above its boiling point. A plot of the anode spot temperature versus melting point of niobium is given along with the equipment description and techniques used for arc melting. For high purification, low electrode melt-off rates were recommended.

253

REFINING OF VANADIUM, NIOBIUM, AND TANTALUM, A.R. Powell and J.G. Day, *British Patent* 835,316 (1960).

Nitrogen and oxygen can be removed from niobium by adding about two percent of a pure rare earth element, preferably yttrium, cerium, or lanthanum, and melting in an arc furnace under an argon atmosphere.

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VACUUM REFINING OF NIOBIUM, R.A. Ul'yanov, E. (Ye.) P. Nechiporenko, and N.D. Tarasov, NP-tr-723, Translated from *Fiz. Metal. Metalloved.*, 2, 461 (1961).

Vacuum refining of niobium between 2300 to 2350° removes iron, lead, silicon, and dissolved and adsorbed gases. Results of the mechanical and structural properties of the compact metal are given.

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ARC MELTING IN HIGH-VACUUM ENVIRONMENTS, T.E. Butler and R.P. Morgan, *J. Metals*, 14, 200 (1962).

Niobium was purified using the consumable electrode, open-arc process. Factors investigated were control of processing rates, arc instability, and chemical purity.

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PURIFICATION OF Nb USING VACUUM TECHNIQUES, A.N. Lord, *Trans. Vacuum Met. Conf.*, 7th, New York City 1964, 269 (1965).

Purification of niobium from nitrogen and oxygen is accomplished by induction heating and high vacuum techniques. The purification procedure is described in which 400 grams of niobium, after heating at 2300° for 80 hours under a pressure of 2×10^{-8} torr, resulted in containing only one ppm of nitrogen and two ppm of oxygen.

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HIGH-VACUUM PURIFICATION OF NIOBIUM AND TANTALUM AND ITS INFLUENCE ON THE ELECTRIC RESISTIVITY, R.J. Dinter, *Z. Metallk.*, 58, 70 (1967).

The annealing of niobium in high vacuum reduced interstitially dissolved impurity atoms from 3800 or 300 to 10 at.-% ppm.

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KINETICS OF GASSING AND DEGASSING OF GROUP VA METALS, G. Hoerz, *Metall*, 22, 1201 (1968).

The kinetics of degassing niobium and adsorption of oxygen and nitrogen by niobium were investigated.

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PURIFICATION OF VANADIUM AND COLUMBIUM (NIOBIUM), F.A. Schmidt, O.N. Carlson, W.E. Krupp, and H.A. Wilhelm, *U.S. Patent* 3,425,826 (1969).

Purification of niobium metal after aluminothermic reduction of the oxide by heating below, but close to, the melting point of niobium and at 10^{-4} torr is claimed. Further purification was accomplished by electron beam vacuum melting during casting.

Iodide Refining

Miscellaneous Techniques

260

IODIDE REFINING OF NIOBIUM, D.M. Chizhikov and A.M. Grin'ko, *Zhur. Neorg. Khim.*, 4, 982 (1959).

A method for preparing pure niobium by the thermal decomposition of niobium iodide is described. The conditions are given for metal refining. A study of the iodide microstructure and microhardness indicates the removal of metallic and gaseous contaminants.

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A STUDY OF THE IODIDE NIOBIUM PROCESS, R.F. Rolsten, *J. Electrochem. Soc.*, 106, 975 (1959).

The rate of niobium deposition from niobium iodide was studied under a variety of temperatures and starting materials.

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IODIDE NIOBIUM AND SOME OF ITS PROPERTIES, V.S. Emel'yanov, A.I. Evstyukhin, and G.A. Leont'ev, *Met. Metalloved. Chistykh. Metal., Sb. Nauch. Rabot.*, No. 2, 27 (1960).

Niobium relatively free of carbon, nitrogen and oxygen was prepared by the thermal decomposition of niobium iodide using the Van Arkel method. For obtaining easily forged plastic niobium, the following optimum conditions were used: temperature of the reaction vessel and depositing were 350 and $<960^{\circ}$, respectively; temperature of branch tube was 40 to 80° ; and 2.5 to 5 grams of iodine per 100 grams of niobium chips were used. Mechanical properties are given.

263

IODIDE REFINING OF NIOBIUM, V.S. Emel'yanov, G.A. Leont'ev, and A.I. Evstyukhin, *Met. Metalloved. Chistykh. Metal., Sb. Nauch. Rabot.*, No. 3, 127 (1961).

Various parameters were studied concerning the refining of niobium by the thermal decomposition of niobium iodide. Various vapor pressures and deposition rates of niobium iodide at 350 to 700° were plotted.

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PURIFICATION OF Ta AND Nb, G.L. Miller, *Ind. Chemist*, 38, 406 (1962).

Niobium refining methods and techniques are described including sintering, arc melting, float-zone melting, electron-beam melting, and the iodide technique.

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ELECTROREFINING OF NIOBIUM, R.E. Cummings and F.R. Cattoir, *U.S. Bur. Mines, Rept. Invest.* 6506 (9) (1964).

Niobium (94%) was electrorefined to purity of 99.8%. A mixture of 55% potassium fluoride, 27% sodium fluoride, and 18% K_2NbF_7 was heated in a graphite lined pot to about 900° and 0.5 to 0.9 volts was applied to the niobium anode. Reduction of the following major impurities was accomplished: carbon from 9700 to 25, iron 9200 to 630, nitrogen 2100 to 90, oxygen 19,800 to 500, and silicon 750 ppm to none detected.

266

PRODUCTION OF METALLIC NIOBIUM FROM NIOBIUM CARBIDE BY MOLTEN SALT ELECTROLYSIS. IV. METALLIC NIOBIUM AND ITS ELECTROREFINING, M. Sakawa and T. Kuroda, *Denki Kagaku*, 36 (11), 792 (1968).

Niobium obtained by molten salt electrolysis was electrorefined in an electrolyte containing 5 w/o K_2NbF_7 and a eutectic mixture of sodium and potassium chloride. The salt was maintained at 800° and an initial current density of 0.05 amp/sq.cm. was applied. Zone refining followed the electrorefining and reduced the following impurities in the niobium: aluminum, carbon, iron, nickel, and silicon.

267

EXTRACTION OF NITROGEN, CARBON, AND OXYGEN FROM NIOBIUM BY REACTION WITH ZINC, R.A. Meussner, R.J. Goode, and B.F. Brown, *J. Less-Common Metals*, 17 (4), 411 (1969).

The removal of nitrogen, carbon, and oxygen from niobium using a zinc-rich compound of niobium through the formation of ternary phases of the η -carbide ($E9_3$) structures was studied along with the rate determining steps between 850 and 1230° .

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ORGANIZATION SYMBOLS

Symbol	Title of Organization	Symbol	Title of Organization
A	Calif. Univ., Berkeley, Radiation Lab.	IS	Ames Laboratory, Ames, Iowa.
AECD	U.S. Atomic Energy Commission De-classified Document.	JAERI	Japan Atomic Energy Research Inst., Tokyo, Japan.
AECL	Atomic Energy of Canada Ltd., Chalk River, Ontario.	JENER	Joint Establishment for Nuclear Energy Research, Kjeller, Norway.
AEC-tr	Division of Technical Information Extension, AEC.	JINR-P	Joint Institute for Nuclear Research, Laboratory of Nuclear Problems, Dubna, USSR.
AECU	Division of Technical Information Extension, AEC.	JPRS	Joint Publications Research Service, New York.
AERE	United Kingdom Atomic Energy Authority, Atomic Energy Research Establishment, Harwell, Berkshire, England.	K	Carbide and Carbon Chemicals Corp., K-25 Plant, Oak Ridge, Tenn.
ANL	Argonne National Laboratory, Argonne, Ill.	KAPL	Knolls Atomic Power Lab., Schenectady, N.Y.
BARC	Bhabha Atomic Research Center, India Atomic Energy Commission.	KR	Norway Institutt for Atomenergi, Kjeller.
BMI	Battelle Memorial Inst., Columbus, Ohio.	LA	Los Alamos Scientific Lab., New Mexico.
BNL	Brookhaven National Laboratories, Upton, N.Y.	MLM	Mount Laboratory, Miamisburg, Ohio, Operated for USAEC.
CF	University of Chicago, Metallurgical Laboratory.	NASA	National Aeronautics and Space Administration.
CF-(yr-mo)	Oak Ridge National Laboratory, Tennessee.	NP	Division of Technical Information, AEC. Non-AEC Report.
CK	University of Chicago, Metallurgical Laboratory.	ORNL	Oak Ridge National Laboratory, Tennessee.
CONF	Serial number assigned to conference cataloged by the AEC Division of Technical Information Extension.	ORNL-TM	Oak Ridge National Laboratory, Tennessee.
DP	DuPont de Nemours (E.I.) and Co., Wilmington, Del.	RDB(W)	United Kingdom Atomic Energy Authority, Industrial Group, Windscale Works, Sellafield, Cumb., England.
EURAECE	United States - Euratom Joint Research and Development Program.	RFP	Rocky Flats Plant - Operated by the Dow Chemical Co. for USAEC.
GAT	Goodyear Atomic Corp., Piketon, Ohio.	RI	Wright Patterson AF Base, Ohio.
GEAP	General Electric Co.	SGAE	Oesterrerrchische Studiengesellschaft fuer Atomenergie G. m. b. H., Seibersdorf, Institut fuer Metallurgie.
HW	General Electric Co., Hanford Atomic Products Operation, Richland, Wash.	TID	Division Technical Information Extension, AEC.
IGO	Gt. Britain, Windscale Works, Sellafield, Cumb., England, Industrial Group Report.	WAPD	Westinghouse Electric Corp., Atomic Power Division, Pittsburgh.

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