NIOBIUM AND NIOBIUM COMPOUNDS

1. Niobium

Niobium, discovered by Hatchett in 1801, was first named columbium. In 1844, Rosed thought he had found a new element associated with tantalum (see TANTALUM AND TANTALUM COMPOUNDS). He called the new element niobium, for Niobe, daughter of Tantalus of Greek mythology. In 1949, the Union of Pure and Applied Chemistry settled on the name niobium, but in the United States this metal is still known also as columbium. Sometimes called a rare metal, niobium is actually more abundant in the earth's crust than lead.

Niobium is important as an alloy addition in steels (see STEEL). This use consumes over 90% of the niobium produced. Niobium is also vital as an alloying element in superalloys for aircraft turbine engines. Other uses, mainly in aerospace applications, take advantage of its heat resistance when alloyed singly or with groups of elements such as titanium, zirconium, hafnium, or tungsten. Niobium alloyed with titanium or with tin is also important in the superconductor industry (see HIGH TEMPERATURE ALLOYS; REFRACTORIES).

1.1. Properties. Elemental niobium [7440-03-1], Nb, has a cosmic abundance of 0.9 relative to silicon $\equiv$ 106 (1), an average value of 24 ppm in the earth's crust (2), and a comparable value on the lunar surface (3). Niobium is a monoisotopic element, although a search for residual radionuclides from the formation of the solar system has established the natural abundance of ^{92}Nb [13982-37-1], having a half-life, $t_{1/2}$, of 1.7×10^8 yr, to be $1.2 \times 10^{-10}\%$ (4). In addition, minute amounts of ^{94}Nb [14681-63-1], $t_{1/2} = 2.03 \times 10^4$ yr, and ^{95}Nb [13967-76-5], $t_{1/2} = 35$ d, occur in nature; the former from neutron capture by the stable isotope, and the latter as the daughter of ^{95}Zr in the fission products of ^{235}U. Niobium-93 has a nuclear spin of 9/2 and a thermal neutron-capture cross section of $1.1 \pm 0.1 \times 10^{-28}$ m^2 (1.1 barns) which makes it of much interest to the nuclear industry (see NUCLEAR REACTORS).

Niobium, like vanadium, undergoes no phase transitions from room temperature to the melting point. It is a steel-gray, ductile, refractory metal having a higher melting point than molybdenum and a lower electron work function than tantalum, tungsten, or molybdenum. Niobium closely resembles tantalum in its properties; the former is only slightly more chemically reactive. The metal is resistant to most gases below 200°C, but is air oxidized at 350°C, developing an oxide film of increasing thickness which changes from pale yellow to blue to black at 400°C. Absorption of hydrogen at 250°C and nitrogen at 300°C occurs to form interstitial solid solutions which greatly affect the mechanical properties. Niobium is attacked by fluorine and gaseous hydrogen fluoride and is embrittled by nascent hydrogen at room temperature. It is unaffected by aqua regia and mineral acids at ordinary temperatures, except hydrofluoric acid in which it dissolves. Niobium is attacked by hot concentrated hydrochloric and sulfuric acids, dissolving at 170°C in concentrated sulfuric acid, and by hot alkali carbonates and hydroxides, which cause embrittling.

The most common oxidation state of niobium is +5, although many anhydrous compounds have been made with lower oxidation states, notably +4 and +3, and Nb^{5+} can be reduced in aqueous solution to Nb^{4+} by zinc. The aqueous chemistry primarily involves halo- and organic acid anionic complexes. Virtually no cationic chemistry exists because of the irreversible hydrolysis of the cation in dilute solutions. Metal–metal bonding is common. Extensive polymeric anions form. Niobium resembles tantalum and titanium in its chemistry, and separation from these elements is difficult. In the solid state, niobium has the same atomic radius as tantalum and essentially the same ionic radius as well, ie, $Nb^{5+} \approx Ta^{5+} = 68$ pm. This is the same size as Ti^{4+} (68 pm) and Li^{+} (69 pm). Some properties of niobium are listed in Table 1; corrosion data are presented in Table 2.

1.2. Occurrence. Niobium and tantalum usually occur together. Niobium never occurs as the metal, ie, in the free state. Sometimes it occurs as a hydroxide, silicate, or borate; most often it is combined with oxygen and another metal, forming a niobate or tantalate in which the niobium and tantalum isomorphously replace one another with little change in physical properties except density. Ore concentrations of niobium usually occur as carbonatites and are associated with tantalum in pegmatites and alluvial deposits. Principal niobium-bearing minerals can be divided into two groups, the titano- and tantalo-niobates.

Titano-niobates consist of the salts of niobic and titanic acids. The important minerals of this group are pyrochlore [12174-36-6], loparite [12173-83-0], koppite [12198-49-1], and others. Pyrochlore, the most important, is complex and of varying composition. The general formula for a typical Canadian pyrochlore is $(Na,Ca)_2(Nb,Ti)_2O_6[F,OH]$; for a typical Brazilian pyrochlore, $(Ba,Ca)_2(Nb,Ti,Ce)_2O_6[O,OH]$. The color of pyrochlore ranges from dark gray to brown to orange-brown. Pyrochlore occurs in carbonatite complexes primarily in Brazil and Canada as well as in Kenya, Uganda, Nigeria, Zaire, Norway, and the United States (14). It also occurs with calcite, dolomite, apatite, magnetite, and some silicates. The density of pyrochlore is ca 4.0–4.4 g/cm^3. The tantalum content usually is low, ca 0.1–0.3% on a metal basis.

Tantalo-niobates consist of the salts of niobic and tantalic acids. The general formula for this group is $(Fe,Mn)(Nb,Ta)_2O_6$. The minerals consist of isomorphic mixtures of the four possible salts. The compound is a columbite if niobium is predominant and a tantalite if tantalum is predominant. These minerals are brown to black and usually contain titanium, tin, tungsten, and other impurities. The density of columbite is ca 5 g/cm^3 and that of tantalite is ca 8 g/cm^3. A regular gradation exists between these limits in proportion to the tantalum content. Columbite–tantalite minerals usually are finely disseminated in granitic rocks and associated pegmatites or occur as enriched concentrations in alluvial (placer) deposits. The main sources of columbites are Nigeria, Zaire, and Malaysia, and a number of smaller occurrences in other parts of the world. Columbites frequently are associated with cassiterite [1317-45-9], SnO_2, deposits and niobium occurs in tin slags during processing. These slags from Thailand and Malaysia are processed for their niobium and tantalum content.

1.3. Extraction, Refining, and Metallurgy. The stability of niobium ores is reflected in their occurrence as the residues of advanced weathering processes. Thus, stringent conditions are required to render the niobium extractable. The process of extracting and refining niobium consists of a series of consecutive operations. Frequently, several steps are combined: the upgrading of ores by preconcentration (15); an ore-opening procedure to disrupt the niobium-containing matrix; preparation of a pure niobium compound; reduction to metallic niobium; and refining, consolidation, and fabrication of the metal.

The most straightforward process is the direct conversion, ie, reduction, of the niobium concentrate to metallic niobium. The primary method is the aluminothermic reduction of a pyrochlore and iron–iron oxide mixture (16). A single batch usually contains from 1–10 metric tons of pyrochlore together with the necessary aluminum powder, iron scrap, and/or iron oxide. Frequently small amounts of lime or fluorspar are used as fluxing agents. Sometimes a small quantity of a powerful oxidizer, eg, sodium chlorate, which provides additional reaction heat is added. A typical reactor consists of a refractory-lined steel shell. Sometimes a floor of slag from previous reduction reactions is used. Typically, a small amount of a starter mixture, eg, aluminum powder and sodium chlorate, is ignited electrically to start the reaction. In another variation, aluminum powder and barium peroxide are used, and a small water spray initiates the aluminum–peroxide combustion. In some cases, the reaction is started using a small quantity of reactants, and additional pyrochlore–aluminum–iron mixture is fed into the reaction mixture by means of a chute until the reactor vessel is filled with molten metal and slag.

In the batch reactions, the reaction time varies from 2–3 min to ca 25 min, primarily depending on the size of the reaction. At the completion of the reaction, the molten ferroniobium is at the bottom of the reactor and the slag floats on top. Most of the impurities go into the slag. Some of the more easily reduced metals go into the ferroniobium. Typical commercial-grade ferroniobium has the following wt% composition: Nb, 62–67; Fe, 28–32; Si, 1–2.5; Al, 0.5–2.0; Ti, 0.1–0.4; P, 0.05–0.15; S, 0.05–0.1; and C, 0.05–0.1.

Minor amounts of tantalum, tin, lead, bismuth, and other elements also occur in the ferroniobium. After cooling for 12–30 h, the metal is separated from the slag and crushed and sized for shipment. The recovery of niobium in the aluminothermic reaction is 87–93%. Larger reactions generally give better recoveries.

Ferroniobium also is produced in an electric furnace procedure. Essentially the same reactants are used as in the aluminothermic method. Because the electric furnace provides additional energy input, the quantity of aluminum can be substantially reduced and partly substituted by other reducing agents, eg, ferrosilicon. The total heat input can be better controlled using the electric furnace procedure. Recovery of niobium is therefore generally better than for the aluminothermic method. The metal and slag which are produced can be tapped or skimmed using standard electric furnace procedures. Generally, the production volume of ferroniobium is not sufficient to take advantage of the control and semicontinuous operation offered by the electric furnace (see FURNACES; ELECTRIC).

In addition to the standard ferroniobium, there is a lesser but significant demand for high purity niobium alloys, mainly high purity ferroniobium and nickel–niobium. These high purity alloys are used in the fabrication of nickel- and cobalt-based superalloys which are used primarily in jet engine and aerospace applications (see HIGH TEMPERATURE ALLOYS). The alloys are produced by reducing a pure niobium oxide in the presence of iron or nickel in an aluminothermic reaction using carefully controlled conditions and raw materials (17).

In some cases, the reactions are carried out in water-cooled copper reactors to avoid contamination by refractory materials. The key raw material for these alloys is a pure (99 wt%) niobium oxide, which can be produced only by chemical procedures.

Direct attack by hot 70–80 wt% hydrofluoric acid, sometimes with nitric acid (qv), is effective for processing columbites and tantalo-columbites. Yields are >90 wt%. This method, used in the first commercial separation of tantalum and niobium, is used commercially as a lead-in to solvent extraction procedures. The method is not suited to direct processing of pyrochlores because of the large alkali and alkaline-earth oxide content therein, ie, ca 30 wt%, and the corresponding high consumption of acid.

Concentrated sulfuric acid (97 wt%) at 300–400°C has been used to solubilize niobium from columbite and pyrochlore (18,19). The exothermic reaction is performed in iron or silicon–iron crucibles to yield a stable sulfato complex. The complex is filtered free of residue and is hydrolyzed by dilution with water and boiling to yield niobic acid which is removed by filtration as a white colloidal precipitate.

Fusion with caustic soda at 500–800°C in an iron crucible is an effective method for opening pyrochlores and columbites (20). The reaction mixture is flaked and leached with water to yield an insoluble niobate which can be converted to niobic acid in yields >90 wt% by washing with hydrochloric acid.

The reaction of chlorine gas with a mixture of ore and carbon at 500–1000°C yields volatile chlorides of niobium and other metals. These can be separated by fractional condensation (21–23). This method, used on columbites, is less suited to the chlorination of pyrochlore because of the formation of nonvolatile alkali and alkaline-earth chlorides which remain in the reaction zone as a residue. The chlorination of ferroniobium, however, is used commercially. The product mixture of niobium pentachloride, iron chlorides, and chlorides of other impurities is passed through a heated column of sodium chloride pellets at 400°C to remove iron and aluminum by formation of a low melting eutectic compound which drains from the bottom of the column. The niobium pentachloride passes through the column and is selectively condensed; the more volatile chlorides pass through the condenser in the off-gas. The niobium pentachloride then can be processed further.

The reaction of finely ground ores and an excess of carbon at high temperatures produces a mixture of metal carbides. The reaction of pyrochlore and carbon starts at 950°C and proceeds vigorously. After being heated to 1800–2000°C, the cooled friable mixture is acid-leached leaving an insoluble residue of carbides of niobium, tantalum, and titanium. These may be dissolved in HF or may be chlorinated or burned to oxides for further processing.

Once the niobium ore has been opened, the niobium must be separated from the tantalum and/or impurities. The classical method of doing this is the addition of an excess of potassium fluoride to hydrofluoric acid solutions of niobium ores to precipitate the complex fluorides and oxyfluorides of niobium, tantalum, and titanium. These are redissolved in dilute hydrofluoric acid, a 3 wt% HF solution containing $K_2NbOF_5{\cdot}H_2O$ [19200-74-9], $K_2TiF_6H_2O$, and K_2TaF_7 with respective solubilities at 15°C of ca 77 g/L, 12 g/L, and 5 g/L. The hydrate of ${}_2TiF_6$ is isomorphous with $K_2NbOF_5{\cdot}H_2O$, but K_2TaF_7 is not. Dipotassium tantalum heptafluoride is stable in 3 wt% HF. Its solubility increases with HF concentration and temperature, and it is 50 times more soluble at 85°C than at 15°C. The species changes to $KTaF_6$ at 45% HF. The solubility of K_2NbOF_5 is high in 3 wt% HF and increases with temperature. At 6–40 wt% HF, the occurring species are dipotassium niobium heptafluoride [36354-32-2], K_2NbF_7, and $KNbF_6$ [16919-14-5] at >45 wt% HF. Increasing the concentration of KF depresses the solubility of all the complex salts. Repeated recrystallization can produce K_2TaF_7 with less than 0.01 wt% niobium content, but the niobium remains in the mother liquor and, hence, still contains the titanium impurity. Thus, the compound's usefulness is limited in the economical production of pure niobium.

The recrystallization of complex fluoride salts has been replaced completely by solvent extraction techniques which are used extensively. Many acid–solvent combinations have been reported in the literature, eg, HF–methyl isobutyl ketone (MIBK) (24), HF–HNO_3–MIBK (25), HF–HCl–MIBK (26), and HF–H_2SO_4–MIBK (27). Commercial processes involve the use of various acids in combination with HF and either MIBK or tributyl phosphate (TBP), generally starting with an upgraded feed material. The dissolution in HF is performed in rubber or polyethylene-lined tanks to form the fluoride feed solution. The separation then may be effected by two methods: either the tantalum is extracted first by contacting a low acidity aqueous feed with the organic phase and subsequently raising the acidity of the aqueous phase and extracting the niobium with fresh organic, or both niobium and tantalum are extracted into the organic phase from a strongly acidic medium and the niobium is back-extracted form the organic with dilute acid. The metal values are back-extracted from the organic phase with a low acidity aqueous phase from which they are precipitated as oxides by addition of ammonia or as double fluorides by addition of potassium fluoride.

Another solvent extraction scheme uses the mixed anhydrous chlorides from a chlorination process as the feed (28). The chlorides, which are mostly of niobium, tantalum, and iron, are dissolved in an organic phase and are extracted with 12 N hydrochloric acid. The best separation occurs from a mixture of MIBK and diisobutyl ketone (DIBK). The tantalum transfers to the hydrochloric acid leaving the niobium and iron, the DIBK enhancing the separation factor in the organic phase. Niobium and iron are stripped with hot 14–20 wt% H_2SO_4 which is boiled to precipitate niobic acid, leaving the iron in solution.

Another method of purifying niobium is by distillation of the anhydrous mixed chlorides (29). Niobium and tantalum pentachlorides boil within about 15°C of one another which makes control of the process difficult. Additionally, process materials must withstand the corrosion effects of the chloride. The system must be kept meticulously anhydrous and air-free to avoid plugging resulting from the formation of niobium oxide trichloride, $NbOCl_3$. Distillation has been used commercially in the past.

A process has been developed to recover niobium from ferroniobium (30). The need for this process came about when Brazil would only export niobium in the form of ferroniobium. The process starts with a hydriding step, so as to be able to crush the alloy. Screening precedes a nitriding step, followed by an acid leach of the iron nitrides. This leaves the niobium nitride for further processing to the pure niobium metal.

Once purification of the niobium has been effected, the niobium can be reduced to the metallic form. The double fluoride salt with potassium, K_2NbF_7, can be reduced using sodium metal. The reaction is carried out in a cylindrical iron vessel filled with alternating layers of K_2NbF_7 and oxygen-free sodium:

$$K_2NbF_7 + 5\ Na \longrightarrow Nb + 2\ KF + 5\ NaF$$

Use of excess sodium drives the reaction, usually done under an argon or helium blanket, to completion. After cooling, the excess sodium is leached with alcohol and the sodium and potassium fluorides are extracted with water, leaving a mass of metal powder. The metal powder is leached with hydrochloric acid to remove iron contamination from the crucible.

Fused-salt electrolysis of K_2NbF_7 is not an economically feasible process because of the low current efficiency (31). However, electrowinning has been used to obtain niobium from molten alkali halide electrolytes (32). The oxide is dissolved in molten alkali halide and is deposited in a molten metal cathode, either cadmium or zinc. The reaction is carried out in a ceramic or glass container using a carbon anode; the niobium alloys with the cathode metal, from which it is freed by vacuum distillation, and the niobium powder is left behind.

Niobium pentoxide can be reduced with carbon in a two-step process, called the Balke process. Formation of the carbide is the first step. The oxide is mixed with the stoichiometric amount of lamp black, placed in a carbon crucible, and heated in vacuum to 1800°C:

$$Nb_2O_5 + 7\ C \longrightarrow 2\ NbC + 5\ CO$$

The carbide then is remixed with a stoichiometric amount of oxide, compacted into chunks, and refired to >2000°C under reduced pressure:

$$5\ NbC + Nb_2O_5 \longrightarrow 7\ Nb + 5\ CO$$

The product chunks are hydrided, crushed, and dehydrided. The resultant powder is blended and pressed into bars which are purified by high temperature sintering. The sintering removes all of the carbon and most of the oxygen and is followed by consolidation by either arc or electron-beam melting.

Niobium pentoxide also is reduced to metal commercially by the aluminothermic process. The finely ground powder is mixed with atomized aluminum and an accelerator compound which gives extra heat during reaction, then is ignited. The reaction is completed quickly and, after cooling, the slag is broken loose to free the metal derby which is purified by electron-beam melting.

The pentachloride $NbCl_5$ can be reduced with hydrogen to yield a metal powder of high purity comparable to electron-beam-melted metal from other reduction processes. However, the large excess of hydrogen which is required and the attending safety problems make this route undesirable. Niobium pentachloride also can be reduced by the Kroll process, ie, decomposition of the halide using magnesium, and by reduction with oxygen-free sodium to yield niobium sponge, which must be consolidated.

Powder and sponge may be compacted at 0.7 GPa (6900 atm) into bars which are presintered at 1400–1500°C in vacuum. The bars then are resistance-heated in high vacuum to slightly below the melting point. After cooling, the bars are rolled to consolidate the pores and are resintered at 2300°C to yield a fabricable metal product of 98% theoretical density.

Arc melting also can be used to consolidate bars of metal that are pressed from powder or sponge and used as consumable electrodes in a low voltage, high current arc. The bar is suspended vertically and the molten metal falls from the bottom of the bar onto a water-cooled copper crucible, from which it is removed as an ingot.

The most common method in commercial use is electron-beam melting. The furnace essentially is a large thermionic vacuum tube. A high current, ie, >10 A, beam of electrons at >20 kV is focused magnetically on the bottom of a suspended metal compact bar. The molten metal globules fall into a pool on top of the ingot which is contained in a water-cooled copper cylinder. The ingot is retracted at the rate that it is formed. Impurities are boiled out of the molten pool and either are pumped away or deposited on the furnace walls as slag. The final ingot is 20–30 cm in diameter and 1.5–2 m long. Scrap niobium can be recycled by the hydride–dehydride process. Heating niobium under hydrogen pressure results in the formation of niobium hydride, which is brittle enough to crush and size. The crushed hydride can be reheated in a vacuum to form niobium metal powder.

Niobium metal is available as ingot, sheet, rod, and wire and can be fabricated and formed by most metallurgical and engineering techniques (33). Cold working is necessary to avoid embrittlement which results from absorption of oxygen and nitrogen. Niobium can be reduced by 50–90% without intermediate annealing. When work hardening occurs, annealing may be done in an inert atmosphere or at <10 mPa (7.5×10^{-5} mm Hg) at 1300–1400°C. The recrystallization temperature is 1050°C.

1.4. Economic Aspects. Information on the economic aspects of niobium and its compounds is available (34–36). As can be seen in Table 3, Brazil has the most niobium-bearing ore reserves, and is also the leading producer of niobium mineral concentrates (36). Canada is a distant second. United States has some small reserves, but as of this writing is not doing any mining of niobium ores. In Brazil, pyrochlore is mainly mined by mechanized open pit, whereas Canada's pyrochlore comes from underground mining. Columbite, which is the chief ore being mined in most of the other countries, is mined by simple hand operations to hydraulic monitors and dredges at placer deposits. The principal concentrate producers are the Araxa (Companhia Brasilira de Metallurgica e Mineracao) and Catala mines (Mineracao Catalao de Goias Ltda) in Brazil, plus Niobec (a Teck/Cambior 50/50 joint venture) in Canada.

The average price of contained niobium in concentrates for the period of 1960 to 1993 is shown in Figure 1. The peak price of almost \$22/kg in 1977 resulted from increasing demand, inflation, and higher labor costs. The gradual decrease of prices throughout the 1980s was because of the large quantities of pyrochlore produced in Brazil and Canada. Prices for principal niobium products are shown in Table 4. Surplus supplies of niobium coming from the former USSR in the early 1990s kept prices low throughout the niobium industry.

The value of contained metal in annual production of niobium in the Western World was $90 million at the average 1993 price (34). U.S. consumption of niobium has been in the range of 3300 to 3500 metric tons from 1989–1993 of which over 90% is used as alloy additions in steels. Of the five domestic U.S. producers of niobium products in 1993, only two integrated niobium from raw material to end products: Shieldalloy Metallurgical Corp. and Cabot Corp., which process both niobium and tantalum products. Reading Alloys, Inc. and Teledyne Wah Chang Corp. are principal producers of high purity niobium products; Kennametal Inc. is a principal producer of niobium carbides.

1.5. Analytical Methods. The lack of stable niobium compounds resulting from a pronounced tendency for hydrolysis to colloidal suspensions of the hydrated oxide has given rise to an extensive body of literature (37–40). The analysis of niobium by classical methods involves solubilization of the sample; separation of gross interferences by selective precipitation, solvent extraction, ion exchange (qv), or another chromatographic technique; and determination by colorimetric, spectrometric, or gravimetric methods. Three of the most popular methods for dissolution of niobium metal, alloys, oxide, ores, and minerals include the following. (*1*) Addition of nitric acid to hydrofluoric acid aids in dissolving metal and alloy samples. Hydrofluoric acid solutions are used in anion-exchange separations, colorimetric determinations, and solvent-extraction separations. (*2*) Fusion of niobium ores with potassium hydroxide or potassium carbonate followed by water leaching of the cooled melts dissolves the niobium and tantalum as niobates and tantalates, respectively. Addition of sodium chloride to saturate the alkaline solution precipitates almost quantitatively the sparingly soluble sodium salts. Acidification of a suspension of the sodium salts precipitates the mixed hydrated oxides for further processing. (*3*) Fusion with potassium bisulfate, $KHSO_4$, or pyrosulfate, $K_2S_2O_7$, is the most widely used route to solubilize the ignited oxides or ores. The sample is fused in a quartz crucible with an eight- to tenfold excess of reagent to a clear melt at 650–800°C. The cooled melt is dissolved in a saturated solution of ammonium oxalate or 20 wt% tartaric acid or citric acid. A large number of precipitation reactions for the determination of niobium and tantalum are based on these acid solutions of oxalato or tartrato complexes. It is necessary to maintain a large excess of oxalic or tartaric acid for stability of the complexes. The oxalic acid solutions are more stable but the tartrato complexes can be made alkaline without precipitation of niobic and tantalic acids.

Of the gravimetric procedures, precipitation with tannin from a slightly acidic oxalate solution is probably the best known. The hot oxalate extract of a potassium pyrosulfate fusion is adjusted with ammonia to pH 3.7–4.0 using Bromothymol Blue. Dropwise addition of a 2 wt% solution of tannin precipitates the lemon-yellow complex of tantalum. Several grams of ammonium chloride are added to prevent peptization, and the solution is digested and filtered. Further addition of tannin and ammonia to the clear filtrate precipitates the vermilion-colored complex of niobium. Many other organic precipitants have been used, including Cupferron, *N*-benzoyl-*N*-phenylhydroxylamine, 8-quinolinol (Oxine), phenylarsonic acid, pyrogallol, and others.

Solvent extraction techniques are useful in the quantitative analysis of niobium. The fluoro complexes are amenable to extraction by a wide variety of ketones. Some of the water-insoluble complexes with organic precipitants are extractable by organic solvents and colorimetry is performed on the extract. An example is the extraction of the niobium–oxine complex with chloroform (41). The extraction of the niobium–pyrocatechol violet complex with tridodecylethylammonium bromide and the extraction of niobium–pyrocatechol–sparteine complex with chloroform are examples of extractions of water-soluble complexes. Colorimetry is performed on the extract (42,43). Colorimetry may also be performed directly on the water-soluble complex, eg, using ascorbic acid and 5-nitrosalicylic acid (44,45).

Chromatographic methods play a prominent role in the clean separation of niobium from tantalum and other metals (see CHROMATOGRAPHY). The use of methyl ethyl ketone as the eluent for the cellulose column separation of niobium and tantalum in dilute hydrofluoric acid–ammonium fluoride solutions represented a significant advance over the tannin precipitations. The anionic-exchange separation of niobium from tantalum and other metals from a hydrochloric–hydrofluoric acid medium is accomplished by elution with ammonium chloride–hydrofluoric acid (46). This same type of ion-exchange separation is used to determine Nb along with Ta in various matrices, such as Inconel (47), FeNb alloys (48), and titanium (49). Once niobium is isolated, it may be determined gravimetrically by precipitation as an insoluble complex or as the earth acid, by firing to the oxide at 800–1000°C, and by weighing. Alternatively, the precipitate may be redissolved and determined colorimetrically in aqueous solution or in an organic extract.

The determination of the presence of the interstitial gases hydrogen, nitrogen and oxygen, and carbon are important because of the affects they have on the physical properties of niobium and its alloys. Hydrogen, nitrogen, and oxygen are most often determined by inert gas fusion. The gas of interest is captured and separated through a chromatographic procedure where detection is by infrared spectroscopy or thermal conductivity (50, 51). Carbon determinations are almost entirely done by automated instrumentation. Combustion of the sample in a stream of oxygen converts the carbon to carbon dioxide which is then detected by infrared spectroscopy (52).

Spectroscopic methods for the determination of impurities in niobium include the older arc and spark emission procedures (53) along with newer inductively coupled plasma source optical emission methods (54). Some work has been done using inductively coupled mass spectroscopy to determine impurities in niobium (55,56). X-ray fluorescence analysis, a widely used method for niobium analysis, is used for routine work by niobium concentrates producers (57,58). Paying careful attention to matrix effects, precision and accuracy of x-ray fluorescence analyses are at least equal to those of the gravimetric and ion-exchange methods.

1.6. Health and Safety Factors. Toxicity data on niobium and its compounds are sparse. The most common materials, eg, niobium concentrates, ferroniobium, niobium metal, and niobium alloys, appear to be relatively inert biologically. Limited animal experiments show high toxicity for some salts which are related to disturbance of enzyme action. Niobium hydride has moderate fibrogenic and general toxic action. Recommended maximum allowable concentrations are 6 mg/m^3 (59). Recommended maximum permissible concentration of Nb in reservoir water is 0.01 mg/L. Rats receiving 0.005 mg/kg daily over nine months show changes in cholinesterase activity but no effects are observed at doses of 0.0005 mg/kg. The threshold for affecting clarity and biological oxygen demand (BOD) is 0.1 mg/L (60).

Unstable niobium isotopes that are produced in nuclear reactors or similar fission reactions have typical radiation hazards (see RADIOISOTOPES). The metastable ^{93}Nb, $t_{1/2} = 14$ yr, decays by 0.03 MeV gamma emission to stable ^{93}Nb; ^{95}Nb, $t_{1/2} = 35$ d, a fission product of ^{235}U, decays to stable ^{95}Mo by emission of 0.16 MeV β- and 0.77 MeV γ-radiation; and ^{97}Nb, $t_{1/2} = 72$ min, decays to stable ^{97}Mo by emission of 1.27 MeV β- and 0.66 MeV γ-radiation. Inhalation experiments on mice have been performed using aerosols of radioniobium ^{95}Nb, prepared at 100, 250, 600, and 1100°C to give particles of different chemical compositions (61). The activity was restricted to the lungs for the 600 and 1100°C aerosols, but was translocated partially to the skeleton for the 100 and 250°C aerosols. At the lower temperatures, the highest radiation dose was to the lungs, skeleton, and liver; whereas the radiation dose was delivered exclusively to the lungs at the higher temperatures.

Coarse metal niobium and niobium alloy powders are difficult to ignite, even at 300°C, but high surface area material such as 5 micrometer powder may autoignite at room temperature. Fire fighting procedures for niobium and niobium hydride powder suggest letting the fire burn itself out. Small fires can be controlled by smothering with dry table salt or using Type D dry powder fire-extinguishing material. Under no circumstances should water be used, as a violent explosion may result. Carbon dioxide is not effective in extinguishing burning metal (62).

1.7. Uses. Niobium, as ferroniobium, is used extensively in the steel industry as an additive in the manufacture of high strength, low alloy (HSLA) and carbon steels. The ferroniobium acts as a grain refiner to increase yield and tensile strength at additions as low as 0.02 wt%. Normal usage is 0.03–0.1 wt%. The most important application of niobium HSLA or microalloyed steels is in oil- and gas-pipeline steels, particularly those which may experience operating conditions below −25°C (see PIPELINES). The niobium microalloyed steels also are widely used in automobiles, buildings, bridges, ships, towers, concrete reinforcing bars, etc, where the strength-to weight and cost-per-unit-strength ratios are particularly advantageous. The distribution of the uses of iron and nickel alloys of niobium is given in Table 5.

Addition of niobium to austenitic stainless steels inhibits intergranular corrosion by forming niobium carbide with the carbon that is present in the steel. Without the niobium addition, chromium precipitates as a chromium carbide film at the grain boundaries and thus depletes the adjacent areas of chromium and reduces the corrosion resistance. An amount of niobium equal to 10 times the carbon content is necessary to prevent precipitation of the chromium carbide.

Niobium is also important in nonferrous metallurgy. Addition of niobium to zirconium reduces the corrosion resistance somewhat but increases the mechanical strength. Because niobium has a low thermal-neutron cross section, it can be alloyed with zirconium for use in the cladding of nuclear fuel rods. A Zr–1%Nb [11107-78-1] alloy has been used as primary cladding in the countries of the former USSR and in Canada. A Zr–2.5 wt% Nb alloy has been used to replace

Zircaloy-2 as the cladding in Candu-PHW (pressurized hot water) reactors and has resulted in a 20% reduction in wall thickness of cladding (63) (see NUCLEAR REACTORS).

Niobium is a common additive to the nickel- and cobalt-based superalloys. Addition levels are typically 1–2.5 wt% in the cobalt-based alloys and 2–5 wt% in the nickel-based alloys. Niobium-based alloys with tungsten, titanium, and zirconium have superior strength and corrosion resistance up to 1200°C; eg, C-103, a Nb–Hf–Ti alloy having Ta, Ti, and Zr as low impurities, retains high yield strength at higher temperatures. It is used in rocket nozzles and afterburners along with turbine blades by the aerospace and aircraft industries. Sodium vapor lamps take advantage of the Nb–1%Zr alloy's resistance to metallic sodium heated to 871–927°C (64). The alloy is also used in the SP 100 reactor program for the space station.

A use small in total quantity consumed is as anodized niobium for jewelry. Niobium oxide is used as a substitute for the higher priced tantalum oxide in optical glass (65). A niobium alloy, Ti–45% Nb, originally developed as a superconducting material, has found another application in ventlines of gold mining autoclaves where these autoclaves better resist corrosion and erosion while resisting the danger of ignition (66).

The corrosion resistance of niobium and its high electrical conductivity and ductility make it a valuable structural material for chemical and metallurgical applications. The heat-transfer coefficient of niobium is more than twice that of titanium and three times higher than zirconium and stainless steels. Niobium is corrosion-resistant to most media, with the exception of hydrofluoric acid and hot concentrated hydrochloric and sulfuric acids. The pickling solution for removal of normal surface oxides is one part nitric acid, one part sulfuric acid, two parts hydrofluoric acid, and four parts water (by volume) (see METAL SURFACE TREATMENTS). Niobium also shows good corrosion resistance to sulfidizing atmospheres of low oxygen potential, which may be used in the production of substitute natural gas from sulfur-containing materials (67). Liquid sodium, potassium, sodium–potassium alloys, or lithium have little effect on niobium up to 1000°C, and its resistance to many other liquid metals is good.

Niobium is used as a substrate for platinum in impressed-current cathodic protection anodes because of its high anodic breakdown potential (100 V in seawater), good mechanical properties, good electrical conductivity, and the formation of an adherent passive oxide film when it is anodized. Other uses for niobium metal are in vacuum tubes, high pressure sodium vapor lamps, and in the manufacture of catalysts.

Niobium carbide is used as a component of hard metals, eg, mixtures of metal carbides that are cemented with cobalt, iron, and nickel. Along with tantalum carbide, niobium carbide is added to impart toughness and shock and erosion resistance. The spiraling rise in the price of tantalum has spurred the development of a hafnium carbide–niobium carbide substitute for tantalum carbide (68). These cemented carbides are used for tool bits, drill bits, shovel teeth, and other wear-resistant components; turbine blades; and as dies in high pressure apparatus (see CARBIDES).

Niobium and many of its alloys exhibit superconductivity, ie, the lack of electrical resistance at very low temperatures, thus they are of great interest for power generation (qv), propulsion devices, fusion energy (qv) research, electronic devices, particle accelerators, and other applications. Niobium becomes superconducting at 9.15 K. Other niobium compounds and their transitional temperatures are NbTi [12384-42-8], 9.5 K; Nb_3In [12030-07-8], 9 K; Nb_3Sn [12035-04-0], 18 K; Nb_3Al [12003-75-7], 18.8 K; Nb_3Ga [12024-05-4], 20 K; and Nb_3Ge [12025-22-8], 23 K. Most superconducting devices use niobium–titanium because of ease of its fabrication into magnet wire, which is its most common application. Where very high magnetic fields are necessary, niobium–tin, Nb_3Sn, conductors are used, even though the intermetallic nature of Nb_3Sn makes fabrication difficult. Improved methods of fabrication should lead to wider use of Nb_3Sn and to commercial application of niobium–aluminum and niobium–aluminum–germanium superconductors. Niobium oxide has started replacing the more expensive tantalum oxide as a raw material in lanthanum borate glasses. It has also found wide applications in ultrasonics, acoustooptics, electrooptics, and nonlinear optics.

2. Niobium Compounds

2.1. Niobium Boride. A number of niobium boride phases have been described in the literature, ie, Nb_2B [12344-74-0], Nb_3B [56450-58-9], Nb_3B_4 [12045-89-5], NbB, and NbB_2. Only the monoboride and the diboride melt congruently; NbB_2 decomposes at the melting point to NbB and boron (69). Some of the properties of these niobium borides together with other niobium compounds are listed in Table 6.

The most common methods of preparation have been hot-pressing, sintering, or remelting powdered mixtures of elemental boron with niobium or niobium hydride (103,104). Other methods are the reduction of a mixture of Nb_2O_5 and B_2O_3 with aluminum, silicon, or magnesium (105); carbon reduction at 2000°C of Nb_2O_5 and B_2O_3 (106); reaction of carbon with B_4C and Nb_2O_5 (107); electrolysis of molten mixtures of Nb_2O_5 with alkali metal and alkaline-earth metal borates and fluorides to produce NbB_2 (108,109); chemical vapor deposition (CVD) onto a hot substrate by a mixture of niobium halide, boron halide, hydrogen, and argon (77–110); and CVD of boron on niobium accompanied or followed by diffusion into the substrate (111) (see THIN FILMS, FILM FORMATION TECHNIQUES). Niobium diboride generally is a gray powder; is unattacked by hydrochloric acid, nitric acid, or aqua regia; is attacked slowly by hot sulfuric or hydrofluoric acid; and is dissolved rapidly by molten alkali, hydroxides, carbonates, bisulfates, and sodium peroxide. It is oxidized in air at red heat.

2.2. Niobium Carbide. Apparently three solid single-phase regions exist in the niobium–carbon system, ie, a solid solution of carbon in niobium (bcc), Nb_2C (hexagonal), and NbC (fcc) (77). The compositional range of Nb_2C is very limited, whereas NbC varies from $NbC_{0.7}$ to nearly stoichiometric NbC. Thermodynamic values for these phases have been reported (81,112,113). Industrial preparation utilizes Nb_2O_5 and carbon as starting materials. The reaction starts at ca 675°C but temperatures of 1800–2000°C are needed for completion of the reaction. Heating the elemental powders also produces NbC if a sufficiently high final temperature is used. Chemical vapor deposition (CVD) can be used to deposit NbC on a hot surface by reaction of $NbCl_5$ and hydrogen and hydrocarbons. Niobium carbide powder has a gray metallic color up to a composition of $NbC_{0.9}$; the color changes to lavender upon addition of carbon up to $NbC_{0.99}$. NbC is unreactive and resists boiling in aqua regia; a mixture of HNO_3 and HF is needed for dissolution. NbC burns on heating in air to >1100°C and can be converted to the nitride by heating in nitrogen or ammonia.

2.3. Niobium Halides and Oxyhalides.

All possible halides of pentavalent niobium are known and preparations of lower valent halides generally start with the pentahalide. Ease of reduction decreases from iodide to fluoride.

Niobium Pentafluoride. Niobium pentafluoride is prepared best by direct fluorination of the metal with either fluorine or anhydrous hydrofluoric acid at 250–300°C. The volatile NbF_5 is condensed in a pyrex or quartz cold trap, from which it can be vacuum-sublimed at 120°C to yield colorless monoclinic crystals. It is very hygroscopic and reacts vigorously with water to give a clear solution of hydrofluoric acid and H_2NbOF_5 [12062-01-0]. This acid also is formed by dissolving niobium metal or niobic acid in hydrofluoric acid and, at high acid concentrations, it is converted to H_2NbF_7. Addition of potassium fluoride to a solution of H_2NbOF_5 precipitates $K_2NbOF_5{\cdot}H_2O$, which is soluble in hot water and can be recrystallized from a saturated solution to give large monoclinic platelets. The high solubility of $K_2NbOF_5{\cdot}H_2O$ in water was the basis for early separations of tantalum and niobium because the corresponding tantalum salt K_2TaF_7 is 12 times less soluble in 1 wt% HF at 20°C.

Niobium Dioxide Fluoride. Niobium dioxide fluoride, NbO_2F, is formed on dissolution of niobium pentoxide in 48 wt% aqueous hydrofluoric acid, evaporation of the solution to dryness, and heating to 250°C.

Niobium Pentachloride. Niobium pentachloride can be prepared in a variety of ways but most easily by direct chlorination of niobium metal. The reaction takes place at 300–350°C. Chlorination of a niobium pentoxide–carbon mixture also yields the pentachloride; however, generally the latter is contaminated with niobium oxide trichloride. The pentachloride is a lemon-yellow crystalline solid that melts to a red-orange liquid and hydrolyzes readily to hydrochloric acid and niobic acid. It is soluble in concentrated hydrochloric and sulfuric acids, sulfur monochloride, and many organic solvents.

Niobium Oxide Trichloride. Niobium oxide trichloride, $NbOCl_3$, also can be prepared in a variety of ways, ie, oxidation of the pentachloride by air, reaction of the pentoxide with the pentachloride, reaction of carbon tetrachloride or HCl gas with the pentoxide at 400–700°C, and as an intractable impurity in most preparations of the pentachloride. It is a white solid that sublimes at ca 200°C, is thermally unstable, and forms the pentoxide and the pentachloride at higher temperatures.

Niobium Pentabromide. Niobium pentabromide is most conveniently prepared by reaction of bromine with niobium metal at ca 500°C. It is a fairly volatile yellow-red compound that is hygroscopic and readily hydrolyzes. It is soluble in water, alcohol, and ethyl bromide.

Niobium Oxide Tribromide. Niobium oxide tribromide, NbOBr, is a yellowbrown solid which is readily hydrolyzed by moist air. It is prepared by reaction of bromine with a mixture of niobium pentoxide and carbon at 550°C. It decomposes in vacuum to the pentabromide and pentoxide at 320°C.

Niobium Pentaiodide. Brass-yellow crystals of niobium pentaiodide are formed by direct reaction of excess iodine with niobium metal in a sealed tube (114). It is thermally unstable and decomposes to the tetraiodide [13870-21-8] at 206–270°C in vacuum (115).

2.4. Niobium Hydride.

Hydrogen reacts exothermically with niobium to form a stable interstitial solid solution. In a gas-phase reaction at 300–1500°C and hydrogen pressures of 0–101 kPa (0–1 atm), the lattice parameter and heat of solution increase with hydrogen content up to the composition $NbH_{0.85}$ (116). X-ray studies show a linear relation between atomic volume and the hydrogen content (117). The absorption of hydrogen is proportional to the square root of the hydrogen pressure, which indicates dissociation of molecular hydrogen at the metal surface and diffusion of hydrogen atoms (118). Although the hydride is stable at room temperature, heating to 500°C at 0.67 kPa (5 mm Hg) decomposes the hydride to hydrogen and niobium metal. The expansion on absorption and contraction on desorption of hydrogen by the metal lattice usually leaves the metal in powder form after a hydride–dehydride operation, a process of commercial value for production of metal powder and recovery of metal scrap. An unstable dihydride, NbH_2 [13981-96-9], has been prepared by the cathodic hydrogenation of niobium foil in 6 *N* sulfuric acid (119). It is very unstable and decomposes in vacuum or air to the monohydride (see HYDRIDES).

2.5. Niobium Nitrides.

The uptake of nitrogen by niobium metal proceeds by the exothermic formation of an interstitial solid solution of nitrogen atoms in the bcc lattice of the metal. The solubility of nitrogen in the metal is proportional to the square root of the nitrogen partial pressure until the formation of the nitride phase Nb_2N. This relation holds from 1200–2400°C and over almost 10 orders of magnitude of the pressure (120). At the solubility limit of the solid solution α-phase, the hcp β-phase appears, which has a composition of $NbN_{0.4}$ to $NbN_{0.5}$. Further absorption of nitrogen leads to the formation of a fcc δ-phase with a homogeneity of $NbN_{0.88}$ to $NbN_{0.98}$. This δ-phase is stable only above 1230°C. Niobium nitride can be prepared by heating the metal in nitrogen or ammonia to 700–1100°C (83), by heating the pentoxide and carbon to 1250°C in the presence of nitrogen (121), and by CVD using $NbCl_5$, H_2, and N_2 (111,122). The nitride is a light gray powder with a yellow cast; it is insoluble in HCl, HNO_3, and H_2SO_4; it is attacked by hot caustic, lime, or strong alkalies with the evolution of ammonia. It reacts when heated in air to form the pentoxide and to liberate nitrogen. Nb_2N is resistant to acids by reacts with strong alkali to liberate nitrogen rather than ammonia (see NITRIDES).

2.6. Niobium Oxides.

The solubility of oxygen in niobium obeys Henry's law to the solubility limit of the first oxide phase of 850–1300°C (123). The amount of oxygen in solution in niobium is 1.3 at. % at 850°C and nearly 2 at. % at 1000°C (124). Only three clearly defined anhydrous oxides of niobium have been obtained in bulk, ie, NbO, NbO_2, and Nb_2O_5. Niobium monoxide, NbO, is obtained by hydrogen reduction of the pentoxide at 1300–1700°C or by heating a compressed mixture of the metal powder with NbO_2 in argon at 1700°C. It has a gray metallic appearance. Niobium dioxide, NbO_2, also can be obtained by hydrogen reduction of the pentoxide at 800–1300°C, by heating a properly proportioned mixture of the pentoxide and the metal, or by thermal dissociation of the pentoxide at 1150°C in an argon sweep. Niobium dioxide is black with a bluish cast, is a strong reducing agent in the dry state, and is converted to the pentoxide on ignition in air.

The considerable confusion existing in the literature regarding the polymorphism of Nb_2O_5 seems to have been resolved. Three distinct phases have been identified: a low temperature phase T, a middle temperature phase M, and a high temperature phase H (125). With regard to the amorphous oxide produced from the hydrolysis of niobic acid, conversion to the crystalline T form occurs at 500°C and is followed by transformation to the M form at 1000°C and to the H form at 1100°C. These phases were renamed γ (T), β (M), and α (H). The transition of amorphous to γ-phase was found to occur at 440°C (126). Heating at 830°C irreversibly converts the γ-phase to a mixture of β- and α-forms and further heating to 1095°C irreversibly transforms this mixture to the pure α-form. It was concluded that the β-form is an imperfectly crystallized α-phase. Subsequent work has demonstrated the existence of another metastable phase, ε (127). It appears that three crystalline forms of Nb_2O_5 are detectable at atmospheric pressure and that the α-form is the only stable structure and is monoclinic with 14 formula units in the unit cell (99,128). The α-phase can be prepared by heating the metal carbide, nitride, or niobic acid at >1100°C and has been prepared in the form of large single crystals (129). It generally is an eggshell-white powder, and turns yellowish on heating because of the formation of oxygen vacancies in the lattice. It is insoluble in acids, except hydrofluoric, and can be dissolved by fused alkali pyrosulfates, carbonates, or hydroxides.

2.7. Niobic Acid and Salts.

Niobic Acid. Niobic acid, $Nb_2O_5{\cdot}xH_2O$, includes all hydrated forms of niobium pentoxide, where the degree of hydration depends on the method of preparation, age, etc. It is a white insoluble precipitate formed by acid hydrolysis of niobates that are prepared by alkali pyrosulfate, carbonate, or hydroxide fusion; base hydrolysis of niobium fluoride solutions; or aqueous hydrolysis of chlorides or bromides. When it is formed in the presence of tannin, a voluminous red complex forms. Freshly precipitated niobic acid usually is colloidal and is peptized by water washing, thus it is difficult to free from traces of electrolyte. Its properties vary with age and reactivity is noticeably diminished on standing for even a few days. It is soluble in concentrated hydrochloric and sulfuric acids but is reprecipitated on dilution and boiling and can be complexed when it is freshly made with oxalic or tartaric acid. It is soluble in hydrofluoric acid of any concentration.

Niobates. Niobic acid is amphoteric and can act as an acid radical in several series of compounds, which are referred to as niobates. Niobic acid is soluble in solutions of the hydroxides of alkali metals to form niobates. Fusion of the anhydrous pentoxide with alkali metal hydroxides or carbonates also yields niobates. Most niobates are insoluble in water with the exception of those alkali metal niobates having a base-to-acid ratio greater than one. The most well-known water-soluble niobates are the 4:3 ad the 7:6 salts (base:acid), having empirical formulas $M_8Nb_6O_{19}$ (aq) and $M_{14}Nb_{12}O_{37}$ (aq), respectively. The hexaniobate is hydrolyzed in aqueous solution according to the pH-dependent reversible equilibria (130), when the pH is ca 9.

$$Nb_6O_{19}^{8-}\ (aq) \overset{H_2O}{\rightleftharpoons} HNb_6O_{19}^{7-}\ (aq) + OH^-$$

$$Nb_6O_{19}^{7-}\ (aq) \overset{H_2O}{\rightleftharpoons} HNb_6O_{18}^{6-}\ (aq) + OH^-$$

The 7:6 salts are the acid salts of the normal 4:3 hexaniobates. The formulas can be written as $M_7H(Nb_6O_{19})$ (aq). Further hydrolysis can take place. At pH ca 4.5, the irreversible precipitation of niobic acid occurs.

$$H_6(Nb_6O_{18}) \longrightarrow 3\ Nb_2O_5 + 3\ H_2O$$

The potassium salts are the most soluble and other salts usually are precipitated by addition of the appropriate metal chloride to a solution of the corresponding potassium salt. The metaniobates, $MNbO_3$, and orthoniobates, $MNbO_4$, generally are prepared by fusion of the anhydrous mixed oxides. The metaniobates crystallize with the perovskite structure and are ferroelectric (131) (see FERROELECTRICS). The orthoniobates are narrow band-gap semiconductors (qv) (132).

Sodium metaniobate(1:1) [67211-31-8], $Na_2O{\cdot}Nb_2O_5{\cdot}7H_2O$ or $Na_2Nb_2O_6{\cdot}7H_2O$, separates as colorless triclinic crystals as a result of concentrating the mother liquor from the preparation of the 7:6 sodium niobate by spontaneous evaporation. It also can be obtained by fusion of the anhydrous pentoxide in sodium hydroxide or carbonate.

Potassium niobate(4:3) [12502-31-7], $4K_2O{\cdot}3Nb_2O_5{\cdot}16H_2O$ or $K_8Nb_6O_{19}{\cdot}16H_2O$, is obtained by dissolving niobic acid in a concentrated solution of potassium hydroxide. The large monoclinic crystals are separated by concentrating the solution. The salt is very soluble; at room temperature a saturated solution contains 425 g/100 g of water. It is much more soluble in hot water and prone to form supersaturated solutions.

Sodium niobate(7:6) [12201-59-1], $7Na_2O{\cdot}6Nb_2O_5{\cdot}31H_2O$ or $Na_{14}Nb_{12}O_{37}{\cdot}31H_2O$, forms a crystalline precipitate when a hot solution of a soluble niobium compound is added to a hot concentrated sodium hydroxide solution. It is insoluble in the presence of excess sodium hydroxide but is sparingly soluble in pure water. It also can be formed by addition of sodium hydroxide or chloride to a solution of the 4:3 potassium niobate.

Lithium niobate [12031-63-9], $Li_2O{\cdot}Nb_2O_5$ or $LiNbO_3$, is prepared by the solid-state reaction of lithium carbonate with niobium pentoxide. After being separately predried at 150–200°C, the stoichiometric amounts of the oxides are carefully mixed and heated to 600°C in a platinum crucible. The temperature is increased slowly for 12 h from 600 to 800°C and is maintained at 800°C for another 12 h. The mixture is cooled, crushed, and reheated to 900°C for 12 h. The product may not be completely homogeneous.

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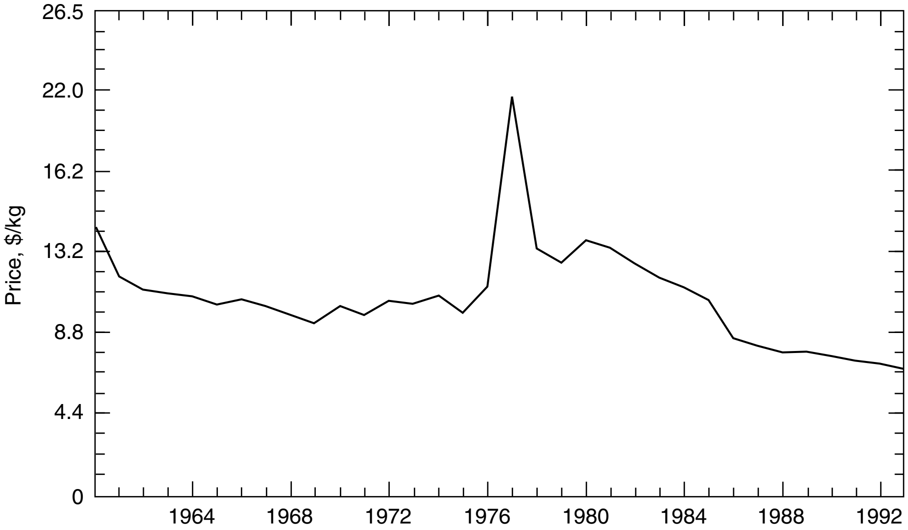

Fig. 1. Average annual price of contained niobium in concentrates, based on constant 1987 dollars (36).

Table 1. **Properties of Niobium**

Property	Value	Reference
atomic number	41	
atomic weight	92.906	
atomic volume, cm^3/mol	10.8	
atomic radius, nm	0.147	
electronic configuration	$[Kr]4d^45s^1$	
ionization potential, eV	6.77	
crystal structure	bcc	
lattice constant at 0°C, pm	330.04	
density at 20°C, g/cm^3	8.66	
mp, °C	2468 ± 10	
bp, °C	5127	
latent heat of fusion, kJ/mol[a]	26.8	5
latent heat of vaporization, kJ/mol[a]	697	5
heat of combustion, kJ/mol[a]	949	6
heat capacity, J/(mol·K)[a]		5
at 298 K	24.7	
1500 K	29.7	
3000 K	33.5	
entropy, J/(mol·K)[a]		5
at 298 K	36.5	
1500 K	79.6	
3000 K	111.6	
vapor pressure at 2573 K, mPa[b]	22	7
evaporation rate at 2573 K, $\mu g/(cm^2 \cdot s)$	1.9	7
thermal conductivity at 298 K, W/(m·K)	52.3	
coefficient of linear thermal expansion, 291–373 K, $°C^{-1}$	7.1×10^6	8
volume electrical conductivity, % IACS[c]	13.3	9
electrical resistivity, $\Omega \cdot m$	$13 - 16 \times 10^{-6}$	
temperature coefficient of resistivity, $°C^{-1}$	3.95×10^{-3}	
work function, eV	4.01	
secondary emission (primary $\delta_{max} = 400$ V), eV	1.18	
positive ion emission, eV	5.52	

[a] To convert J to cal, divide by 4.184.

[b] To convert mPa to μm Hg, divide by 133.3.

[c] IACS = International Annealed Copper Standard. Pure copper = 100%.

Table 2. **Corrosion of Niobium Metal**[a]

Medium	Temperature, °C	Concentration, wt%	Corrosion rate, μm/yr
sulfuric acid[b]	23	96	0.5
	50	40	5
	100	20	0.5
	145	96	5,000
	bp	70	dissolves
	190[c]	1–10	slight
	250[c]	1–10	slight
	250[c]	20	250
	250[c]	30	1,300
hydrochloric acid	bp	1	
	bp	5	
	bp	10	100
	bp	15	450
	bp	20	1,000
	190[c]	5	30
	190[c]	10	500
	190[c]	15	14,000
hydrofluoric acid	23	all	very high
nitric acid	bp	70	
	190[c]	70	
	250[c]	70	
phosphoric acid	23	85	0.5
	100	85	80
aqua regia	23		0.5
	55		20
sodium hydroxide	23	10	20
	23	40	30
potassium hydroxide	23	40	90
zinc chloride	bp	40	
ferric chloride	23	10	
formic acid	bp	10	
acetic acid	bp	≤99.7	
oxalic acid	bp	10	20
citric acid	bp	10	20
lactic acid	bp	10	10
	bp	85	2.5
trichloroacetic acid	bp	50	
trichloroethylene	bp	99[d]	

[a] Refs. (10–13).

[b] Hydrogen embrittlement at higher temperatures.

[c] In sealed tubes.

[d] 1% water present.

Table 3. **World Niobium Reserves and Reserves Base, $t \times 10^3$**[a,b]

Country	Reserves	Reserve base[c]	1993 Production
Brazil	3311	3629	9.756
Canada	136	408	2.393
United States		[d]	
Africa			
Nigeria	64	91	0.017
Zaire	32	91	0.455
Rwanda			0.030
Zimbabwe			0.014
other[e]			<0.001
Australia			0.050
Thailand			0.001
other market economy countries	6	9	
World total	*3549*	*4228*	

[a] Ref. 36.

[b] Niobium content.

[c] Reserve base includes demonstrated resources that are economic (reserves), marginally economic (marginal reserves), and some that are subeconomic (subeconomic resources).

[d] Negligible.

[e] Namibia and South Africa each had <0.5 t.

Table 4. **1995 Prices of Niobium Products**

Product	Price, $/kg
columbite ore, contained Nb	6.17–7.05
ferroniobium	
regular grade	14.51
vacuum grade	39.68
niobium metal	
sheet or foil	150–220
powder finer mesh	165–190
powder coarser mesh	100–130
niobium oxide	17.64
niobium–titanium alloys, billet, and rod	100–150
Nb alloys (C-103, Nb–1%Zr) as bar, rod, or sheet	165–285

Table 5. **1992 U.S. Consumption of Ferroniobium and Nickel Niobium, kg of Nb**[a]

Material	Quantity
steel	
carbon	837,541
stainless and heat resisting	346,936
high strength, low alloy	931,096
tool and unspecified	8,044
superalloys	411,620
alloys and miscellaneous	20,076
Total consumption	*2,555,313*

[a] Ref. 36.

Table 6. **Properties of Niobium Compounds**

Compounds	CAS Registry number	Molecular formula	Lattice	Lattice constant, pm	Density, g/cm^3	Mp, °C	Bp, °C	Specific resistivity, $\mu\Omega \cdot$ cm	Refs.
niobium boride	[12045-19-1]	NbB	orthorhombic	$a = 329.8$ $b = 872.4$ $c = 316.6$	7.5	2000		64.5^{a}	(70,71)
niobium diboride	[12007-29-3]	NbB_2	hexagonal	$a = 308.9$ $c = 330.3$	6.9^{b}	3050		65^{a,c}	(70–76)
diniobium carbide	[12011-99-3]	Nb_2C	hcp	$a = 312.7$ $c = 497.2$	7.8	3090			77
niobium carbide	[12069-94-2]	NbC	fcc	$a = 447.1$	7.788^{d}	3600	4300	180 max^{e}	(75,78–83)
niobium pentafluoride	[7783-68-8]	NbF_5	monoclinic	$a = 963$ $b = 1443$ $c = 512$ $\beta = 96.1^{f}$	3.54	79	234		(84,85)
niobium fluorodioxide	[15195-33-2]	NbO_2F	cubic	$a = 390.2$					86
niobium pentachloride	[10026-12-7]	$NbCl_5$	monoclinic	$a = 183.0$ $b = 1798$ $c = 588.8$ $\beta = 90.6^{f}$	2.74^{g}	208.3	248.2		(87,88)
niobium trichloro-monoxide	[13597-20-1]	$NbOCl_3$	tetragonal	$a = 1087$ $c = 396$	3.72	vacuum sublimes at ca 200			(89,90)
niobium pentabromide	[13478-45-0]	$NbBr_5$	orthorhombic	$a = 612.7$ $b = 658$ $c = 1855$	4.36	254	365		(91,92)

Table 6. (*Continued*)

Compounds	CAS Registry number	Molecular formula	Lattice	Lattice constant, pm	Density, g/cm^3	Mp, °C	Bp, °C	Specific resistivity, μΩ · cm	Refs.
niobium tribromo-monoxide	[14459-75-7]	$NbOBr_3$				vacuum sublimes at 180	ca 320 dec		
niobium pentaiodide	[13981-86-7]	NbI_5	monoclinic	$a = 1058$ $b = 658$ $c = 1388$ $\beta = 109.1^f$		ca 200 dec			93
niobium hydride	[13981-86-7]	NbH	bcc		6–6.6				
diniobium nitride	[12033-43-1]	Nb_2N	hcp	$a = 305.6$ -304.8 $c = 495.6$	8.08	2050			(94,95)
niobium nitride	[24621-21-4]	NbN	fcc	$a = 438.2$ -439.2	8.4^a		200^a, 450 (at mp)		(95–97)
niobium oxide	[12034-57-0]	NbO	cubic	$a = 421.08$	7.30				98
niobium dioxide	[12034-59-2]	NbO_2	tetragonal	$a = 1371$	5.90				99
α-niobiumpentoxide	[1313-96-8]	$\alpha\text{-}Nb_2O_5$	monoclinic	$a = 2116$	4.55	1491 ± 2			(100–102)

[a] At 25°C.
[b] Has a Mohs' hardness of 8+.
[c] Thermal conductivity value is 17 W/(m · k) at 23°C.
[d] Has a Mohs' hardness of 9+.
[e] Thermal conductivity value is 14 W/(m · k) at 23°C.
[f] Units are degrees.
[g] Has a hardness of 208.3.