NIOBIUM

Wah Chang

An Allegheny Technologies Company

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INTRODUCTION

Niobium, as an alloy in HSLA (High Strength Low Alloy) specialty steels, is employed in a wide variety of end uses, such as beams and girders in buildings and offshore drilling towers, special industrial machinery, oil and gas pipelines, railroad equipment, and automobiles. Niobium is also used as an additive in superalloys for jet and turbine engines and as a carbide in machining cutting tools. Its volume in nuclear, aerospace, and superconducting applications is increasing each year.

As a metal, niobium was first used in the atomic reactor program. Because niobium is relatively lightweight and can maintain its strength at elevated temperatures, it is used extensively in aerospace equipment and missiles.

Since niobium exhibits excellent corrosion resistance to liquid metal, it is used in sodium vapor lamps for highway lighting. Alloyed with titanium or tin, it has become the primary material used in superconducting applications.

At Wah Chang, niobium is processed from niobium concentrates. The raw material is extracted by chlorination followed by a metallic reduction. The metal is then electron beam melted into various ingot sizes for added purification. This metal is of the highest purity on the market today.

Vacuum consumable arc cast ingots of niobium and its alloys, up to 23 inches in diameter and 13,000 pounds in weight, are melted, extruded or forged, rolled, swaged and further fabricated into wrought products.

Niobium is available in the form of sheet, foil, rod, wire, and tubing, and can be fabricated without difficulty by most metal working techniques. It can be joined mechanically; it can be fusion or resistance welded; and, it can be brazed as long as proper techniques are employed. Many chemical products, including oxide, carbide, and halides, are also produced at Wah Chang.

The following information will help familiarize you with niobium's properties, explain its general working characteristics, and present useful specification data.

WAH CHANG - ALABAMA FACILITY

In the early 1980's, the interest in niobium-titanium products developed to the point that Teledyne, Inc. began researching the feasibility of building a new plant specializing in the development and manufacture of low temperature superconducting alloys and composites. Research and production performed at Wah Chang Albany over the previous 23 years indicated that such a facility would help promote further growth in the superconducting field.

Construction of Teledyne SC began in 1984 in Huntsville, Alabama. Less than 10 years later, the company had grown into one of the world's primary producers of superconducting rod and wire.

In the spring of 1993, Teledyne, Inc. announced that, as a part of realignment of its operating divisions, Teledyne SC would become part of its larger sister company Wah Chang, with headquarters in Albany, Oregon. The realignment joined two of the world's largest and most experienced suppliers of niobium-titanium and other superconducting alloys. In the process, Teledyne SC was re-named Wah Chang, Alabama Facility.

Niobium-titanium alloys and composites for solenoid magnet applications are used in Magnetic Resonance Imaging (MRI) devices, (which safely scan the body's soft tissues), and in high energy particle accelerators. In addition, emerging technologies, such as improved energy storage, energy generation, and ultra-efficient motors, use niobium-titanium. A niobium-titanium alloy is even used in pipe and other forms in gold mining autoclaves, where corrosion and ignition of materials are a great concern

But niobium-titanium is exceptionally well suited for applications involved with superconductivity. Niobium superconducts at 9.3K, which is nearly twice the temperature for any other known element. In addition, its ductility is excellent, for making wire and other forms. Alloying niobium with titanium creates a superconducting material with pinning sites which prevent the magnetic flux (called fluxons) from moving. Flux motion causes energy dissipation and thereby a voltage to appear. Titanium also strengthens the alloy, without causing much loss in ductility. All of these properties add up to make niobium-titanium uniquely suited for fabricating superconducting rod and wire.

Wah Chang's Huntsville, Alabama site was designed specifically for that purpose. Its principal products include niobium-titanium alloy rod and niobium-titanium rivet-grade wire used in the aerospace industry. Wah Chang's protective surfaces on all machinery and handling equipment enable it to maintain high yields and unparalleled metallurgical and surface qualities.

The Alabama Facility makes numerous sizes of superconducting grade niobium-titanium rod dependent upon the needs of it's customers. Rivet grade niobium-titanium sizes include 0.248", 0.185", 0.1555", 0.1245", 0.093" diameter. The rivet grade wire is supplied in the unannealed condition with lubricant left on the material to help in the heading of rivets by the end user.

Huntsville's facilities include an 8F 4 die swage that can swage-point up to 4.0-inch diameter multifilamentary bar for drawing on its 200,000-pound drawbench. This drawbench is capable of processing bars up to 80 foot length with a capacity of 100,000 pounds per month. A second drawbench is capable of drawing nominal 2.4-inch diameter bars and lengths up to 180 feet. Additional equipment in Huntsville's rod processing department includes a continuous annealing furnace, an aging furnace, straightening equipment, and ultrasonic testing equipment.

The site's numerous bull blocks process material from 0.625 inches diameter to 0.015 inches in diameter.

Huntsville can anneal up to 2,000 pounds of material in its vacuum anneal furnace. After annealing, material is pickled in an acid cleaning facility, which includes 30-foot tanks for rods up to 28 feet long and tanks for pickling coils ~ 48 inches in diameter.

The applications for Wah Chang's niobium-titanium and other specialty metal products appear to be extensive. If you would like to discuss a potential application or would like more information on the Huntsville site capabilities, call Sales at 256-722-2304.

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NIOBIUM ALLOYS

Generally, Niobium alloys are used in applications where high strength is required at elevated temperatures. Like all reactive metals, this material oxidizes at elevated temperatures in air and must be protected by a coating. The type of coating may change with the temperature range or atmosphere encountered and should be discussed with us. The electrical properties of the material, e.g., superconductivity, are beyond the scope of these datasheets, but information on the types of alloys and fabrication methods is available on request. The niobium alloys are generally divided into three categories: low, medium, and high strength.

LOW STRENGTH

The standard of the industry is the niobium 1% zirconium alloy. It is used at temperatures of 1800-2200º F where ease of manufacture and low cost are critical or in reactor applications where the properties of the alloy are useful.

MEDIUM STRENGTH

These include C-103 (Nb-10 Hf-1 Ti) and C-129Y. C-103, which is probably the best all-around alloy on the market today, has a useful range of 1800-2400º F with good strength. Its combination of strength, excellent fabricability, and intermediate cost has made it a favorite choice of the aerospace industry.

HIGH STRENGTH

Where high strength with low density is required, two of the alloys to be considered are C-129Y and Cb-752. Although more difficult to fabricate and produce than the lower strength alloys, these alloys should be considered where higher stresses are encountered. C-129Y (Nb-10W-10Hf-.1Y) has the best welding characteristics of the high strength alloys. For even higher strength applications up to 2400º F, Wah Chang alloy C-3009 (Nb-3OHf-9W) should be considered. It must be remembered that, to obtain the higher strengths, fabricability and cost are, to an extent, sacrificed. The useful range of these alloys is 1800-3000º F.

PROPERTIES

Niobium is a soft, ductile metal which can be cold worked over 90 percent before annealing becomes necessary. In appearance, the metal is somewhat similar to stainless steel. The density of 8.57 gm/cc is moderate compared to the majority of the high melting point metals, being less than molybdenum at 10.2 gm/cc and only half that of tantalum at 16.6 gm/cc.

TABLE 1 - PROPERTIES of NIOBIUM (Nb)

Physical			
Melting Point	(°C)		2468
Boiling Point	(°C)		4927
Density	(g/cm^3)		8.57
Crystal Structure			Body Centered Cubic
Lattice Constant	Angstroms		3.30
Covalent Radius	Angstroms		1.34
Thermal Neutron Absorption Cross Section	(barns)		1.1
Electronegativity	(Pauling's)		1.6
Thermal Conductivity	@ 0° C J (sec cm °C)		0.523
	@ 1600° C J (sec cm °C)		0.691
Coefficient of Thermal Expansion	@ 20° C (x 10^{-6}/°C)		7.1
Electric Resistivity	(Microhm)		15
Temperature Coefficient	(x 10^{-3}/°C)		3.95
Volume Electric Conductivity	%IACS*		13.3
Specific Heat	@ 15°C (J/g)		.268
	@ 1227°C (J/g)		.320
Heat Capacity (Cp)	(J/mol °C)	0°C	24.9
		1200°C	29.7
		2700°C	33.5
Mechanical			
Modulus of Elasticity	(x 10^{-6} kg/cm^6)		1.05
Poisson's ratio			0.38
Hardness	(VHN)		60-100
Resistance to Thermal Shock			Good
Workability	ductile to brittle transition		-150°C
Recrystallization Temp.	GR I and II		900-1300 °C (1652-2372 °F)
	RRR Grade		750-850 °C (1382-1562 °F)
Stress Relieving Temp. (SRT)	GR I and II		800 °C (1472 °F)
	RRR Grade		649-663 °C (1200-1225 °F)

*International Annealed Copper Standard

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GENERAL CORROSION DATA

Niobium, like other reactive metals, owes its corrosion resistance to a readily formed, adherent, passive oxide film. Niobium's corrosion properties resemble those of tantalum, although it is slightly less resistant in aggressive media such as hot concentrated mineral acids. Table 2 gives some typical corrosion data for niobium. Like tantalum, it is susceptible to hydrogen embrittlement if cathodically polarized by either galvanic coupling or by impressed potential. In addition to being stable, the anodic niobium oxide film has a high dielectric constant and a high breakdown potential. These properties, coupled with its good electric conductivity, have led to niobium's use as a substrate for platinum-group metals in impressed-current cathodic-protection anodes.

TABLE 2 - CORROSION DATA of NIOBIUM in AQUEOUS MEDIA

Solutions	Concentration (Weight%)	Temp. (°C)	Corrosion Rate mm/y (mpy) ≤
Acid Solutions			
Hydrochloric Acid	1	boiling	nil
Hydrochloric Acid (aerated)	15	RT-60	nil
Hydrochloric Acid (aerated)	15	100	0.025 (1.0)
Hydrochloric Acid (aerated)	30	35	0.025 (1.0)
Hydrochloric Acid (aerated)	30	60	0.05 (2.0)
Hydrochloric Acid (aerated)	30	100	0.125 (5.0)
Hydrochloric Acid	37	RT	0.025 (1.0)
Hydrochloric Acid	37	60	0.25 (10)
Hydrochloric Acid	37% with Cl_2	60	0.5 (20)
Hydrochloric Acid	10% with 0.1% $FeCl_3$	boiling	0.025 (1.0)
Hydrochloric Acid	10% with 0.6% $FeCl_3$	boiling	0.125 (5.0)
Hydrochloric Acid	10% with 35% $FeCl_2$ and 2% $FeCl_3$	boiling	0.05 (2.0)
Hydrochloric Acid	80% with 20% HNO_3	30	0.025 (1.0)
Nitric Acid	30%+200 ppm F-1	80	0.125 (5.0)
Nitric Acid	30%+ 100 ppm F-1	80	0.125 (5.0)
Nitric Acid	30% +100 ppm F-1	80	0.025 (1.0)
Nitric Acid	50% + 50 ppm	80	nil
Nitric Acid	50	80	0.125 (5.0)
Nitric Acid	65	RT	nil
Nitric Acid	65	boiling	0.025 (1.0)
Nitric Acid	70% + 200 ppm F-1	80	0.5 (20)
Nitric Acid	70% + 50 ppm F-1	80	0.025 (1.0)
NitricAcid	70%+100 ppm F-1	80	0.125 (5.0)
Nitric Acid	70	250	0.025 (1.0)
Phosphoric Acid	50	30	nil
Phosphoric Acid	50	90	0.125 (5.0)
Phosphoric Acid	60	boiling	0.5 (20)
Phosphoric Acid	85	30	nil
Phosphoric Acid	85	RT	0.0025 (0.1)
Phosphoric Acid	85	88	0.05 (2.0)
Phosphoric Acid	85	100	0.125 (5.0)
Phosphoric Acid	85	155	3.75 (150)

TABLE 2 - CORROSION DATA of NIOBIUM in AQUEOUS MEDIA, CONT., P. 2

Solutions	Concentration (Weight%)	Temp. (°C)	Corrosion Rate mm/y (mpy)≤
Acid Solutions			
Phosphoric Acid	85	boiling	3.75 (150)
Phosphoric Acid	85% with 4% HNO$_3$	88	0.025 (1.0)
Phosphoric Acid	30% + 5 ppm F-1	boiling	0.25 (10)
Phosphoric Acid	40% + 5 ppm F-1	boiling	0.25 (10)
Phosphoric Acid	50% + 5 ppm F-1	boiling	0.5 (20)
Phosphoric Acid	68% with 3.5% HNO$_3$ and 5% acetic acid	45	0.125 (5.0)
Sulfuric Acid	5-40	RT	nil
Sulfuric Acid	25	100	0.125 (5.0)
Sulfuric Acid	98	RT	Embrittle
Sulfuric Acid	10	boiling	0.125 (5.0)
Sulfuric Acid	25% + 200 ppm Cl-1 +5ppm F-1	boiling	0.25 (10)
Sulfuric Acid	40	boiling	0.5 (20)
Sulfuric Add	40% with 2% FeCl$_3$	boiling	0.25 (10)
Sulfuric Acid	60	boiling	1.25 (50)
Sulfuric Acid	60% with 01-1% FeCl$_3$	boiling	0.5 (20)
Sulfuric Acid	20% with 7% HCl +50 ppm F-1	boiling	0.5 (20)
Sulfuric Acid	20% with 7% HCl +100 ppm F-1	boiling	0.5 (20)
Sulfuric Acid	20% with 7% HCl and 100 ppm F-	boiling	0.25 (10)
Sulfuric Acid	50% with 20 HNO$_3$	50-80	nil
Sulfuric Acid	50% with 20% HNO$_3$	boiling	0.25 (10)
Sulfuric Acid	60	90	0.05 (2.0)
Sulfuric Acid	65	153	2.5 (100)
Sulfuric Acid	70% + 200 ppm CuCl$_2$	158	>5.0 (200)
Sulfuric Acid	70% + 1000 ppm CuCl$_2$	153	2.5 (100)
Sulfuric Acid	70% + 10000 ppm CuCl$_2$	154	2.5 (100)
Sulfuric Acid	70	167	5.0 (200)
Sulfuric Acid	70% with 15% Cr+3	100	nil
Sulfuric Acid	70% with 15% Cr+3	125	0.125 (5.0)
Sulfuric Acid	70% with 15% Cr+3	boiling	3.75 (150)
Sulfuric Acid	72% + 3% CrO$_3$	100	0.025 (1.0)
Sulfuric Acid	72% + 3% CrO$_3$	125	0.125 (5.0)
Sulfuric Acid	72% + 3% CrO$_3$	boiling	3.75 (150)

TABLE 2 - CORROSION DATA of NIOBIUM in AQUEOUS MEDIA, CONT., P. 3

Solutions	Concentration (Weight%)	Temp. (°C)	Corrosion Rate mm/y (mpy)≤
Organic Acid			
Acetic Acid	5-99.7	boiling	nil
Acetic Acid	50% with 50% Acetic Anhydride	boiling	0.025 (1.0)
Acid Solutions			
Citric Acid	10	boiling	0.025 (1.0)
Formaldehyde	6	boiling	nil
Formaldehyde	37	boiling	0.0025 (0.1)
Formic Acid	10	boiling	nil
Formic Acid	50	boiling	0.025 (1.0)
Lactic Acid	10-85	boiling	0.025 (1.0)
Oxalic Acid	10	boiling	1.25 (50)
Tartaric Acid	20	RT-boiling	nil
Trichloroacetic	50	boiling	nil
Trichloroethylene	99	boiling	nil
Alkaline			
NaOH	1-40	RT	0.125 (5.0)
NaOH	1-10	98	Embrittle
KOH	5-40	RT	Embrittle
KOH	1-5	98	Embrittle
NH_4OH		RT	nil
Salts			
$AlCl_3$	25	boiling	0.005 (0.2)
$Al_2(SO_4)_3$	25	boiling	nil
$AlK(SO_4)_2$	10	boiling	nil
$CaCl_2$	70	boiling	nil
$CaCl_2$	.2% with .1% $MgCl_2$ +200 ppm NaF +100 ppm CaF_2	80	0.025 (1.0)
$CaCl_2$	.2% with .1% $MgCl_2$	80	0.25 (10)
$CaCl_2$	2% with 1% $MgCl_2$ + 200 ppm NaF + 2800 CaF_2	80	0.025 (1.0)
$CaCl_2$	2% with 1% $MgCl_2$	80	0.125(5.0)
$CaCl_2$	6.6% with 3.3% $MgCl_2$ + 200 ppm NaF + 9800 ppm CaF_2	80	0.025 (1.0)
$CaCl_2$	6.6% with 3.3% $MgCl_2$	80	0.025 (1.0)
$Cu(NO_3)_2$	40	boiling	nil
$CuSO_4$	40	104	0.025 (1.0)

TABLE 2 - CORROSION DATA of NIOBIUM in AQUEOUS MEDIA, CONT., P. 4

$FeCl_3$	10	RT-boiling	nil
Fe+3	12% with 6 HCl	66	nil
Solutions	Concentration (Weight%)	Temp. (^{o}C)	Corrosion Rate mm/y (mpy)≤
$HgCl_2$	saturated	boiling	0.0025 (0.1)
H_2SiF_6	10	90	5.0 (200)
HSO_3NH_2	10	boiling	0.025 (1.0)
K_2CO_3	1-10	RT	0.025(1.0)
K_2CO_3	10-20	98	Embrittle
K_3PO_4	10	RT	0.025 (1.0)
$MgCl_2$	47	boiling	0.025 (1.0)
NaCl	saturated and pH =1	boiling	0.025 (1.0)
NaCl	5% with 0.5% Acetic Acid	100	nil
Na_2CO_3	10	RT	0.025 (1.0)
Na_2CO_3	10	boiling	0.5 (20)
Na_2HSO_4	40	boiling	0.125 (5.0)
NaOCl	6	50	1.25 (50)
Na_3PO_4	5-10	RT	0.025 (1.0)
Na_3PO_4	2.5	98	Embrittle
NH_4Cl	40	boiling	0.025 (1.0)
NH_2SO_3H	10	boiling	0.025 (1.0)
$NiCl_2$	30	boiling	nil
$Ni(NO_3)_2$	40	104	0.025 (1.0)
$ZnCl_2$	40-70	boiling	nil
$ZrCl_4$	70	boiling	nil
$ZrCl_4$	88	boiling	nil
Miscellaneous			
Bromine	liquid	20	nil
Bromine	vapor	20	0.025 (1.0)
Chrome Plating	25% CrO_3, 12% H_2SO_4	92	0.125 (50)
Seawater	natural	boiling	nil
Chrome Plating	17% CrO_3, 2% Na_2SiF_6, 92 trace H_2SO_4		0.125 (5.0)
Solution	H_2O		
H_2O_2	30	RT	0.025(1.0)
H_2O_2	30	boiling	0.5 (20)

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GENERAL CORROSION DATA

ACID SOLUTIONS

Niobium is resistant to most organic and mineral acids at all concentrations below 100° C, except hydrofluoric acid. This list of acids includes the halogen acids (hydrochloric, hydroiodic and hydrobromic), nitric acid, sulfuric acid, and phosphoric acid. It is especially resistant under oxidizing conditions such as: concentrated sulfuric acid and ferric chloride or cupric chloride solutions. Niobium is completely resistant in nitric acid, having a corrosion rate of 0.025 mm/y (1 mpy) in 70% nitric acid at 250° C. It is completely resistant in 20% sulfuric acid at 100° C. In concentrated sulfuric acid, at the same temperature, it has a corrosion rate of 0.25 mm/y (10 mpy). In chrome plating solutions, niobium experiences only a slight weight change, and in the presence of small amounts of fluoride catalyst, it exceeds the corrosion resistance of tantalum.

Niobium is inert in mixtures of nitric acid and hydrochloric acid. It has a corrosion rate of less than 0.025 mm/y (1 mpy) in aqua regia at 55 °C. In boiling 40% and 50% phosphoric acid with small amounts of fluoride ion impurity (5 ppm), niobium has a corrosion rate of 0.25 mm/y (10 mpy). In mixtures of nitric acid and sulfuric acid, niobium dissolves readily.

ALKALINE SOLUTIONS

In ambient aqueous alkaline solutions, niobium has corrosion rates of less than 0.025 mm/y (1 mpy). At higher temperatures, even though the corrosion rate does not seem excessive, niobium is embrittled even at low concentrations (5%) of sodium hydroxide and potassium hydroxide. Like tantalum, niobium is embrittled in salts that hydrolyze to form alkaline solutions. These salts include sodium and potassium carbonates and phosphates.

SALT SOLUTIONS

Niobium has excellent corrosion resistance in salt solutions, except those that hydrolyze to form alkalis. It is resistant to chloride solutions even with oxidizing agents present. It does not corrode in 10% ferric chloride at room temperature, and it is resistant to attack in sea water. Niobium exhibits resistance similar to tantalum in salt solutions.

GASES

Niobium is easily oxidized. It will oxidize in air above 200° C. The reaction, however, does not become rapid until above red heat (about 500° C). At 980° C, the oxidation rate is 0.025 mm/y (17,000 mpy.) The attack is catastrophic at 390° C in pure oxygen which freely diffuses through the metal causing embrittlement. Niobium reacts with nitrogen above 350° C; with water vapor above 300° C; with chlorine above 200° C; and with carbon dioxide, carbon monoxide and hydrogen above 250° C. At 100° C, niobium is inert in most common gases, e.g., bromine, chlorine, nitrogen, hydrogen, oxygen, carbon dioxide, carbon monoxide, and sulfur dioxide (wet or dry).

GENERAL CORROSION DATA, CONT., P. 2

LIQUID METALS

Niobium is resistant to attack in many liquid metals to relatively high temperatures. These include bismuth below 510º C; gallium below 400º C; lead below 850º C; lithium below 1000º C; mercury below 600º C; sodium, potassium, and sodium-potassium alloys below 1000º C; thorium-magnesium eutectic below 850º C; uranium below 1400º C; and zinc below 450º C. The presence of excessive amounts of gas impurities may reduce niobium's resistance to these liquid metals.

Since liquid metals are excellent heat-transfer media, they can be used in very compact thermal systems, such as fast breeder reactors, reactors for space vehicles, and fusion reactors.

Niobium resists attack both by sodium vapor at high temperatures and pressures. The Nb-1% Zr alloy is in use as the end caps on high pressure sodium vapor lamps.

GALVANIC EFFECTS

If niobium is polarized cathodically either by galvanic coupling or chemical attack, it can be destroyed by hydrogen embrittlement.

If niobium is polarized anodically, however, it forms a very stable, passive film which protects the metal from corrosion. The stability of the passive film, combined with good electrical conductivity (13% that of copper) and good mechanical properties, has led to the use of niobium as a substrate for platinized anodes used in cathodic protection systems. Niobium's anodic breakdown potential in chloride solutions is about 115 V compared to 10 V for titanium. Platinized niobium anodes are used in high resistivity waters and other environments requiring high driving potential to obtain good current distribution. In this application, niobium has an advantage over tantalum, because it is less expensive. This cost advantage is further enhanced by using a composite electrode with a copper core, which increases the conductivity of the anodes.

SUMMARY

Niobium's corrosion properties are similar to those of tantalum; however, it is less expensive and should be considered in all applications requiring tantalum. Niobium has replaced tantalum in some hydrochloric acid applications.

CORROSION TESTING

Wah Chang has a well-equipped corrosion testing laboratory designed to generate new corrosion data on our products and to assist in customer evaluations of media and materials. This laboratory is available for immersion, electrochemical, high temperature autoclave, and stress corrosion testing. Velocity effects can also be studied. Testing equipment includes electrochemical systems for

- cyclic polarization
- linear polarization
- galvanic coupling and AC impedance studies
- a slow strain rate Stress Corrosion Cracking (SCC) testing machine
- autoclaves capable of withstanding severe corrosives at pressures to 500º psi and temperatures to 250º C
- sufficient immersion testing equipment to perform 200 simultaneous immersion tests.

Diagnostic equipment includes a Zeiss metallograph, a scanning electron microscope with EDAX capabilities, an electron microprobe and the services of our Analytical Laboratory, one of the finest in the world. On-site analytical capabilities include:

- emission
- mass and atomic absorption spectroscopy
- xray fluorescence diffraction
- classical wet chemistry and interstitial gas analysis.

Wah Chang also offers complimentary corrosion coupons for customer laboratory or in-plant testing.

CORROSION TESTING of NIOBIUM in HCL

The recent jump in tantalum pricing for the CPI has many end users asking about replacement materials. This has caused us to reassess the corrosion resistance of niobium in media where Ta is used extensively. Initially the effort has been aimed at hydrochloric acid media. In general, niobium is similar to both Ta and Ti since it is more resistant to oxidizing media. But as with Ta and Ti, Nb can absorb hydrogen from the corrosion process and become embrittled, even at relatively low temperatures. It is particularly susceptible to hydrogen pickup in alkaline solutions. Table 1 compares Nb's corrosion resistance to Zr, Ta, and Ti in various media.

Other aspects of niobium that apply to its use in corrosive applications include:

- Resistant to most organic and mineral acids below 100ºC
- Good in nitric acid to 250ºC
- Good in chrome plating baths
- Inert to aqua regia at 55ºC
- Low levels of fluoride ion improves resistance in phosphoric acid
- Resistant in alkalis, but can absorb hydrogen and become embrittled
- Inert to 10% Ferric Chloride at ambient temperature
- Oxidizes in air above 200ºC
- Nitrides above 350ºC
- Resists water vapor to 300ºC
- Resists chlorine gas to 200ºC
- Resists carbon monoxide and dioxide, and hydrogen gas to 250ºC

TABLE 1

Media	Niobium	Zirconium	Tantalum	Titanium
HCl	Fair	Excellent	Excellent	Poor
H_2SO_4	Fair	Very Good	Excellent	Poor
Oxidizing without Cl^-	Excellent	Excellent	Excellent	Excellent
Oxidizing with Cl^-	Excellent	Poor	Excellent	Excellent
HNO_3	Excellent	Excellent	Excellent	Very Good
H_2O_2	Good	Excellent	Excellent	Good
Acid with F^-	Good	Poor	Poor	Poor
Acetic Acid	Excellent	Excellent	Excellent	Excellent
NaOH	Embrittled	Very Good	Embrittled	Embrittled

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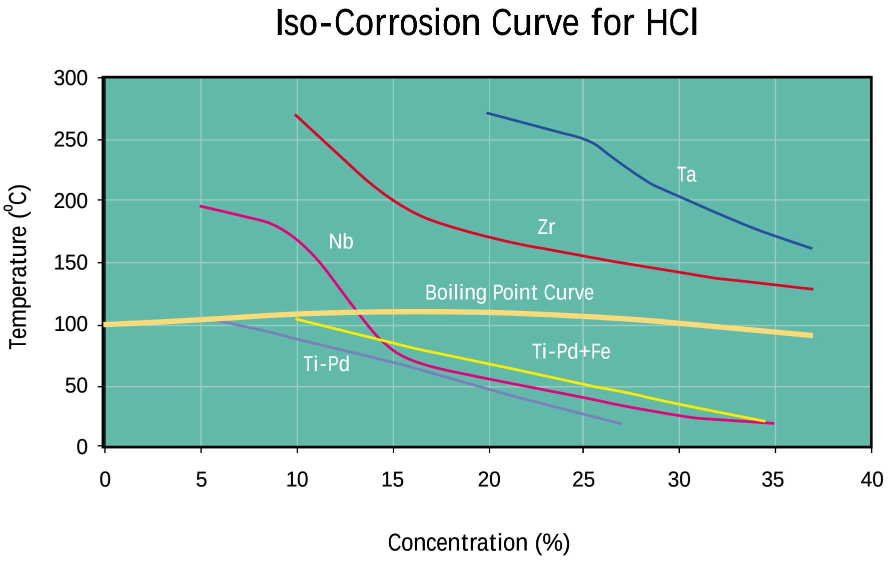
CORROSION TESTING of NIOBIUM in HCL, CONT., P. 2

Figure 1 contains 5 mpy (mil per year corrosion rate) iso-corrosion curves for Nb, Zr, Ta, and the Ti-Pd alloy (tailored for oxidizing conditions).

As you can see, the resistance of niobium in pure HCl is good to above the boiling point up to about 13% concentration. At higher concentrations, the 5-mpy line drops gradually to 50ºC. The Ti-Pd alloy is useful in pure acid to boiling condition at concentrations less than 5%. Its 5-mpy curve drops to near room temperature at just over 25% concentration. These curves for Zr and Ta, on the other hand, remain above the boiling point curve at all concentrations.

The current testing program was aimed at determining the gain in corrosion resistance of Nb by adding an oxidizer (ferric ion) to the HCl acid.

FIGURE 1



CORROSION TESTING of NIOBIUM in HCL, CONT., P. 3

TABLE 3 - CORROSION RESISTANCE of Nb

			Corrosion Rate - mils per year (mpy)	
HCl %	Ferric ppm	Temperature ^{0}C	Nb	Nb IZr
15	500	60	.1	.1
15	1000	60	.1	.1
15	1500	60	.2	.1
15	500	100	.4	.4
15	1000	100	.6	.6
15	1500	100	.4	.4
15	15%	93	.2	.3
20	500	60	1.8	1.9
20	1000	60	2.3	2.7
20	1500	60	1.2	1.2
20	500	100	2.2	1.1
20	1000	100	2.2	3.6
20	1500	100	2.6	2.5
20	15%	93	.2	.3
20	2%	93	2.1	2.1
25	500	60	.3	.2
25	1000	60	.2	.2
25	1500	60	.2	.2
25	500	100	12	12.4
25	1000	100	8.5	9
25	1500	100	13	12

The presence of the ferric ion improves Nb's corrosion resistance to higher temperatures. For example, Nb in pure 15% HCl at 100°C corrodes at a rate >20 mpy (from Figure 1). With 500 ppm ferric, the corrosion rate drops below 1 mpy. Even in 20% acid at 100°C, the corrosion rate drops from >50 mpy to <3 mpy. The 5-mpy iso-corrosion curve for the Ti-Pd alloy in HC1 with 125 ppm ferric lies just below the Nb curve in Figure 1. The Ti-Pd curve (with 125 ppm ferric) drops rapidly to room temperature at just over 30% acid, while the Nb line remains above 50°C.

We plan to conduct similar tests at concentrations to 37% acid, since it is expected that the ferric additions will raise the Nb curve in this region to near the boiling point curve.

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GENERAL WORKING CHARACTERISTICS

Two unusual features affect the working characteristics of niobium. First, no appreciable softening occurs below 400° C, the temperature at which niobium reacts vigorously with the atmosphere, because of its high melting point—approximately 2400° C. Secondly, sheathing for protection is not practical, because the sheathing material is likely to be softer that the metal to be worked.

Fortunately, niobium's cold working properties are excellent, and the metal can be easily forged, rolled, or swaged directly from the ingot at room temperature. After the cross-sectional area has been reduced by about 90 percent, annealing is necessary. Heat treatment at 1200° C for one hour causes complete recrystallization of material cold worked over 50 percent. The annealing process must be performed in an inert gas or in a high vacuum at pressures below 1 x 10 -4 Torr. The use of a vacuum is preferred, because it is difficult to ensure the purity of inert gas. It is also less expensive to use vacuum furnaces for these operations.

Niobium is well suited to deep drawing, and the metal may be cupped and drawn to tube, although special care must be taken with lubrication. The sheet metal can be formed easily by general sheet metal working techniques. The low rate of work hardening facilitates these operations by reducing springback.

MACHINING

Niobium may be machined by the usual techniques although, due to the tendency of the material to gall, special attention should be paid to tool angles and lubrication.

TURNING

Lathe turning is best carried out with High Speed Steel tools, using air, soluble oil, Rapid-Tap, or other suitable products for cooling and lubricating. This material turns very much like lead or soft copper. It must be sheared and the chip allowed to slide off the tool surface. If any buildup of the material is allowed, the pressure will break the cutting edge and ruin the tool. Carbide tooling should be used only for fast, light cuts, with a depth of .010 to .015 inches, to work efficiently. Tooling recommendations are given in Table 3, which contains data applying to High Speed Steel and Carbide tools.

Contact Wah Chang for special tooling information and technical assistance.

TABLE 3 - TOOLING RECOMMENDATIONS for MACHINING Nb

Approach Angle	15° to 20°
Side Rake	30° to 35°
Side and End Clearance	5°
Plan Relief Angle	15° to 20°
Nose Radius	0.020" to 0.030"
Cutting Speed	60 to 80 feet/minute with HSS
	250 to 300 feet/minute with carbide
Feed, Roughing	0.008" to 0.01 2"/revolution
Feed, Finishing	0.005" maximum/revolution
Depth of Cut	0.030" to 0.125"

MACHINING, CONT., P. 2

DRILLING

Standard High Speed drills, ground normal angles, may be used, but the peripheral lands wear badly and care must be taken to see that the drill has not worn undersize.

SCREW CUTTING

Provided that plenty of lubricant is used, niobium may be screw-cut using a standard die-cutting head. The use of ample lubricant prevents galling on the die and consequent tearing of the thread. Roll threading is a preferred method.

SPINNING

Normal techniques of metal spinning may be applied successfully to niobium, with minor modifications. It is generally better to work the metal in stages; for example, when spinning a right-angled cup from flat sheet, several formers should be used to give steps of approximately 10º. Wooden formers may be used for rough spinning, but a brass or bronze former is essential for finishing, because the metal is soft and takes up the contour of the former. For small work, aluminum bronze or Narite tools should be used with a radius of approximately 3/8 inch. If sharp angles are required, the tool must be shaped accordingly. Yellow soap, or tallow, is suitable for lubricating the material, which must be cold worked continually. The peripheral speed of the work-piece should be about 500 feet per minute. When spinning, niobium is prone to "thinning" and care must be taken to avoid this. The tool should be worked in many long sweeping strokes using a light pressure rather than a few heavy strokes.

WELDING

Niobium is a highly active metal, reacting to temperatures well below its melting point with all the common gases, such as: nitrogen, oxygen, hydrogen, and carbon dioxide. At the melting point and above, niobium will react with all known fluxes, thus severely restricting the choice of welding methods. Cleanliness of the metal prior to welding is critical. An acid pickle is recommended before welding. For ambient temperature pickling, a typical solution would be 25-30% HF, 25-33% HNO_2, balance H_20. Coupons should be used prior to immersing the part to check the etchant rate. Removal of approximately 0.0001" is generally accepted.

FUSION WELDING

The TIG welding method is recommended for niobium. This process is commonly applied to the welding of stainless steel, aluminum, magnesium, and their alloys. It is now being used to weld reactive metals, such as titanium, zirconium and tantalum. However, some modifications in this technique are required. It is essential to completely cover the area of the molten pool and the heated zone with inert gas to avoid contamination on the weld metal. This protection must be given to the back of the weld as well as to the face. When welding butt joints without filler rod in sheet less than 0.050", the torch provides sufficient coverage to the face of the weld; the back of the weld may be protected with a stream of argon from a manifold positioned just below the weld bead. A trailing shield will afford further protection to the hot metal after the main shield has passed.

Niobium can be welded satisfactorily by using standard gas-tungsten-arc (GAT), heli-arc procedures. The resulting welds are superior to those made under similar conditions with an alternating current. The argon from the torch seems to provide better protection for a smaller pool.

WELDING, CONT., P. 2

(Fusion welding, cont.) As mentioned earlier, metal exposed to air at reaction temperatures acquires a relatively thick, adherent oxide film which is extremely difficult to remove. Upon vacuum annealing, the oxide film is diffused rapidly into the metal which results in hardening of the weld bead and the heat affected zone. An equilibrium oxide film is present on the metal at room temperature, but it is relatively thin and causes no significant hardening of the metal upon annealing except in the case of extremely fine wire or foil.

Niobium is amenable to machine welding. Faster speeds ensure minimum weld contamination from the atmosphere.

If sheet greater that 0.050" thick is to be welded, the current required for full penetration becomes high enough to cause a spread of the molten pool outside the protection of the argon shield. Similarly, when welding with filler rod, the molten pool is too large. Further complications arise when the filler rod becomes heated at a distance from the arc and outside the protection of the argon shield. If it oxidizes, this contaminated metal is melted into the weld bead. The solution is to provide complete protection by using an argon-filled box. The box is constructed so that the leads of the torch are sealed through one wall—allowing the torch to be operated from the outside of the box, using rubber gloves. The rubber gloves are blanked-off and the air is evacuated from the box and back filled with positive pressure argon or helium. The box is vacuum tight and contains a vacuum pump to assist in argon flushing.

Contamination-free welds can be produced under totally inert atmospheres as compared to welds produced employing inert shielding only.

In a typical niobium fusion weld, there is a gradual transition from a work-hardened sheet structure to the annealed structure of the heat affected zone and, finally, to the large grains typical of the cast metal.

Niobium's corrosion resistance has proved beneficial in applications where welded material is used. Simple tests, carried out under conditions known to cause attack on the parent metal, have shown the weld to be as resistant to corrosion as the parent metal.

The TIG spot welding technique is suitable for spot welding niobium. This torch design employs an argon shield which provides complete protection of the molten zone. Because complete penetration is not desirable, no backup argon is required. For niobium, the applications for this technique are somewhat limited.

RESISTANCE WELDING

While TIG welding is the preferred method for welding niobium sheet in thicknesses of 0.020" or greater, the method is somewhat limited for thinner sheet. It is possible to weld sheet as thin as 0.012" or even thinner, but special attention must be paid to the shape of the electrode tip. Extremely careful jigging is essential for accurate alignment and prevention of distortion and misalignment during welding. For sheet thinner than 0.020", it is generally better to use the resistance method of welding.

The problem of contamination during resistance welding is not as great as in TIG welding, because the duration of the weld can be kept short. Spot welding may be carried out in air providing the weld time is restricted to one or two cycles, but welding should be carried out under water. The water does not protect the weld from contamination in the same way as the argon shield does during arc welding. Its function is to remove heat from the weld as quickly as possible—thus keeping the time that the metal is hot to a minimum.

Either transformer or stored-energy type equipment can be used, but the welding heads should be of the low-inertia type so that, as the welds are made, proper pressure will be maintained throughout the welding cycle. The surfaces to be welded should be cleaned and degreased before welding. Any copper pick-up from the electrode, which contaminates the sheet after welding, may be removed by pickling in nitric acid.

NIOBIUM-TITANIUM ALLOYS

Wah Chang, with a capacity of 100,000 lbs./month, has been producing ingots of niobium/45%-55% titanium alloy for over 25 years. In recent years, various programs have been outlined for large superconducting devices which will require thousands of pounds of fine composite wire. The alloy is fabricated in all forms, although it is generally used in multifilamentary cables. Quantities of the various alloys and types are generally available.

The alloys are manufactured in both Grade 1 and Grade 2 types. The primary difference is that the Grade 2 material has a higher allowable tantalum content which has no effect on the superconducting properties.

One of the major improvements in Nb/Ti superconducting alloys has been the development of high homogeneity (Hi-Ho) grade alloys, which have improved the current density of the final material, while permitting smaller diameters to be drawn with less fabrication difficulties. While not intended for all applications, Hi-Ho grade is necessary for 10-micron sized filaments.

Wah Chang also manufactures copper clad Nb/Ti alloys which can be made with or without a barrier of vanadium or niobium. This monofilamentary material can be furnished as extruded rod or drawn to whatever final size is specified.

A special high purity grade of niobium, known as Residual Resistivity Ratio (RRR), has been developed for use in radio frequency (RF) accelerator cavities. The RRR is the ratio of the resistance of Nb at room temperature to that at 4.2 Kelvin in a magnetic field of 1.7 Teslas. Ratios of 250 and up are routinely produced at Wah Chang in all the standard forms and shapes.

The niobium/55% titanium alloy is also used in the aircraft industry for rivets. The alloy requires approximately one quarter of the bucking force necessary to head a CP titanium rivet of the same size. The Nb/55% Ti alloy, as specified in AMS 4982, is stronger than CP titanium at temperatures in excess of 600º F. Material can be manufactured into all rivet shapes and forms, including the composite types.

For more detailed information regarding either the superconducting Nb/Ti alloys or the rivet grade material, contact Wah Chang.

C-103 ALLOY (Nb-10 Hf-1 Ti)

Refractory metals capable of withstanding high-stress levels at elevated temperatures have been developed to meet the needs of high-performance, lightweight aerospace propulsion systems. The metals which best demonstrate this are the niobium based alloys. C-103 (Nb-10 Hf-1 Ti) was selected to satisfy initial design requirements because of its excellent fabrication capabilities. It is the most "forgivable" niobium alloy for welding and shape forming. Although it is considered a first generation alloy (succeeding Nb1 Zr), C-103 was developed to replace the weaker alloys, while retaining the desirable forming and welding properties. This niobium-hafnium-titanium alloy satisfies most rocket engine requirements for temperatures up to 2700° F.

Extensive testing has shown that niobium's reliability has greatly reduced program costs. Niobium-based alloys offer tremendous weight savings over many other rocket engine design materials. Consequently, they are being used, in many different fabricated forms, in most major aerospace programs.

TABLE 4 MECHANICAL PROPERTIES

Density	0.320 lbs/in^3 or 8.85 gm/cm^3
Melting Point	2350 ±50°C, 4260 ± 90°F
Thermal Expansion	$8.73 \pm 0.09 \times 10^{-6} (\frac{CM}{CM})°C^{-1}$
Specific Heat	0.832 BTU/°F/lb

TABLE 5 PHYSICAL PROPERTIES

Bend Ductility:	C-103 sheet will pass a - 320°F, 90°, 2T bend in both parent metal and weldment	
Modulus of Elasticity	Temperature °F	PSI
	Room Temperature	13.1×10^6
	2200	9.3×10^6

C-103 ALLOY (Nb-10 Hf-1 Ti), CONT., P. 2

C-103 is fully recrystallized by heat treating one to two hours at 2400° F with more than 80% cold work.
C-103 products include:

- Sheet .030" thick, grain size ASTM 6 minimum
- Plate 1-1/2" thick, extruded, and rolled, grain size ASTM 4-6
- Round bar 3-1/4" diameter, forged and machined, grain size ASTM 3-5

Hardness: VAR Ingot 130-145 BHN
Recrystallized product: 130-145 DPH

C-103 alloy can be easily fabricated and worked. Forging, rolling, and swaging are accomplished by general sheet metal working methods; in fact, the low rate of work hardening facilitates these operations by reducing spring-back. C-103 may be machined by the usual techniques, although the surface galling tendency makes it similar to lead or soft copper.

TIG is the best welding method. Electron beam welding is also excellent, but the necessary equipment is not always available. For TIG, properly designed trailer shields and backup grooves with adequate gas flow can produce welds of a quality equal to those made with electron beam equipment.

MECHANICAL PROPERTIES

Figures 1 and 2 contain mechanical properties data that has been used successfully as design criteria for over 20 years, while providing necessary safety margins.

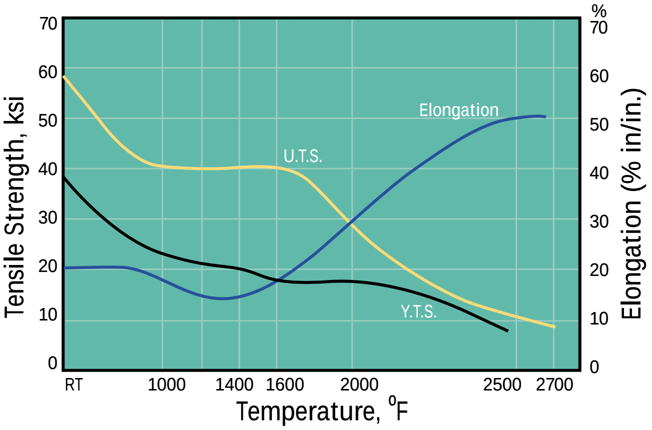


Figure 1. Guaranteed mechanical properties of C-103 recrystallized one hour at 2400°F.

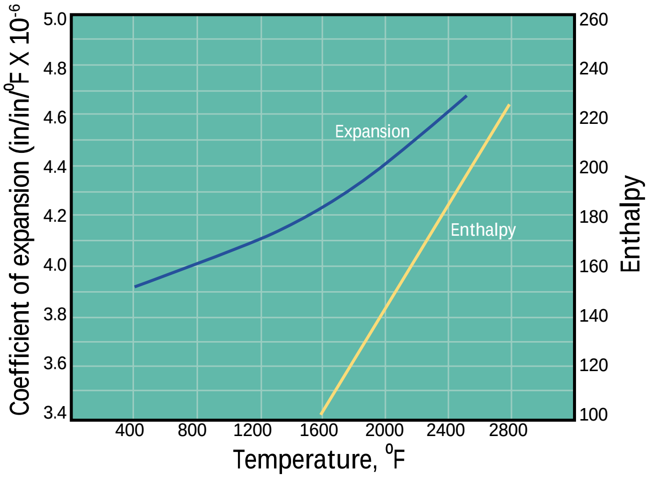


Figure 2. Linear coefficient of thermal expansion and enthalpy

C-103 ALLOY (Nb-10 Hf-1 Ti), CONT., P. 3

WELDING

A pickling operation should be conducted before and after welding as well as before and after heat treating. If pickling is performed, welding of niobium alloys can be readily accomplished by both the TIG and electron beam processes. It is recommended that all copper tooling should have a hard chrome plating approximately 0.002" thick without a nickel strike. This prevents copper contamination which causes brittle welds. For TIG, properly designed trailer shields and backup grooves with adequate gas flow can give weldments a quality equal to welds made with electron beam equipment. Where possible, the argon and helium should be passed through hot zirconium chips, maintained at 800º F to gather minute impurities in the gas. Stake welds of any length can be made without mismatch or burn-out. Girth welds by the TIG process have been made up to 54", matching .010" to .020" material. Welding of C-103 sheet is often accomplished without weld wire. Material over 0.100" thick should be welded by electron beam process.

Generally, weld wire is not used because of the high cleanliness requirements for welding. The weld wire often has slight serrations and laps that are difficult to clean. Sheet is welded within two hours after pickling, and it is not possible to pickle a roll of welding wire each time it is used.

In addition, the high melting point of niobium causes the wire to deflect and give an uneven weld bead and incomplete penetration. To eliminate these problems and still get a weld with an adequate crown and root, the mating edges to be welded are rolled up to form a "burned-down flange". The material is then welded with back-up gas and a trailer shield at a very high welding speed. The gases are a combination of helium and argon. TIG welding of second generation niobium alloy, C-129Y to C-103 has been very successful.

TABLE 6 - MECHANICAL PROPERTIES OF BARE METAL TIG WELDMENTS (C-103 TO C-103)

Specimen	Thickness in inches	Condition	UTS psi	YTS psi	% Elongation in 1 inch
1	.030 to .030	Annealed	59,500	40,400	27.0
2	.020 to .020	As Welded	61,700	44,900	14.0
3	.020 to .020	Annealed	60,200	42,100	16.0
4	.020 to .020	As Welded	62,400	46,500	13.0
5	.020 to .020	Annealed	59,600	41,400	18.0
6	.020 to .030	As Welded	61,100	46,200	13.0
7	.020 to .030	As Welded	61,600	45,200	14.0
8	.020 to .030	Annealed	60,200	42,200	14.0
9	.020 to .030	Annealed	60,400	42,700	13.0
10	.030 to .030	As Welded	61,900	45,500	20.0
11	.020 to .030	Annealed	60,000	40,700	25.0
12	.030 to .030	As Welded	61,000	46,900	20.0

Note: All above anneals one hour at 2200ºF

Nb-1 Zr ALLOY

SCOPE

It was discovered that the addition of 1% zirconium to niobium greatly improved the creep strength over the soft pure metal. Thus Nb-1Zr became the replacement for pure niobium in applications requiring the chemical resistance of niobium and a material with high melting temperature. In addition to greatly increasing the strength of pure niobium at higher temperatures, Nb-1Zr also has low thermal nuclear capture cross-section properties. Therefore, this alloy has been closely associated with the nuclear industry, which requires specified elevated temperature strength in the range of 1800º F to 2200º F. Because of the increasing need for better strengths, as technology has advanced, Nb-1Zr has been replaced by alloys such as C-103, which has greater strength and thus improved reliability, but still retains all the desirable characteristics of Nb-1Zr. Nb-1Zr has the advantage of being less expensive than the higher strength alloys, and can be used in applications where a high-temperature material is needed with low loads such as a loadfree thermal shield. Due to the excellent fabricability and ductility, this material is readily available in all desired mill product forms.

TABLE 7 - PHYSICAL PROPERTIES of Nb-1Zr

Density	0.31 lbs./cu. in. or 8.57 gms./cu. cm.	
Melting Point	4365 ± 15ºF or 2410 ± 10ºC	
Thermal Conductivity	24.2 BTu/(hr.) (ft.)$_2$ (ºF/ft.) (25ºC)	
Specific Heat	0.065 BTu/ºF/lb. (70ºF)	
Emissivity	Total Hemispherical Emittance	
	TemperatureºC	WC-1 Zr
	500	0.103
	600	0.110
	700	0.117
	800	0.130
	900	0.142
	1000	0.154
	1100	0.167
	1200	0.179

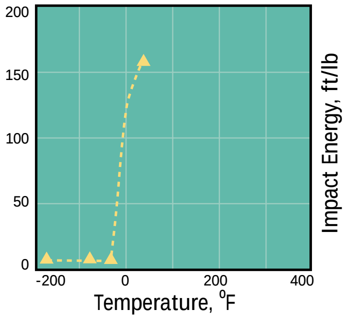
TABLE 8 - MECHANICAL PROPERTIES of Nb-1Zr

Bend Ductility

Nb-1 Zr recrystallized sheet will pass a -320º F, 90º, 2T bend in both parent metal and weldment.

Modulus of Elasticity

10 x 10^6 psi (estimated) at room temperature.



Charpy Impact Data, Nb-1Zr, recrystallized

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TABLE 9 - METALLURGICAL PROPERTIES of Nb-1Zr

Grain Size	
1. Sheet and strip	ASTM avg. 6
2. Plate	ASTM avg. 5
3. Rod <2 1/2" diameter	ASTM avg. 4
4. Billet > 2 1/2" diameter	ASTM avg. 3

METALLOGRAPHY

It is typical of niobium alloys to display “stringers" in heavily worked material. These stringers result from interstitial impurities which occur predominately in the grain boundaries during casting. In subsequent fabrication, these impurities are stretched along the work direction and eventually form very fine intermetallic compounds which show up after recrystallization annealing as peppery areas.

CHEMICALS

Wah Chang also has a large chemical products department which manufactures the major chemical products in the primary metals produced at this facility. These made-to-special-order chemicals include:

- Niobium Carbide
- Niobium Hydride
- Niobium Oxide
- Tantalum/Niobium Carbide

We would be happy to review your requirements for any chemicals in the refractory metal line.

ALLOY ADDITIONS

Wah Chang manufactures a variety of material for alloy additions such as vacuum grade ferro niobium, nickel niobium, and pure material of niobium.

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NIOBIUM and Nb ALLOY PLATE, SHEET and STRIP

1. Scope

1.1 Scope. This specification establishes the requirements for niobium alloy plates, sheets and strip.

1.2 Classification. Niobium alloy plates, sheet, and strip procured to this specification shall be supplied in the following

Type	Composition
Nb	Nb
C-103	Nb-lOHf-1Ti
C-129Y	Nb-lOHf-1OW-.1Y
Nb1Zr	Nb-1Zr
Cb-752	Nb- 1OW-2.5Zr

2. Applicable Documents

The following documents of the issue in effect on the date of invitation for bids or request for proposal form a part of this specification to the extent specified herein.

STANDARDS

- *Federal* - Federal Test Method Standard No.151. MIL-STD-271. Metals, Test Methods, Non-Destructive Testing (X-Ray, Sonics, Dye Penetrant)
- *American Society for Testing Materials*. ASTM E-12. Methods for Determining Average Grain Size
- *National Academy of Science*, Material Advisory Board, MAB-216-M. Evaluation Test Methods for Refractory Metal Sheet Material
- *Aeronautical Material Specifications*. AMS 2242. Tolerance

3. Requirements

3.1 Materials

3.1.1 Production Methods. The ingot metal shall be double vacuum melted in a furnace of a type suited for reactive metals.

3.1.2 Alloy Identification. The identity of all alloys with respect to ingot melt number shall be maintained at all stages of fabrication.

3.1.3 Condition. Unless otherwise specified, all material shall be supplied in the recrystallization annealed condition in accordance with Table 1.

Table 1

Thickness	% Recrystallization				
	Nb*	C-103	C-129Y	Nb-1 Zr	Cb-752
Less than .005	A/W	A/W	A/W	A/W	A/W
.005-.150	80	95-100%	95-100%	95-100%	95-100%
.151-250	75	90-100%	90-100%	90-100%	90-100%
.251-1.00	50	85-100%	85-100%	85-100%	85-100%

*Material supplied in the As Worked (A/W) condition unless otherwise specified.

NIOBIUM and ALLOY PLATE, SHEET and STRIP, CONT., P. 2

3.2 Chemical Composition. The chemical composition of each alloy ingot shall conform to Table II.

Table II

Element	Nb Grade I	Nb Grade II	C-103	C-129Y	Nb-1Zr	Cb-752
Tungsten	500 ppm*	500 ppm*	5000 ppm*	9-11%	500 ppm*	9-11%
Hafnium	---	---	9-11%	9-11%	200 ppm*	2000 ppm*
Tantalum	1000 ppm*	3000 ppm*	5000 ppm*	5000 ppm*	1000 ppm*	5000 ppm*
Yttrium	---	---	---	.05-3%	---	---
Titanium	---	---	.7-1.3%	---	---	---
Zirconium	500 ppm*	500 ppm*	7000 ppm*	5000 ppm*	.8-1.2%	2.0-3.0%
Carbon	100 ppm*	100 ppm*	150 ppm*	150 ppm*	200 ppm*	150 ppm*
Oxygen	200 ppm*	200 ppm*	250 ppm*	225 ppm*	300 ppm*	225 ppm*
Nitrogen	100 ppm*	100 ppm*	100 ppm*	100 ppm*	100 ppm*	100 ppm*
Hydrogen	15 ppm*	15 ppm*	15 ppm*	15 ppm*	15 ppm*	15 ppm*
Niobium	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.

*Maximum limits unless otherwise indicated.

ppm = Parts Per Million.

3.2.1 Product Analysis. If specified, product analysis shall be performed on C, 0_2, N and H with maximum levels specified as follows on Parts Per Million (ppm).

Element	Nb	C-103	C-129Y	Nb-1Zr	Cb-752
C	100	150	150	200	150
0_2	200	225	225	300	225
N	100	100	100	100	100
H	15	15	15	15	15

3.3 Tensile Properties. Elongation, yield strength, and ultimate tensile strength shall be measured at room temperature on samples transverse to final rolling direction on material which is 0.010" thick or greater. The strain rate shall be maintained at 0.05±0.005”/per inch/per minute through the 0.2% offset yield strength and at 0.05 ±0.005"/per inch/per minute thereafter. The material shall have minimum transverse tensile pmperly values as specified in Table III.

Table III

	Nb	C-103	C-129Y	Nb-1Zr	Cb-752
Ultimate tensile strength, 1000 psi	25	54	80	35	75
Yield strength, 0.2% offset, 1000 psi	11	38	60	15	55
Elongation, % in one inch	35	20	20	20	20

NIOBIUM and ALLOY PLATE, SHEET and STRIP, CONT., P. 3

3.4 Bend Ductility. Representative samples of the materials in final form shall withstand the following bend test at room temperatures without failure when tested according to procedures described in the most recent revision of the Materials Advisory Board report MAB-216-M, "Evaluation Test Methods for Refractory Metal Sheet Materials." The samples shall be sectioned with the long axis of the bend specimens perpendicular to the final rolling direction.

3.4.1 Sheet 0.060" in thickness and under shall be bent over a 1T radius through 105 degrees at a ram speed of one inch/per minute.

3.4.2 Sheet over 0.060" to 0.187" in thickness shall be bent over a 1T radius through 105 degrees at a ram speed of one inch/per minute.

3.5 Grain Size. Unless otherwise specified, the minimum average ASTM grain size number shall be in accordance with Table IV.

Table IV

Thickness	C-103	C-129Y	Nb-1Zr	Cb-752
.006-.150"	6	6	5	6
.151-.500"	5	5	4	5
Greater than .750"	---	---	---	---

4 Dimensions and Tolerances

4.1 Dimensions and Tolerances. Unless otherwise specified, tolerances shall be as defined in AMS 2242.

4.2 Flatness. Total deviation from flatness of sheet and strip shall not exceed 6% as determined by the following formula:

H/L X 100 = percent of flatness deviation.
where H = maximum distance from a flat reference deviation.
and L = minimum distance from this point to the point of contact with the reference surface.

4.3 Marking for Identification. Each plate, sheet, and strip shall be suitably marked with the contract number or order number, ingot melt number, specification number, and composition number.

5. Quality

5.1 General. The finished product shall be visibly free from oxide or scale of any nature, grease, oil residual lubricants, and other extraneous materials. Cracks, laps, seams, gouges, and fins shall be unacceptable.

5.2 Surface Rework. All surface pores, gouges, and other defects deeper than 0.005" or 3% of the thickness, whichever is smaller, shall be unacceptable. Sunace imperfections may be faired smooth to remove any notch effect provided dimensional tolerances are still maintained.

5.3 Edge. The edges shall be produced by shearing, slitting or sawing. The burr height shall not exceed five (5) percent of the thickness of the material.

NIOBIUM and ALLOY PLATE, SHEET and STRIP, CONT., P. 4

6. Reports

The supplier shall submit three certified copies of reports indicating the ingot chemistries. In addition, a certification statement will be added stating that although not tested, the material is in conformance with the specification. Additional tests for tensile yield, elongation, grain size and product analysis for oxygen, carbon, and nitrogen as well as other tests will be furnished when negotiated and specified in the purchase order.

7. Preparation for Delivery

7.1 Packaging. All material shall be packaged in a manner that will prevent damage in transit and in storage.

8. Rejections

Material not conforming to any of the requirements of this specification unless otherwise agreed upon by the purchaser.

9. Definitions

For the purposes of this specification, the following definitions shall apply:

9.1 Sheet Sheet is flat, rolled material up to 0.125" thick, and other material not considered strip, normally supplied in widths to 24".

9.2 Plate. Plate is flat, rolled material 0.125" thick or greater.

9.3 Strip. Strip is flat, rolled material up to 0.060" thick in widths under twelve inches (12").

NIOBIUM-TITANIUM ALLOY

1. Scope

1.1 Scope. This specification establishes the requirements for niobium alloy bar and rod containing 45/48 wt. % titanium for use in manufacturing wire for superconducting magnets.

2. Material

2.1 Ingots shall be at a minimum double arc melted.

2.2 Hardness testing will be performed on all ingots. Maximum average hardness shall be 170 BHN.

2.3 Top and bottom samples shall be taken from each ingot and analyzed to conform with Table I.

2.4 High homogeneity (Hi-Ho) material is available in Grade 1 or 2.

TABLE I – INGOT CHEMISTRY

	Grade I	Grade II
C	200	200
0	1000	1000
N	150	150
H	35	35
Fe	200	200
Si	100	100
Ta	1000	2500
Cr	60	60
Ni	100	100
Cu	100	100
Al	125	125
Ti	45/48% Specified Value ±1.5%	45/48% Specified Value ±1.5%
Nb	Balance	Balance

(Max. ppm unless otherwise noted.)

2.6 TABLE II - PRODUCT CHEMISTRY FINAL of VACUUM ANNEALED MATERIAL ONLY

C	200
0	1000
N	150
H	20

(Max. ppm unless otherwise noted)

NIOBIUM-TITANIUM ALLOY, CONT., P. 2

3. Technical Requirements

3.1 Dimensions: Dimensions shall be specified in the purchase order. Permissible variations in diameter for finished round product shall be as follows:

Diameter, inch	Permissible Variations from Diameter, inch	
	Plus	Minus
1/8 to 3/8, excl.	.002	.002
3/8 to 1/2 excl.	Per Purchase Order	
1/2 to 2, incl.	.006	.006

Finished product shall not be out-of-round by more than one-half the total permissible variations in diameter specified above. Permissible variations in straightness of finished products shall be as follows:

Diameter, inch	Permissible Deviation from Straightness, inch
1/8 to 1/2, excl.	.045 per foot of length
1/2 to 2, incl.	.060 per foot of length

Straightness shall be determined on a calibrated flatness table using standard techniques. No kinks.

3.2 Cleanliness

A. In Process - Precautions shall be taken during manufacture to assure removal of substances that might be deleterious to use of the finished product for the intended application.

B. Finished Product - Final cleaning shall be performed subsequent to performance of all nondestructive examinations. Materials shall be clean to the extent that no contamination is visible to the unaided eye, corrected for 20/20 vision, when viewed under an illumination of at least 100 foot-candles on the surface being inspected.

4. Mechanical Properties

4.1 Tensile Properties. The tensile properties for each lot shall be provided for information, and shall include the ultimate tensile strength, the yield strength, percent elongation, and the percent reduction in area. Tensile testing shall be done in accordance with the applicable provisions of ASTM E 8.

4.2 Hardness. The hardness for each lot shall be less than 170 BHN. Hardness testing shall be done in accordance with the applicable provisions of ASTM E 92, with the magnitude of the test load stated in the test report.

5. Surface Requirements

5.1 Surface Conditions. The finished materials shall be free from visually detectable cracks, seams, slivers, blisters, laps, gouges and other injurious imperfections, and from discontinuities unacceptable to specified nondestructive examination and tolerance requirements.

5.2 Surface Texture. The surface texture shall be 125 microinch finish (arithmetical average) or better for specified finished product. In process (or intermediate) material may be supplied in accordance with standard supplier practices.

NIOBIUM-TITANIUM ALLOY, CONT., P. 3

6. Internal Structure

6.1 Inclusions. The finished product of a size of 5/8" diameter or less shall be free of inclusions in excess of 0.013 inch (equivalent diameter) as determined by ultrasonic inspection between 1/2” and 5/8" diameter. Finished product of a size greater than 5/8" and less than 2" diameter shall be free of inclusions in excess of 3% of the product diameter or 0.032 inch, whichever is smaller. At manufacturer's discretion ultrasonic inspection may be performed at the finished size.

6.2 Defects. Defects such as cracks, laminations, and other ruptures shall be determined by ultrasonic examination per 6.1.

6.3 Grain Size. The grain size of each lot of finished product of 5/8" inch diameter or less shall be ASTM No. 6 or finer. The grain size shall be determined using the applicable provisions of ASTM E 112. Grain size of larger diameter product (greater than 1/2 inch) shall be specified in the purchase order.

7. Manufacture

7.1 Melting. The melting process shall be a process that has been established by the Supplier for melting niobium alloy containing 45/48 wt. % titanium for use in manufacturing wire for superconducting applications.

7.2 Working. Each ingot shall be reduced during manufacturing by a process that has been established by the Supplier for working niobium alloy containing 45/48 wt. % titanium for use in manufacturing wire for superconducting applications.

7.3 Heat Treatment. Heat treatment shall be accomplished in a manner which will preclude contamination that might cause a violation of the chemistry requirements and will ensure that each item in the lot being heat treated has essentially the same heat treated properties.

7.4 Fabrication

A. Manufacturing, dependent on ingot size, shall be in accordance with processes established and shall have a minimum of 75% reduction from ingot to billet. Material shall then be cold reduced by further work, a minimum of 50%.

8. Analytical Chemistry Procedures

The procedures for analyzing to the chemistry requirements of this specification shall be in accordance with standards considered industrially acceptable for the specified alloys.

9. Nondestructive Testing Requirements

All products shall be examined nondestructively in accordance with the following requirements and shall comply with the specified acceptance criteria.

NIOBIUM-TITANIUM ALLOY, CONT., P. 4

10. Ultrasonic Examination

Each finished bar or rod 1/2" diameter or larger, shall be examined as follows:

10.1 All bar and rods 1/2" diameter or larger shall be examined with ultrasonic test equipment calibrated with a flat bottom hole standard. Discontinuities producing indications with an amplitude equal to or greater than that produced in the reference hole shall be cause for rejection.

10.2 Circumferential Angle-Beam Examination. Each finished bar or rod shall be examined by circumferential angle-beam examination. This examination shall be performed by relating the angle-beam ultrasonic inspection procedures described in ASTM A-388 to solid bars or rods. For bars and rods the ultrasonic equipment shall be calibrated using a standard containing the following diameter hole drilled to a depth of 1/2 to 3/4 inch into the end of the standard parallel to the axis and a distance one-fourth of the diameter from the axis.

Rod Diameter, inch	Hole Diameter in Standard, inch
5/8 and less	0.013
greater than 5/8 to 2	3% of product diameter or 0.032 inch, whichever is smaller

The ultrasonic equipment shall also be calibrated for all bars using standards containing a notch which has the following depth and is no longer than 1/4 inch cut parallel to the longitudinal axis.

Rod Diameter; inch	Notch Depth, inch
1/2 to 2	1% of product diameter

Discontinuities producing indications with an amplitude equal to or greater than that produced in the reference or notch shall be cause for rejection or rework.

11. High Homogeneity Material

High homogeneity niobium-titanium alloys are available upon request. The degree of homogeneity will be determined by x-ray comparison to mutually approved standards. When specified, an x-ray positive(s) will be provided with the certification.

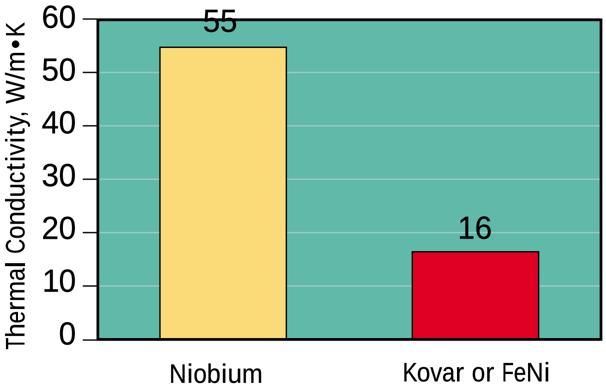
12. Conflicts

Where any of the above standards or specifications appear to be in conflict with the requirements of another reference document, such conflict shall be brought to the attention of the Purchaser for resolution.

THERMAL CONDUCTIVITY

Niobium is a relatively good thermal conductor in comparison to conventional leadframe, seal ring, or pin materials such as Kovar or FeNi alloys. (Figure 1)

FIGURE 1



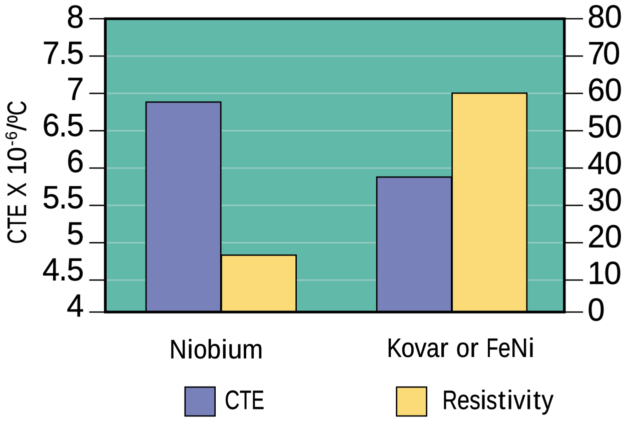
THERMAL EXPANSION

A relatively low coefficient of thermal expansion (CTE) makes niobium compatible with insulating materials like ceramics or glasses, especially GaAs semi-conductor components. (Figure 2)

ELECTRICAL CONDUCTIVITY

Niobium is a superior material, capable of carrying slightly more than four times the current (amperage) of a Kovar conductor of the same cross-sectional area, at a given voltage. (Figure 2)

FIGURE 2



OTHER CHARACTERISTICS

Corrosion resistance, non-magnetism, and fabricability are all desirable characteristics, plus niobium doesn’t require the heat treatments associated with Kovar. Choose niobium

- When additional thermal dissipation is desired
- When bonding of metal components to GaAs semiconductor or to BeO, Al203 et. al. ceramics is required.
- When excellent manufacturability is needed.

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PHYSICAL PROPERTIES OF NIOBIUM

AtomicNumber	41
Atomic Weight	92.91
Atomic Radius Å	1.47
Atomic Volume (cc/g-atom)	10.83
Density (g/cc @20°C)	8.55
(lbs./in3)	0.310
Crystal Structure	Body-centered cubic
Lattice constant, Å @ 20°C	3.294
Isotope, natural	93
Melting point	4474°F
	2468°C
Specific Heat (cal/gm @ 0°C)	0.0647
Thermal conductivity (cal/cm•sec•/°C @ 0-100°C)	0.13
Coefficient of linear expansion per °C	6.8-7.0 x 10^{-6}
Heat of sublimation (K cal/g-atom @ 20°C)	170.9
Heat of combustion (cal/gm @ 20°C)	2379
Electrical resistivity (microhm•cm @ 20°C)	13.2-14.8
Temperature coefficient of electrical resistivity per °C	0.00395
Ionization potential, volts	6.77
Positive ion emission, e.v	5.52
Electron work flinction, e.v	4.01
Electrochemical equivalent, mg/coulomb	0.1926

MECHANICAL PROPERTIES

Tensile strength, (psi) sheet, annealed Grade I	30,000
Yield strength, (psi) 0.2% offset, sheet, annealed	15,000
Elongation, %, annealed	30
Young's Modulus, (psi)	15.2 x 10^6
Hardness, (500 kg) Brinell, arc cast	60 to 75
electron beam cast	45 to 65

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