

# T.I.C.

## TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

### PRESIDENT'S LETTER

Following the successful Thirty-ninth General Assembly in Prague, the Executive Committee met in Boston at the end of November to discuss the position of a promotional officer for the T.I.C.

It was decided to upgrade the role and position of the Secretary General and at the same time to advertise to fill a part-time promotional officer position reporting to Judy Wickens.

The role of this officer will be to promote the metals tantalum and niobium and their properties. The promotion will be directed towards potential new users and applications and will be largely carried out through the electronic media.

Nominations for this position are open and suitably qualified people can register their interest with Judy.

Plans for the Fortieth General Assembly in Perth are progressing but we are still looking for interesting speakers, especially on the applications for tantalum and niobium.

The tantalum industry has survived the 'Asian Crisis' very well and the combination of slower growth in several parts of the world, and the announcement of increased resources has stabilised the industry going into 1999.

JOHN LINDEN

### MEETING IN PERTH

#### Fortieth General Assembly October 24th-26th 1999

The next General Assembly of the Tantalum-Niobium International Study Center will take place on October 25th 1999 as part of a meeting from October 24th to 26th in Perth, Western Australia. The formal sessions will be held at the Burswood Hotel, where delegates will also stay.

The meeting will open on Sunday October 24th with registration and, in the evening, a welcome reception given by the T.I.C.

On Monday October 25th the General Assembly, to carry out the business of the association, will be followed by a full programme of technical presentations. Papers already confirmed cover 'Expansion at Greenbushes and Wodgina' by Mr David Bale, Gwalia, 'Low grade slag supplies in South-east Asia' by Mr Yeap Soon Sit, S.A. Minerals, 'Tantalum and Niobium Production by Paranapanema' by Mr Jorge Jose Correia Salles, Paranapanema. In addition we hope to have papers covering developments in capacitor powder technology, trends in tantalum capacitor usage, and news of alloys and applications of the two metals.

A gala dinner at the Lake Karrinyup Country Club will be hosted by Sons of Gwalia in the evening of Monday.

There will be a choice of plant tours on Tuesday October 26th: participants may select either a trip to see the Wodgina mine, or a tour of the new developments at the Greenbushes operation.

Attractive sightseeing tours and entertainment for those accompanying the delegates will be arranged.

Invitations will be sent to the nominated delegates of member companies. Anyone else interested in attending should contact the T.I.C. without delay.

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**T.I.C. website**  
**[www.tanb.org](http://www.tanb.org)**



# TANTALUM AND NIOBIUM IN CHEMICAL APPLICATIONS

by Mr C.E.D. Rowe, Special Metals Fabrication, presented at the T.I.C. meeting in Prague, October 1998.

## ABSTRACT

The majority of tantalum currently produced is used in the electronics industry in tantalum capacitors. Other uses for tantalum and niobium are in special steels, as carbides in cutting tools, high vacuum furnaces and in the chemical industry. Tantalum and niobium have outstanding corrosion resistance similar to glass and are almost as inert as gold or platinum in the most hostile of environments. This paper explores the physical, thermal and mechanical properties of tantalum and niobium of interest to the chemical engineer, discusses the uses and special fabrication techniques required, as well as their advantages and disadvantages in the chemical and pharmaceutical industries.

## INTRODUCTION

The majority of tantalum currently produced is used in the electronics industry in tantalum capacitors. Other uses for tantalum and niobium are in special steels, as carbides in cutting tools, high vacuum furnaces and in the chemical industry. Tantalum and niobium have outstanding corrosion resistance similar to glass and are almost as inert as gold or platinum in the most hostile of environments. This paper explores the properties of tantalum and niobium of interest to the chemical engineer, discusses the uses and special fabrication techniques required, and their advantages and disadvantages in the chemical and pharmaceutical industries.

## PROPERTIES OF TANTALUM AND NIOBIUM

Tantalum (Ta) is a blue grey metal similar in appearance to lead and of density 16.6 g/cc which is approximately twice that of steel. Niobium (Nb), also known as columbium (Cb), with a density of 8.57g/cc, is a silvery metal, similar in density and appearance to stainless steel. Because their melting points are higher than the melting point of platinum (1750°C), tantalum and niobium are classed as Refractory Metals and these properties are utilised in many high temperature vacuum furnace and electronic applications. The high density of tantalum along with its high melting point and deformation characteristics are utilised in many military and defence applications. Tantalum and niobium are used as an alloy addition in many superalloys and, as carbides in cutting tools. One of the main and important uses of tantalum is in electronic capacitors. Niobium alloyed with tin or titanium is used in superconductors for electrical power transmission. Because of the low thermal neutron cross section, niobium is widely used in the nuclear industry as a canning material. However these applications are beyond the scope of this paper. Only the properties of the materials of interest to the chemical engineer are presented in **Tables 1 and 2**.

## PRODUCTION METHODS FOR TANTALUM AND NIOBIUM

Tantalum and niobium are produced by powder metallurgical, vacuum arc or electron beam melting techniques. The latter gives the purest material, >99.99%, but sheet produced from electron beam melted ingots tends to have a coarse grain structure so components produced from this material by spinning tend to exhibit a rough surface finish. Although grain size can be controlled to some extent by controlling the rolling/annealing schedules, for finer grained material it is necessary to use a powder metallurgical or arc-melted feedstock. These materials are slightly less pure >99.9% but still exhibit the excellent corrosion resistance. To improve specific properties tantalum is

Physical Properties	316 Stainless	Tantalum	Niobium
Density	7.94 g/cc	16.6 g/cc	8.57 g/cc
Melting point	~1600°C	2996°C	2497°C
Coef. of Linear Expansion at 100°C	$16.8 \times 10^{-6}/^{\circ}\text{C}$	$6.5 \times 10^{-6}/^{\circ}\text{C}$	$6.5 \times 10^{-6}/^{\circ}\text{C}$
Specific heat at 100°C	0.500 J/g/°C	0.142 J/g/°C	0.270 J/g/°C
Thermal Conductivity	16.0 W/m°C	50.4 W/m°C	50.0 W/m°C
Neutron cross section	21.3 barns	1.1 barns	
Ductile/Brittle Transition Temperature	ductile above -270°C	ductile above -196°C	ductile above -150°C

Table 1: Physical Properties

Mechanical Properties	316 Stainless	Tantalum	Niobium
Modulus of elasticity 20°C	201000 MPa	180000 MPa	99000 MPa
100°C	193000	179000	97000
200°C	184000	176000	95000
Yield stress 20°C	300 MPa	152 MPa	103 MPa
100°C	225	103	69
200°C	178	83	55
Tensile strength 20°C	600 MPa	276 MPa	207 MPa
100°C	550	262	186
200°C	520	248	172
Hardness Cold worked	250 - 300 Hv	180 - 300- Hv	110 - 180 Hv
Hardness Annealed	160 - 200 Hv	70 - 110 Hv	60 - 100 Hv

Table 2: Mechanical Properties



Alloy	Uses
Tantalum	Chemical, Electronic, Furnace, Aerospace
Tantalum 2 1/2% Tungsten	Chemical
Niobium	Chemical, Electronic, Nuclear
Tantalum 10% Tungsten	Aerospace, Chemical
Tantalum 40% Niobium	Chemical
Niobium 1% Zr	Nuclear, Chemical
Niobium 10%Hf 1%Ti 0.7Zr	Nuclear
Niobium 7.5%Ta	Superconducting
Niobium 46.5%Ti	Superconducting

Table 3: Alloys and their uses

often alloyed with other metals such as tungsten whilst niobium is alloyed with zirconium. The addition of tungsten improves the mechanical properties of the material whilst having little effect on the corrosion resistance. In fact tantalum 2 1/2% tungsten alloy is recommended for certain applications because of its superior corrosion resistance in sulphuric acid. The addition of 1% zirconium to niobium reduces brittleness in certain chemical environments. Some of the alloys currently available and their uses are listed in descending order of importance to the chemical industry in Table 3.

## FABRICATION TECHNIQUES

The cold working properties of both tantalum and niobium are excellent and the metals and their alloys can be fabricated by all the usual metalworking techniques such as shearing, bending, deep drawing, drilling, turning and spinning. Because of the high material cost of tantalum and its high density, only small components such as crucibles and thermocouple pockets are made from solid material, in most applications thin tantalum sheet is clad onto a cheaper base material as will be discussed later in the paper. For drilling and turning, high speed steel or carbide tools are used with soluble oil or trichlorethylene as lubricant, both tantalum and niobium machining like copper. Tantalum and niobium tend to stick to tools during spinning or deep drawing so the use of aluminium bronze tools and mandrels is recommended, lubricated with tallow. Spinning is carried out without heat and components should be annealed after about 90% reduction. Tantalum and niobium pure metals are annealed around 1000°C and tantalum tungsten alloys around 1200°C. Because both tantalum and niobium and their alloys react with oxygen and other non-inert gases above 250°C annealing must be carried out in high vacuum to avoid contamination. Similarly welding of the materials must be carried out in a vacuum chamber back filled with pure argon. One of the welding chambers used at Special Metals Fabrication Ltd. in the UK is shown in Fig. 1 Chambers are available to accommodate an entire shell and tube heat exchanger. Conventional Tungsten Inert Gas welding techniques are used within the chamber without the use of filler wire for material thicknesses up to 0.8mm. Above this thickness the appropriate metal filler wires are used, with conventional weld preparations being applied to the thicker sections and multi pass welding techniques used to complete the weld. Electron beam welding can also be carried out but the set-up is more complex. Nevertheless, electron beam welding is recommended for sheet thinner than 0.3mm. Although some grain growth occurs in the weld region, if welds are performed under the correct conditions, they are as strong as the parent metal and do not suffer the effects of the heat affected zone as do many other materials. Powder Metallurgical materials are not recommended for applications involving welding as residual gases in the materials lead to gassy, low strength porous welds. All the materials listed can be spot welded and, if the cycle time is kept

short, this can be carried out without a protective atmosphere but the use of an argon shield or welding under water is recommended for thicker materials.

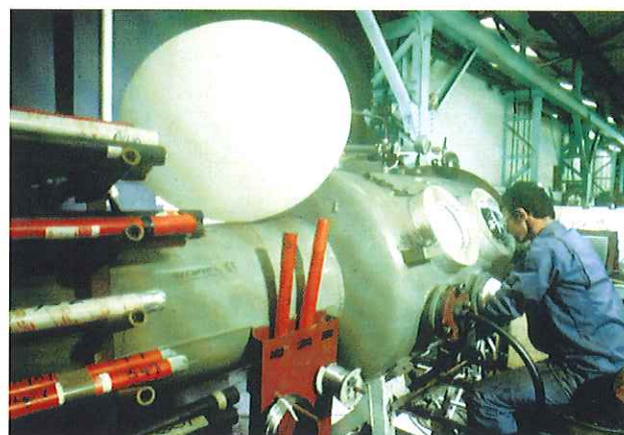


Fig 1: One of the argon filled welding chambers at Special Metals Fabrication Ltd. UK

## CORROSION RESISTANCE

Tantalum has been evaluated for corrosion resistance in over 2000 different reagents and it is only corroded by about 40 of these. Its excellent corrosion resistance is due to the presence of a thin surface film of tantalum pentoxide  $Ta_2O_5$  naturally formed on all tantalum when exposed to air even at room temperature. This makes the metal passive and gives a corrosion resistance similar to glass and on a par with gold or platinum. Reagents which attack this oxide film are the only ones which attack tantalum. These are strong alkalis, fuming sulphuric acid containing free  $SO_3$  or  $SO_2$ , fluorine, hydrofluoric acid and solutions containing fluoride ions in excess of about 10ppm. Sodium and potassium hydroxide solutions attack both tantalum and niobium - solutions greater than 10% for tantalum and 5% for niobium attack and cause embrittlement even at room temperature. Both materials are embrittled by oxalic acid but Nb1%Zr is more resistant. The corrosion resistance of niobium is slightly inferior to tantalum in hot aqua regia, hydrochloric acid, hydrogen peroxide, nitric acid, phosphoric acid, hypochlorites and sulphuric acid stronger than 20%. However, being ~60% cheaper than tantalum, niobium and the Ta60Nb40 alloy are increasingly being used in chemical applications where their corrosion resistance is sufficient. For many strong sulphuric acid applications and when using high pressure steam, tantalum 2 1/2% tungsten alloy is widely used especially for heat exchanger tubes. Tantalum 2 1/2% tungsten alloy also gives a 25% increase in strength with very little increase in weight. Both tantalum and niobium have good resistance to molten metals hence their use as crucibles and casting moulds. Tantalum is one



Liquid Metal	Tantalum	Niobium 1%Zr
Aluminium	Not resistant	Not resistant
Bismuth	<900°C	<560°C
Calcium	<1200°C	<1100°C
Copper	Resistant	Resistant
Iron	Not resistant	Not resistant
Mercury	<600°C	<600°C
Potassium	<1000°C	<1000°C
Lithium	<1000°C	<1000°C
Magnesium	<1150°C	<980°C
Silver	<1200°C	<1150°C
Sodium	<1000°C	<1000°C
Lead	<1000°C	<850°C
Zinc	<450°C	Not resistant

Table 4: Resistance of Ta and Nb to liquid metals

of the only materials to resist attack by molten plutonium. The resistance of tantalum and niobium to liquid metals is summarised in Table 4. The resistance of Nb1Zr to molten sodium permits widespread usage in high pressure sodium lamps.

It should be noted that the oxygen content of the molten metals should be kept lower than 50ppm to prevent reaction with and oxygen embrittlement of the tantalum or niobium.

## APPLICATIONS IN THE CHEMICAL INDUSTRY

Tantalum (and to a lesser extent niobium) is used for heat exchangers, shell and tube heaters, bayonet heaters, condensers, thermocouple pockets, bursting discs, vessel liners and glass vessel repair parts. As mentioned above, tantalum is an expensive and dense material so its use as solid material is limited to smaller parts such as crucibles, tubes for heat exchangers, bursting discs and repair parts. A solid tantalum thermopocket is shown in Fig. 2. and tantalum clad stainless steel thermopockets in Fig. 3.



Fig. 2: A thermopocket made from solid tantalum.

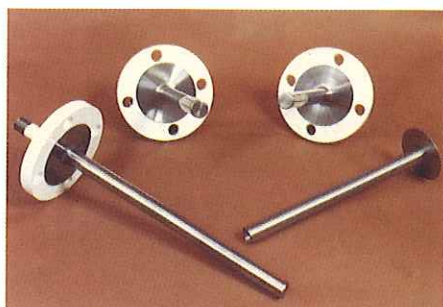


Fig. 3. Thermopockets made from stainless steel tantalum clad.

Larger parts, because of the cost of tantalum and also weight considerations, are made by cladding steel, stainless steel or copper with a thin layer of tantalum or niobium. Such cladding can be by purely physical contact or by explosive bonding. The former method is used for general applications but the latter can be used where the tantalum may be subjected to vacuum or low pressure conditions which would tend to pull the cladding away from the substrate. Explosively clad materials, although theoretically superior, do present fabrication problems especially during welding. To avoid contamination of the weld region with substrate material, it is necessary to insert getter strips between the tantalum and substrate in the weld region. This adds to the fabrication costs and it is more usual to rigidise the sheet as shown in Fig. 4. Some of the modern structural adhesives may prove useful in bonding tantalum to substrate materials. The effect of not rigidising is shown in Fig. 5. Here the lining has pulled off due to the effects of internal vacuum.



Fig. 4: Conical condenser at end of a tantalum shell and tube heat exchanger showing rigidising of the tantalum lining to withstand vacuum conditions.

Tubes for shell and tube heat exchangers are made of pure tantalum, tantalum 2 1/2% tungsten or niobium are available in standard sizes - 3/4 inch diameter with 0.4mm wall thickness, 1 inch diameter with 0.5mm wall thickness. Tubes can be seamless or welded. Tube plates of the heat exchanger are normally made of carbon or stainless steel clad with tantalum on the process side. The tubes are expanded into the tube plate or into copper ferrules inserted in the plate to give rigidity. The tantalum tubes are then seal welded to the tantalum tube plate cladding.

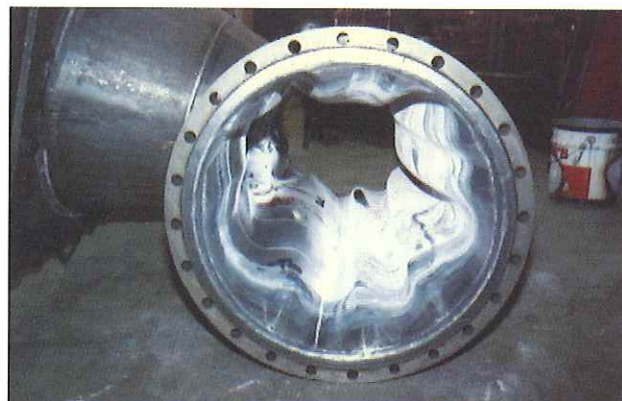


Fig. 5: Failed un-rigidised tantalum lining.



## HYDROGEN EMBRITTLEMENT

There is a major problem with tantalum and niobium which causes disastrous failure in seemingly friendly environments even at room temperature - that problem is hydrogen embrittlement. Tantalum or niobium must never be allowed to become cathodic in the presence of any environment containing hydrogen ions or gas, especially nascent hydrogen which attacks the metal grain boundaries causing the materials literally to crumble to powder. Thus they should not be used in electrolytic contact with Hastelloys® nickel, stainless or carbon steel even in quite low electrolytic concentrations. To survive under such conditions they must be electrically insulated from the rest of the plant or a small area of the tantalum or niobium must be plated with gold or platinum which attracts the hydrogen ions causing them to combine to form hydrogen gas which bubbles safely away leaving the tantalum or niobium free from attack. A plated area of platinum ~5µm thick and of area ~1/20000 that of the area to be protected is sufficient. Thus a 15mm diameter spot is often used to protect an entire 4 tube tantalum bayonet heater.

## WHY USE TANTALUM OR NIOBIUM ?

For normal processing temperature and with the majority of corrosive environments other, less expensive materials such as stainless steel, Hastelloys® titanium, graphite, glass or special ceramics can be used. Why then is tantalum used?

Consider a shell and tube heat exchanger. Made from graphite its cost is only about a third of a similar tantalum exchanger but a graphite exchanger lasts for 3 - 5 years whilst tantalum should last for at least 20 years providing it is not mistreated. The majority of failed tantalum fabrications received back at our works for repair have failed due to incorrect handling during service. Most failures are due to cleaning using sharp tools to remove surface deposits. These can perforate the soft tantalum cladding resulting in rapid failure when the plant is returned to operation.

Another advantage of tantalum over graphite is that graphite heat exchangers are necessarily thick walled and thus respond slowly to temperature changes whereas tantalum and niobium have good thermal conductivity leading to better temperature control in chemical plants. Degradation of the graphite also occurs during service and this can cause contamination of many pharmaceutical products leading to increased use of tantalum in this industry. Glass equipment may be used for many applications but again, glass has the disadvantages of being brittle, suffers from thermal shock, has a low thermal conductivity and is difficult to fabricate. Tantalum and niobium have none of these disadvantages as well as having a low fouling factor leading to less frequent cleaning of the plant being necessary for peak operating efficiency. It is possible to combine the best of both worlds such as a tantalum coil within a glass vessel, typical examples are shown in Figs.6 and 7.

## CONCLUSIONS

Although more expensive and difficult to fabricate than conventional materials used in chemical plant, tantalum and niobium give increased plant life, reduced maintenance, better process control and zero product contamination. They thus provide a very cost effective solution to many process plant problems. Any disadvantages using these materials can be easily overcome and a good future is forecast for their use in the Chemical Process Industry.

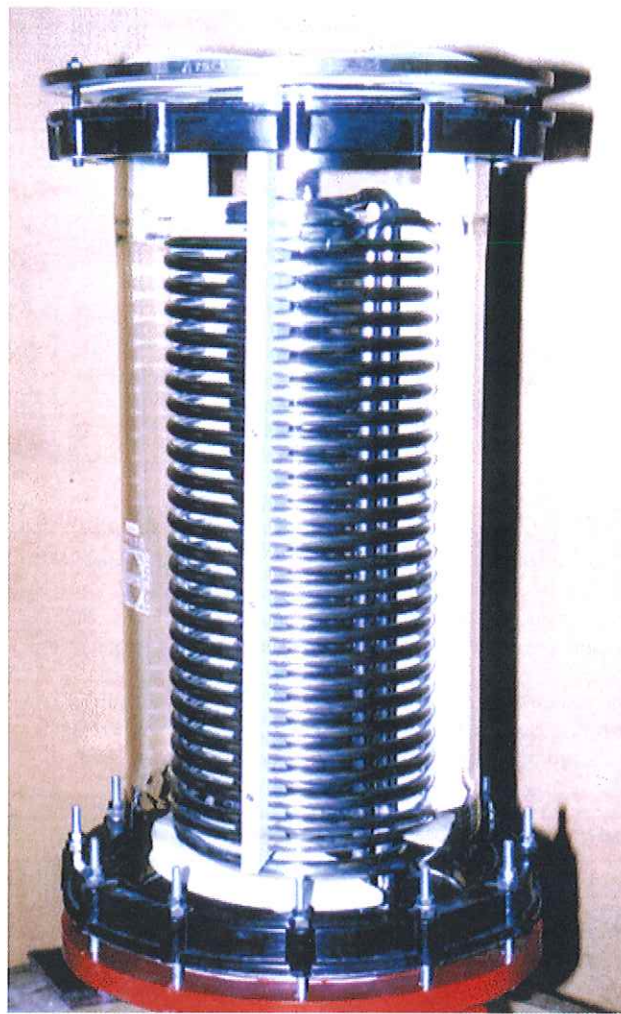


Fig 6: Tantalum coil inside glass vessel



Fig 7: Multi-turn tantalum helix condenser assembly inside a glass vessel



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## POSITION AVAILABLE

### TECHNICAL PROMOTION OFFICER

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We have available a position for someone  
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and their end-use applications

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#### MEMBER COMPANY NEWS

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##### **Mitsui Mining & Smelting Co., Ltd.**

The Head Office of Mitsui Mining & Smelting Co., Ltd. was moved in January 1999 to the West Tower of the Gate City Osaki office complex, to have room to expand for the 21st century. The address is now:

1-11-1, Osaki,  
Shinagawa-ku,  
Tokyo 141-8584, Japan.  
Tel: +81 3 5437 8000 (information)

Rare Metals Business Department:  
Tel.: +81 3 5437 8094  
Fax: +81 3 5437 8095

##### **Kemet**

Although net sales in the quarter ended September 30th 1998 were \$137.7 million compared with \$165.5 million for the same quarter in 1997, Mr David E. Maguire, chief executive officer and president, was 'pleased to report that we at Kemet continue to achieve lower break-even operational costs'. The company was investing for the future to position the company well as the electronics industry recovered, and bookings had increased 8% from the lowest levels of 1998, which had occurred during the previous quarter, he stated. The company remained confident of future growth in revenues and earnings, and of a positive outlook for the electronics industry.

##### **Kennametal**

The fax number for the T.I.C. delegate Mr David Landsperger is now +1 724-539-3951

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