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Oxide films on tantalum and niobium
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Tantalum Bowl, c.1983, by James Brent Ward

Tantalum capacitors in critical medical applications
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Photo credit: Collection: The Worshipful Goldsmiths’ Company; Photography: Clarissa Bruce, Alf Barnes
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President’s Welcome

Dear members and associates:

I write this letter from my home office, in compliance with business and local authority requirements for social distancing and stay-at-home restrictions related to the Covid-19 pandemic. I hope you, your family members and friends are all safe, virus free, and in compliance with whatever safety guidelines are in place in your localities as we all try and stem the growth of this terrible virus.

It is amazing how quickly our daily routines have been upset as we learn how dependent we have become on the very technology many of us are in the business of promulgating. In some respects, it is fortunate this technology has been developed as it is allowing business and commerce to continue, albeit at a lower level than normal, as we learn to manage this situation. Web-enabled remote learning is allowing our children to maintain their educational progress, video-conferencing tools are allowing businesses and individuals to maintain connections with associates, customers, and family whether they be the next town away or half-way around the world. In addition, cloud-based services are allowing us to share all the necessary documents and information, on a secure basis, so we can maintain some level of normalcy.

These very same tools were somewhat taken for granted when things were normal. At that time, we probably were not using them to their fullest. It will be interesting to see how our mindsets will change now that we truly understand what we have built and the benefits and efficiencies these tools represent, once things get back to normal.

Let’s all of us pray that we get this pandemic under control before it affects considerably more individuals, family and friends.

You are in my thoughts and prayers.

Dr Daniel Persico
President
Tantalum capacitors in medical applications: life support monitoring equipment and ventilators

Paper written by Tomas Zednicek Ph.D. of the European Passive Components Institute (EPCI). All views and opinions in this article are those of the authors and not the T.I.C.

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The ability of tantalum capacitors to answer requirements for reliability, robustness and high energy density make them critically important for advanced medical applications such as lung ventilators and life support monitoring equipment. Medical ventilators provide assistance for hospital patients suffering with breathing difficulties; critically important today for patients around the world suffering from critical stages of Covid-19. This was emphasized on April 1st when the European Passive Components Industry Association (EPCIA) and German Electrical and Electronic Manufacturers Association (ZVEI) issued a joint request that countries classify the passive components supply chain as a “key” industry operation which must continue during the crisis.

Motor Controller

High-performance ventilators require stable and reliable computerized turbine control to synchronize the machine’s output with the patient’s own efforts. Tantalum capacitors are found on the printed circuit board (PCB) of the brushless motor controller which helps to deliver precise rotational velocity and direction control of the scroll compressor (see Figure 1).

![Figure 1: Direction control PCB of brushless motor with tantalum capacitors circled in red. Photo: Advanced Motion Controls](image)

Main control processing unit (CPU) board

The main CPU is the ‘brain’ of the system; it requires fast processing with reliable continuous operation. There are multiple input and output circuits, processor coupling and local DC/DC converters that require a long operating life and stable operating conditions. They are supported by tantalum capacitors (see Figure 2).

![Figure 2: Ventilator main CPU PCBs, showing tantalum capacitors circled in red](image)
High accuracy analogue measurement boards
Control accuracy and the precise response of medical devices also depends on reliable measurement and conversion of AC voltage and current inputs from the patient monitoring sensors.

Texas Instruments TI TIDA-01214 is an example of isolated 16-Bit ADC, high-accuracy analog input module with improved trip time performance and reliability suitable for medical applications (see Figure 3). The alarm feature is designed to identify the AC analog input faults on a sample basis for faster fault detection.

Figure 3: Texas Instruments TI TIDA-01214 ADC converter, with tantalum capacitors circled in red
(photo: Texas Instruments)

Main power board and DC/DC Converters
The continuous uninterrupted operation of medical devices depends on the main power supply and the local DC/DC converters to supply a wide range of functionalities. A number of such power supply designs depend, among other components, on the reliability of tantalum capacitors. Figure 4 demonstrates use of tantalum capacitors with DC/DC step down switching regulator LT1374 on a medical board.

Figure 4: DC/DC power converter for medical application, tantalum capacitors circled in red

Summary and conclusion
Tantalum capacitors play a critically important role in medical applications, including life support monitoring equipment and lung ventilators. Today, more than ever, tantalum capacitors are vital in the urgent struggle of healthcare institutions and healthcare professionals battling the global Covid-19 pandemic.

Tantalum capacitor manufacturers are faced with increased customer demand for critical medical equipment including lung ventilators, mobile X-ray, CT systems, ultrasound and patient monitors. All current efforts are focused on ensuring the delivery of parts as soon as possible, with shortest possible lead-times.

Beautiful metal: oxide films on tantalum and niobium

In the last edition of the Bulletin (#180, January 2020) we revealed some of the latest niobium and tantalum creations by the pioneering artist James Brent Ward. In this article Roland Chavasse explores the subject in more detail; introducing the physics behind interference colours and discussing ways that they can be made to appear.

Introduction

Some people say that beauty is only skin deep and when it comes to the coloured oxide films on tantalum and niobium this is true. Such colours may have the appearance of pigmentation, but in fact they are interference colours, a form of structural colour created purely by the way the surface of an object reflects light.

Interference colours occur naturally in a wide range of beetles, birds, and butterflies and they can also appear on seashells, fish, and even plants. We can see interference colours on soap bubbles, as temper colours on steel, on oil films on water, and, of course, on the anodised surfaces of tantalum and niobium metal. In each of these cases the colours we see are created by the way micro- and nano-scale surface structures absorb, refract and reflect light and not by pigments.

Interference colours appear when light waves reflected by the upper and lower boundaries of a thin, semi-transparent film interfere with one another, strengthening some wavelengths and weakening others, to create the appearance of colour.

As well as their use for jewellery and art, interference colours have a number of practical applications too. Notably tantalum and niobium capacitor manufacturers will refer to a colour chart as the first indication that a batch of anodised parts have been made correctly (colour uniformity shows that the oxide thickness is consistent across whole anode surface). Other practical uses for interference colours include optical mineralogy, where they can be used to identify minerals in a technique known as double refraction or birefringence, as security features on high-value bank notes and in the field of next generation computer memory devices.

This article will look at the physics behind interference colours and discuss what causes them to appear, before exploring how to create interference colours on niobium and tantalum, and potential applications of such techniques.
The theory of thin film optical interference

The phenomenon of interference colours depends on the presence of two parallel reflecting surfaces, which have a very small separation, and where the upper surface must be at least semi-transparent.

To understand the effect, consider light as a wave; the colour is specified by its wavelength. Visible light ranges from violet, which has the shortest wavelength at around 380 nanometres (nm), to red, which has the longest wavelength at around 700 nm (see Figure 2). Natural ‘white’ sunlight consists of waves with a countless number of wavelengths between these limits.

Figure 2: The spectrum of visible light

To understand interference colours, consider a simple wave. The wave in Figure 3 has wavelength $\lambda$ and amplitude $A$. It is a single wavelength of light, giving a single colour (i.e. monochromatic light). The wavelength determines the colour we see, while the amplitude determines how bright it appears (the higher the amplitude, the brighter the light is).

If a second similar wave is imposed on the first wave, and if the peaks and troughs of the waves coincide then the amplitude will be reinforced (Figure 4a). When the waves coincide and reinforce each other they are said to be ‘in phase’, and the light appears to be twice as bright. If, however, a second similar wave is imposed on the first wave but the peaks of the second wave coincide with the troughs of the first wave, then the waves will cancel each other out (Figure 4b). They are said to be ‘out of phase’ and to an observer no light can be seen.

Figure 4a: Waves ‘in’ phase combine amplitudes to reinforce each other

Figure 4b: Waves ‘out’ of phase cancel each other out
Now, if two sources of white light, each containing many wavelengths, could be arranged to reach the eye with only one wavelength in phase, then all other wavelengths would be weakened and a single colour would dominate what could be observed. This is what happens when natural light strikes a semi-transparent thin film. Some light is reflected from the upper film surface and some enters the film, reflects off the lower surface and leaves in the same direction as the directly reflected wave (see Figure 5).

![Diagram of optical interference in a thin film](image)

Figure 5: a) Diagrammatic illustration of optical interference in a thin film, b) a nominally thinner film with red ‘out’ of phase and blue ‘in’ phase, c) a nominally thicker film with red ‘in’ phase and blue ‘out’ of phase.

If the two surfaces are parallel and rays arrive at a fixed angle, only a single wavelength out of all the wavelengths making up the ray of light will be equivalent to the path length $(xy+yz)$. Therefore, most reflected rays will have some degree of being ‘out’ of phase and the wavelength that is ‘in’ phase will be the dominant colour seen.

As the thickness of the film increases the colour seen will progressively move through the spectrum, theoretically from violet to red. When the whole spectrum has been traversed, a second and third series of colours may be obtained as film thickness and light path increases and colours can go through integral multiples of 2x and 3x visible light wavelengths. Such colours appear as 1st, 2nd and 3rd order colours, but higher order colours get progressively weaker and hold less aesthetic interest. At voltages over 300 V the oxide layer is so thick that the interference colours are no-longer visible to the human eye, leaving a matt grey appearance.

In practice there is no smooth transition through the spectrum. True monochromatic colours are not generally observed, as adjacent colours may be only partly extinguished and while some tolerate a range of thicknesses others only appear in a precise film thickness. For example, a pure red interference colour has never been obtained on tantalum or niobium; the sequence of colours jumps straight from dark pink to purple.

Since interference colours depend on the apparent film thickness, their appearance will vary according to the angle at which the surface is viewed. The film appears thinnest when viewed at 90° (perpendicular) and as the angle changes the apparent film becomes thicker and the perceived colours change too.

Surface conditions will also affect reflectivity and, therefore, the intensity of colour that is perceived. While non-flat surfaces will show variations in colour, predictably the hue will change if the film is made thinner by rubbing or abrasion over time (which can limit its applications in certain types of jewelry).

To obtain strong colours it is necessary that the amount of light reflected from the outer film surface and that emerging from the surface are roughly similar, or else the intensity of the colour is low. Tantalum and niobium are well suited to creating strong, permanent colours that will never fade or tarnish as long as the film remains undamaged.

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1 - Interference colours in films shift according to Bragg’s law, which states that optical path length is a function of film thickness and index of refraction.
How to create interference colours on niobium and tantalum

As with many other metals, tantalum and niobium can be coloured by either heat or electrolysis. Heat can be used to thicken the oxide coatings relatively easily, but it is very hard to control and for precise control electrolysis offers outstanding results through a combination of superior working properties and strong colours.

According to James Brent Ward, a master craftsman when it comes to creating metal interference colours, although titanium is more commonly studied, it is niobium and tantalum that have the best combination of film colour, metal workability and cost. They also have a higher specific gravity than titanium, closer to silver or gold, making them more attractive for jewellery.

An introduction to anodic oxidation

When current is passed through a conducting aqueous liquid via electrodes immersed in the liquid, oxygen is formed at the positive electrode (anode), where it will react with the anode metal to make a tightly bonded oxide film. The oxide layer acts as an electrical insulator and it will grow uniformly until it is thick enough to stop the passage of current. The higher the voltage, the thicker the layer of oxide, and the creation of a particular colour (see Figure 6).

Figure 6: Film thickness of niobium oxide after anodisation in a 5% citric acid solution showing colours of test pieces and demonstrating the linear relationship between voltage and film thickness (Komatsu et al, 2016).

Figure 7: Tantalum capacitor anode test racks from a dipping line (left) and mixed scrap (right). The colours are specific to anodisation conditions, which for these items would typically be phosphoric acid electrolyte at 60-85°C.
Preparation
There are two main methods of anodising, depending on whether the work piece is immersed in a non-conductive tank of electrolyte or whether the work piece is touched in selected places by a brush moistened with electrolyte.

Before starting the process, the work piece must be thoroughly degreased, clean and dry. Keep in mind also the variations in surface finish, since different textures have different reflectivity and will change the apparent colour significantly.

There are many effective electrolytes, including 5% sulphuric acid, 5% citric acid solution, 10% ammonium sulphate with tap water, phosphoric acid, 15% orthophosphoric acid. Coca-Cola and Lucozade are also said to be “excellent”. Each electrolyte will produce slightly different colours.

Immersion anodising
This process is essentially similar to the anodising technique used to grow the dielectric coating in tantalum capacitors. Firstly, turn off the power, connect the work piece to the positive power supply and immerse it in the electrolyte. Turn on the power and wait until the ammeter shows that the current has stopped (alternatively watch for when the bubbles cease).

The temperature of the electrolyte has a significant influence on the range of colours that are produced.

Note that one can selectively anodise the work piece by covering sections with a temporary insulator such as lacquer or resist. Areas covered by the insulator will not form oxide films and therefore show a different colour.

Brush anodising
This method is more difficult but gives greater control and is good for smaller items or adding artistic details, as can be seen in Figures 9, 10 and 12. Wearing rubber gloves, connect the wooden-handled, metal-ferruled paint brush to the negative side of the power source using an insulated lead. Specialist equipment can also be used (see Figure 9).

The work piece is held firmly in place and connected to the positive side of the power source. Set the voltage, dip the brush in electrolyte, then touch the wet brush on the metal surface; colour will immediately appear.

Although this technique is relatively simple to describe, there is considerable skill required to master the full range of effects that are possible through varying the voltage and speed of brush movements.

It’s worth remembering to anodise with the highest voltage first and then descend through the lower voltages, otherwise if you use lower voltages first the relatively thin oxide layers they create could be thickened and changed when higher voltages are used subsequently.
Some examples of interference colours on items of niobium and tantalum

Strong, permanent interference colours on metal objects hold several practical applications, but one shouldn’t overlook that they can have great aesthetic appeal too. Given their rich tones and hues it should be no surprise to find them employed in art, numismatics (coins) and jewellery; after all, nature has been using structural colours to look spectacular for millions of years.

Art

Arguably the leading artist working with tantalum and niobium today is James Brent Ward (right) whose pioneering work and extensive writing has been a major influence in this field for over 40 years. Talking to the Bulletin, James explained that inspiration for his work came from his extensive travels, particularly in South East Asia, as well as a fascination for colour, both pyramidal and natural.

At his studio in Fife, Scotland, he innovatively combines the traditional goldsmiths’ materials of gold and silver with niobium, tantalum, titanium and zirconium to create truly unique pieces (see Figure 10).

Currently he is working on a sculpture of a small octopus; the wax design is ready, but he seeks a partner who can cast tantalum. If you can help please contact director@tanb.org.

As well as art, James has drawn on his deep understanding of refractory metals to develop a side-business making implants for neurosurgeons working in the field of maxillofacial (jaw and face) re-constructive surgery.

Figure 10a-d: Details from tantalum and niobium bowls created by James Brent Ward. He also created the tantalum bowl on the front cover of this edition of the Bulletin (photos: Roy Pritchard)
Examples from numismatics and jewellery

Figure 11a,b,c: (left to right) Coins from Kazakhstan (tantalum-silver), Canada (niobium-silver) and Austria (niobium-silver)

Figure 11d: Coins from Luxembourg from the “Castles of Luxembourg” series in niobium-silver (2009 to date)

Figure 12: A selection of jewellery designed and made by Holly Yashi using niobium. A wide range of similar items are available to purchase at www.hollyyashi.com or at their shop in Arcata, CA, USA, which also offers tours and demonstrations of the electrolysis process they employ. Photos: Holly Yashi Design Inc.

Notes: Many people helped in the research for this article, generously providing photographs, advice and guidance on practical applications. Special thanks must go to Paul Lubitz at Holly Yashi Inc. and, of course, James Brent Ward whose tantalum bowl (see front cover) first sparked an interest in this subject a decade ago.

Further reading:

Holly Yashi Inc. shares many more photos of niobium jewellery and their production process at www.hollyyashi.com
There are many numismatics websites. One which offers many examples of niobium and tantalum coins is www.coin-database.com
The Goldsmiths’ Company website carries several examples of Mr Brent Ward’s work, www.thegoldsmiths.co.uk
Tantalum polymer capacitors capable of high temperature operation

Paper written by Antony P. Chacko, Qingping “James” Chen, Philip Lessner, Chris Stolarski, and Cristina MotaCaetano at KEMET Electronics Corporation and presented by Qingping “James” Chen on October 14th 2019, as part of the T.I.C.’s 60th General Assembly in Hong Kong. All views and opinions in this article are those of the authors and not the T.I.C.

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I. Introduction

Tantalum polymer capacitors are increasingly being used for applications demanding high reliability in automotive, medical, industrial, military and space systems. Using intrinsically conducting polymer as the cathode in tantalum capacitors has led to significantly lower equivalent series resistance (ESR) when compared to MnO$_2$ cathode systems [1-2]. In addition, tantalum polymer capacitors offer lower derating and benign failure mode. The high volumetric efficiency, stable temperature and voltage coefficients of polymer tantalum capacitors make them a preferred choice over ceramic and Al electrolytic capacitors in many applications. One concern of polymer capacitors has been the poor reliability in high humidity and high temperature environments. KEMET has recently developed several technologies to solve this problem and we were successful in developing polymer capacitors for high humidity and high temperature applications [3-5].

Conducting polymers such as Poly (3,4-ethylenedioxythiophene) (PEDOT) are known to lose conductivity under high temperature due to oxidation and loss of conjugation of the polymer [6]. Reed et al. [7] demonstrated that extended exposure of polymer capacitors to air (primarily oxygen) at elevated temperature resulted in oxidation of the polymer thus increasing the dissipation factor (DF) and ESR of the component. Young et al. [3] and Ye et al. [4] demonstrated enhancements to the component’s construction lowered the rate of polymer oxidation and allowed performance to be stabilized through the 1000 hours of testing. These technological advancements enabled 125°C capable polymer capacitors which could meet the AEC Q-200 requirements for the automotive industry in the past few years [3-4].

In recent years there has been significant increase of electronics content and passive components in automotive systems due to Advanced Driver Assist System (ADAS). Some of these automotive applications require polymer capacitors with temperature capability above 125°C. Other applications such as down-hole oil and gas exploration, geothermal, and aerospace also demand such high temperature performance. In this paper the recent development of tantalum polymer capacitors from KEMET capable of high temperature operation will be presented.

II. Experimental

A. PEDOT film fabrication and characterization

In situ oxidative polymerization of PEDOT was performed by polymerization of 3,4-ethylenedioxythiophene (EDOT) in the presence of iron (III) toluenesulfonate (oxidizer). Polymerization was completed by drying for one hour. The obtained film is in a doped state with tosylate ion (TOS) as counterion. The film from the pre-polymerized polymer (PEDOT: PSSA and additives) was prepared by casting the dispersion on a glass substrate and subsequent drying at 130 °C for 30 min. The sheet resistance of the films was measured by a four-point probe with a Keithley 2400 source meter.

B. Fabrication and characterization of capacitors with PEDOT cathode

Tantalum anodes were anodized in an aqueous solution of 0.1% of phosphoric acid at 80 °C. In situ PEDOT (PEDOT: TOS) cathode was prepared by oxidative polymerization of the monomer EDOT with iron (III) toluenesulfonate as the oxidizer. Pre-polymerized PEDOT (PEDOT: PSSA) cathode was fabricated by dipping the anodes in the prepolymerized PEDOT-PSSA dispersion. A conductive carbon particle filled layer and conductive silver particle layers were applied over the PEDOT layer followed by assembling onto lead frame (LF) using conductive adhesive.
adhesive on the negative terminal and resistance welding of the positive connection. These assembled capacitors were encapsulated in epoxy resin and further processed into finished products. Test samples were mounted to a test board using a lead-free solder reflow process (260 °C peak temperature). Capacitance, DF, ESR and DC leakage current of the capacitors were measured using Agilent E4980A Precision LCR Meter.

III. Discussion

A. High temperature stability of conducting polymers

Thermal stability of the conducting polymers depends on the dopants or counterions used in these materials. Dopants can be monomeric (low molecular weight) anions or polymeric (high molecular weight) anions. The chemical structure of the two types of conducting polymers commonly used in capacitors is shown in Figure 1.

![Figure 1: Structure of PEDOT:Tos and PEDOT:PSS](image)

PEDOT polymers with polymeric dopants such as high molecular weight polystyrene sulphonate (PSSA) have significantly increased temperature stability compared to conducting polymers with low molecular weight dopants such as tosylate (Tos) anions.

![Figure 2: Normalized resistance of PEDOT films on exposure to 85 °C in air](image)

Figure 2 shows a comparison of the thermal stability of the in situ (PEDOT: TOS) films compared to slurry (PEDOT: PSSA) films. X-ray Photoelectron Spectroscopy (XPS) studies showed loss of dopants for the PEDOT: Tos in situ films on exposure to 85°C environment [5]. The enhanced thermal stability of PEDOT: PSSA slurry film is attributed to the protective effect of PSSA shell around PEDOT. However, PEDOT: PSS based polymer films show a conductivity decrease under extended exposure at 150°C in air (see Figure 3). The protective effect of the polymeric shell of PSSA decreases as the temperature approaches the glass transition temperature (140-152°C) of the PSSA. XPS studies of PEDOT: PSS on thermal treatment suggest decreases in the surface PSSA concentration on heating [8]. A decrease in the protective effect and enhanced oxidation rate of polymer at higher temperature contributes to this decrease in conductivity.
B. Polymer oxidation and oxygen pathways in polymer capacitors

Polymer oxidation occurs when oxygen enters the capacitors and migrates into the conducting polymer through the epoxy molding compound (EMC) and conducting layers such as carbon and silver coatings. The primary pathways are those in the EMC through which oxygen from the environment enters the capacitor. Secondary pathways are those through which oxygen that has already penetrated through the EMC permeates into the conducting polymer.

**Primary pathways.** There are three primary pathways for oxygen permeation into the package. The first one is the gap between lead frame and EMC. The second pathway is through the defects such as pin holes, show-throughs, and cracks in EMC. The third is the oxygen diffusion through the bulk of EMC. Figure 4 illustrates these three primary pathways. Oxygen permeation occurs at a faster rate through the first two primary pathways because oxygen encounters no tortuous path. Depending on the adhesion of EMC to lead frame, these ingresses can be larger or smaller. Molding conditions and any residual volatiles in the cathode layers influence the defects’ size and concentration. There is a longer pathway for oxygen permeation through the bulk of EMC due to the hinderance by filler particles which make the oxygen path tortuous. Filler concentration and filler aspect ratio influence the tortuous path in EMC. The glass transition temperature of EMC also influences the oxygen permeation rate through the bulk of EMC at high temperature.

**Secondary pathways.** Once the oxygen enters the package through the primary pathways, it can permeate to the conducting polymer through several secondary pathways (Figure 5).

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Figure 3: Normalized resistance of PEDOT:PSS films on exposure to 85°C and 150°C in air

Figure 4: Primary pathways for oxygen permeation through (a) LF/mold epoxy egress; (b) defects in mold epoxy; (c) bulk of the epoxy molding compound.

Figure 5: Secondary pathways for oxygen permeation through (a) exposed conducting polymer areas (wire side surface not covered by carbon and silver); (b) delamination between polymer interfaces; (c) cracks in cathode layers; (d) the bulk of the silver and carbon layers.
C. High ESR shift in high temperature life test

Figure 6a shows the ESR of 150°C life test from one of the developmental experiments. SEM cross section of one of the high ESR parts was examined and is shown in Figure 6b. Oxygen permeation through the cracks in the cathode layers and subsequent polymer oxidation is responsible for the high ESR shift of this part.

![Figure 6: (a) ESR distribution of 150°C life test at 0 hour, 250 hours, 500 hours, 1000 hours; (b) SEM cross section of a capacitor with high ESR shift](image)

D. Development of new cathode materials

**Interlayer crosslinking.** KEMET’s patented new cathode material system involves cathode and protective coating materials which significantly enhance interlayer crosslinking of cathode layers, lead frame, and epoxy molding compound [9], [10]. This interlayer crosslinking decreases oxygen permeation through several of the primary and secondary oxygen permeation pathways. Crosslinks formed by the covalent bond between polymer chains provide a tortuous path for oxygen permeation. As illustrated in Figure 7, the oxygen permeation path becomes more tortuous as the crosslink density increases.

![Figure 7: Oxygen permeation through (a) non-crosslinked polymer chains; (b) partially crosslinked chains; and (c) more crosslinked polymer chains](image)

**Cathode coatings.** Oxygen permeation through cathode interfaces is high due to the gap between the interfaces. These gaps are generated by poor interfacial adhesion between the cathode layers or through delamination occurred during reflow. The new cathode materials improve interlayer adhesion through interlayer crosslinking [9]. Interlayer crosslinking is the crosslinking between a reactive group in one of the cathode layers and a reactive group in an adjacent layer (see Figures 8 and 9). Such interlayer crosslinks improve adhesion between these layers and prevent delamination during reflow. The reduced interfacial gap and higher crosslink density between the cathode layers significantly reduce the oxygen permeation rate.

![Figure 8: Oxygen permeation through non-crosslinked cathode layer interfaces and interlayer crosslinked cathode interfaces.](image)
Protective coatings. We have developed protective coating materials to address some of the primary oxygen permeation pathways [10]. As Figure 9 illustrates, these novel protective coating materials on the LF provide reactive functional groups to form interlayer crosslinking between the EMC and LF. This improves adhesion between the LF and EMC. The reduced interfacial gap and higher crosslink density between these layers significantly reduce the oxygen permeation rate, which is reflected in the excellent stability of the new polymer capacitors (Figure 10).

![Figure 9: Interlayer crosslinking between mold epoxy layers and lead frame with hydroxy and epoxy functional groups](image)

IV. Conclusions

Oxygen permeation into the conducting polymers through several primary and secondary pathways causes polymer oxidation leading to poor performance in high temperature life tests. We have developed new cathode and protective coating material technologies which provide interlayer crosslinking and strong interlayer adhesion between cathode layers and between epoxy molding compound and lead frame. This interlayer crosslinking significantly decreased oxygen permeation and thus polymer oxidation. This technology enabled 2000 hour 175°C capable polymer capacitors.

![Figure 10: Life test results of new capacitor at 175°C](image)
NORM transport: time to increase the exemption level

The safe transport of tantalum- and niobium-containing raw materials (which can be naturally radioactive) is essential to human life and health and the environment, and also to industry and society as a whole. However, the T.I.C. believes that the current cut-off (exemption) level of 10 Becquerels per gram (Bq/g) for transport of radioactive tantalum/niobium containing materials is set unrealistically low; at a level that is unnecessarily cautious and a detriment to industry. It is important to note that the exemption level has only been 10 Bq/g since 1996; before then it was 70 Bq/g, and in Brazil and many other countries 70 Bq/g is still considered as a safe and reliable exemption level for internal transportation. In recent decades consolidation in the global shipping industry has significantly decreased the number of shipping lines that accept Class 7 shipments, further increasing the importance of appropriate NORM exemption levels.

We propose that an increased exemption level of 30 Bq/g would be safe and sensible.

This issue is relevant because the International Atomic Energy Agency (IAEA) currently has a special working group that is examining this issue. The T.I.C. is part of this group, giving members the opportunity to speak directly to decision makers and help them understand the importance of this subject.

The special working group will make a recommendation at the next meeting of the IAEA Transport Safety Standards Committee (TRANSSC), currently scheduled for June 2020 (although at time of writing it seems possible that this meeting may be postponed due to the Covid-19 pandemic).

We are asking for your help to collect radioactivity data on tantalite shipments and write to your national regulators to explain why this subject is important.

Members and other interested stakeholders are invited to contact the T.I.C. at director@tanb.org or visit our website to learn how, together, we can make a difference.

There is a vital need for the IAEA to set a realistic exemption level which provides safety to human life, health and the environment, but at the same time does not throttle industry.

Your support on this issue is important and appreciated.

References
EPRM launches “Due Diligence Hub”

https://europeanpartnership-responsibleminerals.eu/

What is the European Partnership for Responsible Minerals?
The European Partnership for Responsible Minerals is a multi-stakeholder partnership established with the goal to create better social and economic conditions for mine workers and local mining communities, by increasing the number of mines that adopt responsible mining practices in conflict and high-risk areas (CAHRAs). The T.I.C. has been a member of EPRM since 2017 and has actively participated in the working group behind the “Due Diligence Hub”. The Due Diligence Hub is free to use thanks to the generosity of EPRM members and donors.

What is the “Due Diligence Hub”?
The Due Diligence Hub is a comprehensive resource centre for mineral supply chain due diligence that will be launched in April 2020. It was initially designed to help industry meet the requirements of the EU’s Conflict Minerals Regulation which require all importers to the EU of tantalum, tungsten, tin and gold (“3TG”) over 1000 kg per year to register their company and undertake due diligence on their supply chain. However, in line with the expanding scope of the OECD Due Diligence Guidance for Responsible Supply Chains of Minerals from Conflict-Affected and High-Risk Areas, the EPRM’s Due Diligence Hub will not be limited to 3TG in the foreseeable future.

How can responsible supply chains help my business?
Sourcing your minerals responsibly with effective due diligence is good business practice. Knowing your products, your clients and your suppliers will help your company manage risks, improve your operations, enhance your reputation and generate financial benefits. It will also help contribute to positive developments in the minerals sector. This is what due diligence is all about. It will also help European importers of 3TG to comply with the EU’s Conflict Minerals Regulation, which will become compulsory from January 1st 2021.

Who is the target audience?
The target audience for the Due Diligence Hub is small and medium sized enterprises (SME) directly or indirectly affected by the EU Conflict Minerals Regulation, those who seek guidance to the right resources, and those who strive to implement due diligence and need assistance. By connecting companies to resources in a straightforward and accessible way, the Due Diligence Hub helps smaller organisations to maximise their efforts toward their responsible sourcing goals.

How does the Due Diligence Hub work?
The EPRM Due Diligence Hub is an online digital platform that will provide tailored due diligence resources you need to successfully ensure that the minerals in your supply chain, especially tin, tantalum, tungsten and gold (3TG), are responsibly mined and sourced.

Here you will find:

- A step-by-step guide to conducting due diligence
- 139 international standards and guides classified by minerals and supply chain tier
- 16 case studies demonstrating how other companies undertake due diligence, to share best practice

The Due Diligence Hub will be hosted by the EPRM at europeanpartnership-responsibleminerals.eu.
Recycling e-waste: a report from ICPREM-2020

On March 8th-9th the International Conference On Purification and Recycling of Electronics Materials was held in Hyderabad, India. In attendance were Mr Vinod Kumar (Managing Director) and Mr Pranav Mathur (Executive Director) from T.I.C. member company Metallurgical Products India Pvt Ltd, who kindly sent us this special report (for information only).

ICPREM was organized by C-MET, India’s Centre for Materials for Electronics Technology, in Hyderabad, India to commemorate their annual foundation day (www.icprem2020.in). Hyderabad is the capital of southern India’s Telangana state and is a major hub for information technology, software and electronics manufacturing. The international list of speakers included Dr Animesh Jha, a former judge of the A.G. Ekeberg Tantalum Prize.

India has an impressive capacity for recycling waste electrical and electronic equipment (WEEE, or e-waste), however, it is notable that some 70% of the e-waste which gets processed in India has been imported from other countries. Informal ‘back yard’ operations do not always follow best practice and it is against this background that ICPREM aimed to promote organized and responsible recycling of e-waste in India by highlighting technologies available and government/private initiatives in this direction.

The growing importance of e-waste recycling

With depleting geological reserves of some minerals and the growing international pressure to minimise the environmental impact of mining operations, ‘urban mining’ and other forms of recycling are emerging as a recognisable global industry. According to the United Nations, in 2016, nearly 45 million tonnes of e-waste were generated around the world, or some 6 kg for every person on the planet. With increasingly wide spread usage and more frequent replacement of devices, huge quantities of such e-waste are created each year, presenting a strong case for recovery and re-use of the contained metals. In the future such operations could supply significant quantities of secondary raw materials to industry, including platinum, palladium, germanium, silver, indium, tin, copper, lithium, rare earths and tantalum.

Challenges to e-waste recycling

However, recycling e-waste is far from simple. E-waste comprises a wide variety of discarded electronic devices and components making it impossible to have a single, standardized recycling process. In addition, the quantity of valuable metals in each individual component varies widely and is usually imbedded under layers of various plastics, making their selective recovery a tedious and expensive process.

From identification and segregation of e-waste to finding a cost-effective and environmentally acceptable process for selective recovery of valuable metals and final disposal of the left-overs, there are many challenges to overcome. For example, e-waste can contain seven different types of plastics – each having unique properties. Simple incineration is not the answer; not only does it generate toxic fumes, but valuable elements can be lost in the process.

There can be no doubt about the importance of e-waste recycling, and the Government of India’s plans to improve the recycling industry are admirable. But industry has an important role to play too, and if manufacturers of the primary e-components can design their products keeping ease-of-recycling in mind, it would make recovery easier and be hugely beneficial to society at large. The story of optimizing urban mining has only just started.
Tantalum: a market overview by DERA

Paper written by Sophie Damm, German Mineral Resources Agency (DERA) at the Federal Institute for Geosciences and Natural Resources (BGR) and presented on October 14th 2019, as part of the T.I.C.’s 60th General Assembly in Hong Kong. All views and opinions in this article are those of the authors and not the T.I.C.

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Sophie.Damm@bgr.de, https://www.bgr.bund.de/

Introduction: DERA commodity monitoring

To increase market transparency and as the national information and consultancy platform for mineral raw materials, the German Mineral Resources Agency (DERA), within the Federal Institute of Geosciences and Natural Resources (BGR), provides information and commodity market research to help identify potential price and supply risks and assist with mitigation and supply strategies for mineral raw materials. Subordinate to the German Federal Ministry of Economic Affairs and Energy, DERA was established as a sub-department within BGR in 2010 by decree; DERA services are aimed at supporting the German economy as well as political stakeholders and include a mineral commodity monitoring and the evaluation of mineral resources. Detailed commodity risk analyses are an integral part of DERA’s mineral commodity monitoring program and include its criticality list published biannually with potentially critical mineral raw materials identified covered in comprehensive market research reports.

As a major industrialised and export-focused economy, the availability of mineral raw materials and their secure and sustainable supply is essential for the German economy. With a strong manufacturing industry at its backbone, Germany is a leading consumer of tantalum and tantalum-containing products. Without domestic tantalum mine production, the country is entirely reliant on imports of tantalum raw materials. Low recycling rates, its limited potential for substitution in key applications and a high share of global mine production concentrated in countries associated with high governance risks have increased the likelihood and severity of impact of tantalum supply disruptions on the German economy. Published in 2018, DERA’s commodity risk assessment aims to give a comprehensive overview of the tantalum market with a special focus on current and future supply.

Minor metals markets are traditionally opaque and price volatilities are more often than not attributed to speculation. A high market concentration and mining in countries associated with high country risks can pose additional challenges to a secure and reliable supply. Albeit associated with a low country risk, primary tantalum supply has long been highly concentrated, with mine production from conventional large-scale mining operations focused in Australia between the 1990s and the late 2000s. Since then the tantalum supply base has experienced significant shifts and a substantial increase in artisanal supply from Central Africa has resulted in an equally concentrated market, albeit with much higher associated country risks.

Figure 1: Tantalum price development 1976 – 2016
Tantalum consumption

Demand for tantalum has been relatively stable, with compound annual growth rates for the past ten years of about 1.4% per year. At around 50% of global consumption, the electronics industry is the leading application for tantalum where it is used in the manufacture of capacitors and sputtering targets. Tantalum capacitors are widely used in a range of consumer and communications electronics such as laptop computers, tablets and mobile phones as well as in medical and automotive applications. Tantalum capacitors have a high capacitance, and are the preferred choice of material in high-performance applications where component size and weight are crucial. Tantalum capacitors also played a vital role in an increasing miniaturization of devices. Increasing miniaturization of capacitors has resulted in less tantalum required per unit.

Tantalum-containing alloys accounted for just under one quarter of tantalum consumption, with superalloys as their main application. These are nickel-, cobalt- and iron-based alloys able to perform in high heat- and corrosion environments such as the aerospace and aviation industries in aircraft engines, in power generation such as land-based gas turbines as well as the medical, chemical and petroleum and gas industries.

Tantalum chemicals and mill products made up about one fifth of global consumption. Tantalum chemicals include tantalum oxides and compounds and form the precursors for a number of other applications including tantalum powders, tantalum metal and mill products. Mill products are used in the manufacture of tantalum components and thanks to their high corrosion resistance are used in heat exchangers, valves, tubing and piping for the chemical and pharmaceutical industries.

![Figure 2: Tantalum supply summary 2018](image)

Tantalum mine production

Global tantalum production was approximately 2,450 t in 2018, of which ~80% was primary and 20% secondary. (see Figure 2). Of primary production over 50% originated from the Great Lakes Region of Central Africa. Total African supply accounted for almost 70% of global primary supply and came almost exclusively from artisanal and small-scale operations based on extensive use of labour, low wages, and often with little to no mechanization and weathered ore bodies, thus producing at lower costs than conventional modern mining operations. Artisanal and small-scale mining of tantalum has long been established in developing countries, particularly in Central Africa and to a lesser extent in South America. Typically poverty-driven, it provides daily sustenance and a form of livelihood for large parts of the population. A significant proportion of the sector’s activities can also be regarded as informal and unregulated and its contributions to poverty alleviation and sustainable economic development are often limited.

In Central Africa, specifically in the Democratic Republic of Congo (DRC), tantalum mining has long been associated with rebel funding in the region. It should be noted, however, that tantalum is far from being the most important mineral mined in the DRC and tantalum mining in Eastern DRC, although potentially contributing to prolonging conflicts, is rather a symptom of the conflict than the cause for the conflict itself. Political instability and a weak governance of the country have been identified as the major factors to continue to hamper economic development of the artisanal and small-scale mining sector in the region. The tantalum supply base has witnessed substantial shifts in the last 30 years (see Figure 3).
Conventional mine production from Australia dominated tantalum supply up until the late 2000s until operations were forced to suspend amid weakening global demand resulting in low tantalum prices and rising operating costs. Conventional mines in Australia but also Canada and Mozambique have since closed and have been replaced by artisanal and small-scale mining predominantly in Africa. Overall, artisanal and small-scale mining accounted for close to 50% of tantalum supply in 2018 with over 50% of global supply currently from the Great Lakes Region.

Tantalum mine production - country concentration and weighted country risk

Country concentration for tantalum mine production can be expressed using the Herfindahl-Hirschman-Index (HHI). The HHI is a measure of market concentration and is defined as the sum of the squares of the market shares of each participant within a market (=tantalum mining country) where market shares are expressed as percentages. It approaches zero when a market is occupied by a large number of participants of relatively equal size and reaches its maximum of 10,000 points when a single participant controls the market. The HHI increases both as the number of participants in the market decreases and as the disparity in size between those participants increases. A market in which the HHI is between 1,500 and 2,500 points is considered to be moderately concentrated; a HHI in excess of 2,500 is considered to be highly concentrated.

Tantalum mine production has been ranging from a moderately to highly concentrated market (see Figure 4). At 74% of global tantalum mine production (excluding tin slags), Australian production reached its highest share in 1995, and is reflected in a HHI of over 5,700 points for that year.
The HHI remained in excess of 2,500 points until around 2006 when Australian supply decreased and was replaced by an increasing supply from artisanal sources from the Great Lakes Region and other African countries. The closure of conventional operations in Australia and Canada around 2009 and a subsequent increase in mine production from the Great Lakes Region led to a further increase and the high market concentration for tantalum mine production of today. Market shares are generally calculated on a country basis, and including individual data for Rwanda, DRC and Burundi yields a HHI of 1,930 points. While this is still a moderately concentrated market with the potential supply risks associated with it, this does not adequately reflect the current dominance of the Great Lakes Region for tantalum mine production. Therefore, for calculating market concentration of tantalum mine production, the region has been treated as a single producer, resulting in a HHI in excess of 3,000 points.

![Figure 5: Tantalum mine production world map 2018](image)

The weighted country risk of tantalum mine production was -0.66 in 2018 (see Figure 5). It is calculated as the sum of market share values of the countries in mine production multiplied by their country risk. The weighted country risk is usually an interval between +2.5 and -2.5, with values above +0.5 the risk is classified as low and values below -0.5 the risk considered as critical. The country risk is an aggregation of a set of six world governance indicators assessed and published annually for over 200 countries by The World Bank: 1) Voice and accountability, 2) Political stability and absence of violence, 3) Government effectiveness, 4) Regulatory quality, 5) Rule of law, and 6) Control of corruption.

![Figure 6: Weighted country risk development 1996 – 2018](image)
The high share of mine production in the Great Lakes Region contributed to the critically high weighted country risk of -0.66 for 2018. The region is associated with political unrest and the ranking of the DRC of -1.87 had a particularly negative effect on the overall weighted country risk. While the country concentration for tantalum mine production was equally high in the early 2000s (see Figure 4), at around 0.8 to 0.5 the weighted country risk was substantially lower (see Figure 6). This was due to a much lower country risk rating for Australia, the main producing country. With a declining Australian production and an increase in production in Central Africa, the overall weighted country risk declined from a formerly low-risk to a high-risk situation.

Market balance and outlook
An integral part of DERA’s commodity risk assessments, two scenarios were modelled that take into account additional production from announced expansions of existing mines and new projects as well as future supply from recycling and tin slags.

![Figure 7: Tantalum supply base - base case](image1)

![Figure 8: Tantalum supply base - high case](image2)
Tantalum supply from tin slags is assumed to remain constant at current levels; recycling rates are forecast to go up very slightly. Conventional operations are expected to increase tantalum output by expansions of existing operations as well as new mines coming into production. Tantalum as a by-product on the back of increased lithium mining, particularly from Australia but also Zimbabwe and Canada, could increase the share of conventional mining until 2026 and thus reduce future reliance on artisanal suppliers.

Historically, the market for tantalum has mostly remained in balance, with supply deficits generally compensated by an increase in mine production. Owing to its flexible nature, output from artisanal sources has proven to pick up quickly in line with demand, particularly over the past 20 years. Artisanal and small-scale mining of tantalum, particularly in the Great Lakes Region of Central Africa, will continue to play a major role in future supply and it is likely that any potential future supply deficits will be compensated by an increase in artisanal supply from the region. Changes in the geopolitical and economic framework of the region could, however, have a significant impact on the supply chain of tantalum and need to be closely monitored.

Forecast scenarios of future supply volumes are uncertain and subject a number of variables and should be interpreted with caution. Estimates for future supply from artisanal sources are based on historical production rates and are likely to depend on market conditions. Based on these findings, mitigation strategies such as long-term offtake agreements and a diverse supply chain for tantalum are recommended in order to help minimize exposure to supply shortages and price hikes and ensure a secure and sustainable supply of tantalum to the German industry.

Further reading
“Rohstoffrisikobewertung – Tantal”, the full report by DERA (in German) is available at https://www.deutsche-rohstoffagentur.de/DE/Gemeinsames/Produkte/Downloads/DERA_Rohstoffinformationen/rohstoffinformationen-31.pdf?__blob=publicationFile&v=2 or from the author, Ms Sophie Damm at Sophie.Damm@bgr.de.
Roskill’s new Tantalum: Outlook to 2029 report highlights the main factors affecting tantalum supply and how we expect it to change:

- What are Roskill’s forecasts for Australian and African supply to 2029?
- How will changes in the regional supply balance impact tantalum prices?
- Who are the major players at each stage of the supply chain and how does material flow through this complicated industry?
- Which are the largest applications for tantalum and how is the market for each expected to perform over this decade?

If you have an interest in tungsten and tantalum, register now to explore themes such as:

- Electronics trends—capacitors, semi-conductors and electrical contacts
- Commercial aerospace and military markets
- Additive manufacturing/3D printing technologies, applications and raw materials
- Automotive markets—electrification, on-demand and autonomous driving, and manufacturing tools
- Hardmetals and solutions for industry
- Recycling
- Responsible sourcing

15% discount for all T.I.C. Members

roskill.com
## Tantalum and niobium intellectual property update

This information is taken from the European Patent Office ([www.epo.org](http://www.epo.org)) and similar institutions. Patents listed here were chosen because of their apparent relevance to tantalum and/or niobium. Some may be more relevant than others. Note that European patent applications that are published with a search report are ‘A1’, while those without a search report are ‘A2’. When a patent is granted, it is published as a B document. Disclaimer: This document is for general information only and no liability whatsoever is accepted. The T.I.C. makes no claim as to the accuracy or completeness of the information herein.

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Member company updates

Changes in member contact details

Since the last edition of this newsletter the following changes have been made to delegate contact details:

- **Responsible Minerals Initiative (RMI):** The new delegate is Ms Catherine Tyson. Her email is ctyson@responsiblebusiness.org. All other details remain the same.

Diary of events to be attended by T.I.C. staff *

- IAEA’s 40th TRANSSC meeting in Vienna, Austria, June 1st to 5th 2020
- MIRU Tantalum Summit, Tokyo, Japan, June (tbc) 2020
- Tarantula (Month 12), Salamanca, Spain, June 4th to 5th 2020
- Preparing for the EU ‘conflict mineral regulation’ with Levin Sources, London, UK, June 23rd 2020
- **T.I.C.’s 61st General Assembly and AGM in Geneva, Switzerland, October 11th to 14th 2020**
- 4th Space Passive Component Days, Noordwijk, The Netherlands, October 13th to 16th 2020
- International Conference on Managing NORM in Industry, Vienna, Austria, October 19th to 23rd 2020

* correct at time of print

Join our mailing list to receive the Bulletin by email each quarter

Our mission with the Bulletin is to provide the global tantalum and niobium community with news, information and updates on our work. We hope you enjoy reading it! Recipients will also receive messages about the T.I.C. and our General Assemblies.

Email info@tanb.org to join our mailing list and keep up to date with the T.I.C.
Director’s Notes

London, UK

Dear T.I.C. Members,

Firstly, let me echo Dr Persico’s wishes that you, your family members, colleagues and friends are all safe, healthy and finding ways to get through whatever circumstances have been imposed on you as we fight this terrible virus.

At time of writing we still plan on printing and posting copies of this journal to members as usual. Please discard the envelope appropriately, but be assured that we will pack the magazines wearing gloves and a face mask, in line with current guidance. I hope you find this edition interesting and illuminating, both in the articles it contains and through the hyperlinks to patents and other external materials too.

In this challenging time, when many of us are travelling less, one way in which the T.I.C. can continue to promote tantalum and niobium is through our website and the publications we produce. To that goal, we are adding many more documents and links to our website, both in the public pages and the “members-only” area.

Also, in addition to this quarterly Bulletin, this month we will be publishing editions of our annual “Bulletin Review” in Chinese, Japanese, French and Portuguese, following the excellent feedback we received last year. Please share all Bulletins freely with anyone who shares our interest in tantalum and niobium.

Best wishes, and stay well,
Roland Chavasse, Director

Congratulation on HKSE listing to Guangdong Zhiyuan New Material Co., Ltd

On March 12th the parent company of T.I.C. member Guangdong Zhiyuan New Material Co. Ltd, China's Ximei Resources, listed on the Hong Kong Stock Exchange (HKSE) main board as listing 9936. Guangdong Zhiyuan New Material Co. Ltd is a producer of tantalum-niobium metallurgical products and recently was the platinum sponsor of our 60th General Assembly in Hong Kong.

The T.I.C. congratulates Mr Wu and all his colleagues at Guangdong Zhiyuan New Material Co. Ltd on this momentous occasion! Located in Yingde, Qingyuan, Guangdong Province, China, the company is the operating entity of Ximei Resources in mainland China. Further information is at http://www.zhiyuam.com/.
T.I.C.’s 61st General Assembly
(conference and AGM) will take place in
Geneva, Switzerland
October 11th - 14th 2020

Non-members are welcome to attend this event. The T.I.C. General Assembly attracts industry leaders from around the world. Full details will be made available online at www.tanb.org. Our 2020 conference will explore issues such as:

- Capacitors
- Superalloys
- Superconductors

And much more!

All questions about the General Assembly and requests for abstract submission forms should be sent to Emma Wickens at info@tanb.org. Full details will be published on www.TaNb.org and in future editions of the Bulletin.

The 61st General Assembly will include the award ceremony for the 2020 Anders Gustaf Ekeberg Tantalum Prize, the annual award for excellence in tantalum research and innovation.

This year our field trip will be to CERN, one of the world’s leading centres for scientific research (and a major user of niobium in super-conducting magnets!).
NINGXIA ORIENT TANTALUM INDUSTRY CO., LTD. (OTIC)

CLEAN TANTALUM SUPPLY CHAIN • RESPONSIBLE GLOBAL SUPPLIER

- Founded in 1965 (former Ningxia Nonferrous Metals Smeltery)
- 1052 employees
- The biggest Ta & Nb manufacturer in China
- Certified to CFSI annual audit successively since 2011
- Sourcing all non-conflict tantalum minerals in compliance with RBA requirements
- The first listed Ta & Nb processing company in China in January 2000
- The earliest TIC member in China and an Asian member of the TIC Executive Committee
- Executive member of Ta & Nb Branch of the China Nonferrous Metal Industry Association
- The Chinese National Ta & Nb Special Metal Material Engineering Technology Research Center
- Standardization leader of the tantalum and niobium industry in China

Key Product Show

OTIC specializes in producing Ta and Nb key materials for high-tech industry: powders, compounds and customer-specific fabricated parts of tantalum, niobium and their alloys such as wires, ingots, plates, foils, bars, rods, RF superconducting cavity, sputtering target blank, furnace parts and so on for various applications.

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