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To join the Bulletin mailing list email info@tanb.org
Dear Fellow Members,

As the Executive Committee, staff, and subteams work on the final phase of preparation for the 60th General Assembly (GA60), the market and regulatory supply chain landscape has been refreshingly boring. Needless to say, it would have been most welcome if the news reports about the activities in Hong Kong were as dreary as is our market. While Hong Kong may still be in the press at the time of GA60, I doubt there will be any impact on our conference. Hong Kong, where I am located now, is safe, functional, and transport is operational and proficient. I predict an enjoyable place to serve as host of our conference in October.

I have had the good fortune this quarter to visit a few of our important processor members. In July I went to see Guangdong Zhiyuan New Materials Co. Ltd. Zhiyuan has recently finished a state of the art, and incredibly large, hydrometallurgical processing facility. Not only does this facility take advantage of economies of scale, with its throughput volume, it employs very advanced and effective environmental mitigation technology. We sincerely thank Zhiyuan and its Chairman Mr Wu for his support of our GA60 as the Platinum sponsor.

Thanks and congratulations to Ulba Metallurgical Plant JSC for their upcoming 70th anniversary, which will occur right after our GA60. Ulba is not only a world class supplier of tantalum and niobium products, but has been a vital member of the T.I.C. since joining in 1993. Ulba has actively participated on the Executive Committee since 2009 where they have made meaningful contributions to all areas of our mandate including transporting NORM, supply chain and statistics. Ulba hosted a memorable 52nd General Assembly in the beautiful city of Almaty in October 2011 which all delegates that attended still remember. I am so appreciative for the invitation to the wonderful 70th Anniversary celebration which proved extremely rich in Ulba’s long history and was an example of the legendary Kazakh hospitality!

Congratulations to the winner of the Anders Gustaf Ekeberg Tantalum Prize 2019, who is announced on the back of this newsletter. It will be exciting to hear the winner’s presentation at our GA60 conference. Hong Kong is a wonderful, safe city. The hotel is excellent and there are minimal immigration issues entering Hong Kong for most passports. I look forward to seeing everyone in October.

Sincerely yours,

John Crawley
President
Dear T.I.C. Members,

Welcome to our latest newsletter, one which I sincerely hope that many of you are reading in Hong Kong, as delegates of a successful General Assembly! The tantalum market may be quiet at the moment, but I can assure you that the T.I.C. team has been working harder than ever before to ensure the recent events in that city don’t prevent us from holding our conference and AGM.

Earlier in the Summer it was my privilege to discuss the nature of interference colours with the artist and metallurgist James Brent Ward. For four decades he has been investigating the optical properties of thin oxide films on tantalum, niobium and titanium, with striking results. By controlling the voltage as he ‘paints’ with a brush dipped in electrolyte almost every colour can be created except red. His work, and the science behind creating these pigment-free colours, will be featured in a future edition of this newsletter.

As always there is a great deal going on at the T.I.C. to promote members’ interests and our elements. Many long term projects are reaching conclusion and we are moving forward from strength to strength!

I look forward to meeting you in Hong Kong.

Best wishes,

Roland Chavasse, Director

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**T.I.C.’s 61st General Assembly**

(conference and AGM) will take place in

**Geneva, Switzerland**

**October 11th - 14th 2020**

Full details will be published on [www.TaNb.org](http://www.TaNb.org) and in future editions of the Bulletin.
Reliable supply of tantalum, now and going forward

This essay is based on a presentation given by T.I.C. Director, Roland Chavasse, at the 2nd PCNS Symposium held in Bucharest, Romania, on September 10-13th 2019. It is for information only.

Introduction

Over the last decade supply chain risks in the tantalum industry have decreased considerably and, as a result, consumers of tantalum can have greater confidence in the reliable supply of tantalum, both now and in the future. This is an important subject, since not only is there a perennial stream of misinformed articles forecasting when society will "run out" of various minerals, but because long term trends in the electronics market forecast increased demand for tantalum-based capacitors in several key applications.

1. Tantalum resources around the world

The first point to appreciate about tantalum (Ta) is that while it is an undeniably rare element, there are sufficient likely reserves to satisfy demand for many years to come.

Tantalum exists in the Earth’s continental crust at a similar concentration to uranium, tungsten and molybdenum – a little under 1 ppm. Tantalum-bearing minerals are usually found within an igneous host rock where they form small grains of complex polymetallic oxides that typically amount to no more than 0.05% (Ta₂O₅) of the total mass. Most tantalum is refined from tantalite-columbite (colloquially 'coltan'), but microlite, wodginite and strüverite are important minerals too (figure 1).

Tantalum-bearing minerals are widely distributed across the world. A comprehensive survey in 2010 estimated the 'likely' reserves are around 318,000 tonnes of Ta₂O₅, with the largest likely resources found in South America and Australia (figure 2). However, it is important to note that not all the underlying data was to JORC standards and in central Africa the geological record is incomplete and so this estimate certainly underestimates the total global reserves. It is a well-recognised fact that African production of tantalum ore has been historically underreported and geologically underestimated, and it could account for up to 20% of world resources.

Even allowing for under-reporting of resources in central Africa, the knownlikely resources are equal to at least 100 years at current levels of consumption. Very few other commodities have that sort of resource base.

Figure 1: Important tantalum-bearing minerals

Tantalite-columbite (Mn,Fe)(Ta,Nb)₈O₂₄
Microlite (Na,Ca)₂Ta₂O₆(O,OH,F)
Strüverite (Ti,Ta,Nb>Fe)₂O₆
Wodginite Mn₄(Sn>Ta,Ti,Fe)₄(Te>Nb)₈O₃₂

Figure 2: Estimated likely tantalum resources around the world (after Burt, 2016)
2. The changing context of tantalum supply since 2000

Historically tantalum supply was dominated by a handful of large mines, the largest of which were in Australia, with others located in Africa, Canada, China and Russia. For several decades up to 2008 around half the global supply was produced by just one company in Western Australia called Sons of Gwalia. They were both the single largest producer and the most stable producer.

For many years Sons of Gwalia sold tantalum concentrates from its two tantalum mines, Wodgina and Greenbushes, on long-term contracts to refineries, but in 2004 Sons of Gwalia was driven into administration as result of mistakes made by their gold-mining division3. The tantalum mines were bought by a venture capital fund and production continued much as before, even as mining moved from open pit to underground operations, which are generally much more expensive to operate. Business thrived for a while, but by 2008 rising costs and falling ore grades forced Sons of Gwalia to suspend operations, and total global tantalum supply fell dramatically.

The sudden fall in Australian output created an opportunity for both the established industrial mines in Brazil and elsewhere, and the artisanal and small-scale mines (ASM) in central Africa. Africa has always been an important, although under-reported, producer of tantalum units and since 2010 an increasing amount of material has come from central Africa, much of it from ASM. This trend has steadily increased as mineral traceability programmes have become established in central Africa during the last decade.

What allowed artisanal and small-scale mines in central Africa to take off so quickly was that, in comparison to the high cost of hard-rock mining in Australia and Brazil, the deposits in central Africa are highly weathered and this makes them relatively soft. When rock is decomposed by natural processes over time the tantalum-bearing tantalite crystals remain largely intact. Such rock can be processed with a straightforward washing process and yield up to 85% of the tantalum units, a far higher recovery rate than the 50 to 60 % recovery rates typically achieved at hard-rock mines that must blast and crush the host rock to access the tantalum minerals (figure 4).

An artisanal mine may only produce a few tens of kilos of ore per week. While none of these artisanal and small-scale mines are, of themselves, of any significance, as a whole they make up an important part of the market, in that they can come ‘on stream’ or shut down at a moment’s notice, increasing supply as soon as prices rise and vice versa. In doing so, they further add to market stability2.

That is, of course, only half the story of central African artisanal and small-scale mining. The other half concerns the conflict-mineral legislations and mineral traceability programmes that created a framework for supply chain due diligence that was able to stabilise tantalum supply in central Africa. Due diligence is essentially the process of confirming that the minerals and metals you buy have been mined and processed responsibly.
3. How due diligence stabilised tantalum supply from central Africa

Due diligence and mineral traceability programmes are of the utmost importance to the tantalum industry.

The foundation stone is the OECD’s Due Diligence Guidance for Responsible Supply Chains of Minerals from Conflict-Affected and High-Risk Areas, now in its third edition. The key piece of legislation supporting the adoption of OECD’s guidance was section 1502 of the US Dodd-Frank Act, first implemented in 2012 and still operational today. This act requires US companies that file with the SEC to confirm the conflict-free status of any tin, tantalum, tungsten and gold minerals (so called “3TG”) that are sourced from the Democratic Republic of Congo or its neighbours.

In order to meet the requirements of Dodd-Frank the leading electronics companies established the Conflict-Free Sourcing Initiative (CFSI) and became willing, powerful allies to the central African mining companies and 3Ti industry in their drive to establish mineral traceability programs to monitor minerals entering their supply chains.

It was quickly recognised that smelters and refiners were the pinch point in the supply chain and if they could be shown to be conflict-free, then their downstream customers would also be conflict-free. Smelter audits were set up by the Responsible Minerals Initiative (formerly CFSI) and the tantalum industry quickly adopted them as standard (figure 5). Note that the decline in 2017 and 2018 was due to the Chinese government closing several plants due to environmental concerns. The partnership between the tantalum industry and the electronics industry has been a powerful combination for success.

![Diagram of mineral supply chain with smelters, mines, and users/consumers]

**Figure 5: Mineral supply chain smelter audits**

To support the smelters, on-the-ground programs were needed to monitor conditions at mines and provide mineral traceability data and risk reporting to the smelters for their audits. Today the largest program is called ITSCI and it grew out of initiatives started in 2009 by the tantalum and tin industries, once again tantalum showing leadership in this field. ITSCI is governed by the tin and tantalum trade associations and managed on the ground by Pact and a network of several hundred government employees, local civil society and business organisations. Since ITSCI was established several other programs have sprung up, including TI-CMC, run by the tungsten industry. Today central Africa provides legitimate and ethical tantalum, which is cost competitive as a result of the nature of the highly-weathered deposits found there. ITSCI works across four central African countries. The territory it covers is similar in size to Germany, the US state of California or China’s Sichuan province, but with considerably more challenging logistics. And yet it works because it has the buy-in from the governments, businesses and communities who host it.

ITSCI covers over 2000 mines, giving gainful employment to around 80,000 miners, and supplying over 2000 tonnes of tin, tantalum and tungsten minerals per month, all of which provides considerable tax revenues for the local and national governments in the region.
Clear proof of how mineral traceability programs have created a stable business environment in central Africa can be seen in the investments being made across the region. What we are seeing on the ground is that, as a direct result of ITSCI and the other programs offering a legitimate route to market, the artisanal mines are investing to become small mines or even semi-industrial mines as their business develops. For example, the mine shown in figure 8 joined ITSCI in 2011 as an artisanal site, and by 2015 had invested in a mechanized washing facility. Meanwhile, near Kisengo in southern DRC mines have invested in heavy earth-moving equipment and industrial washing facilities (figure 7).

Figure 7: Stability brings investment and allows mines to invest. Case study of Kisengo Mining Company (source: MRI)

Figure 8: Stability brings investment and allows mines to invest. Case study of Musha Kirimbari Busoro in Rwanda, 2011 vs. 2015 (pictures Pact/ITSCI)
This investment would not have happened without industry having the confidence that central Africa has become predictable and stable; a belief that has been reinforced by the peaceful elections held in the DRC last year.

Participants at every level appreciate that legitimate, conflict-free trade is the only way to gain access to the global marketplace. Mineral supply chains in central Africa today are responsible by choice. They operate mature and robust traceability systems, and this is likely to only improve further in the future with the introduction of new technologies such as blockchain to mineral traceability systems.

4. Due diligence developments in the future

On January 1st 2021 the European Union’s (EU) conflict mineral regulation will come into force, a major piece of legislation built around the OECD’s due diligence guidance. The EU regulation will apply to imports to Europe of tantalum, tin, tungsten and gold from anywhere in the world, not just the DRC and neighbouring countries. This regulation will further normalise mineral traceability in supply chains.

For over a year already, the T.I.C. is proactively working with the European Partnership for Responsible Minerals (EPRM), the European Commission and other key stakeholders to ensure there will be adequate support for those importers that will be impacted by the regulation. We are involved in developing EPRM’s knowledge portal and have invited the European Commission to speak at our 60th General Assembly. The T.I.C. is also working closely with the British government, which at time of writing intends to run a duplicate conflict mineral regulation in the UK, should it leave the EU.

Further information about regulatory requirements will be made public on the official “Due Diligence Ready!” website from November 20th 2019. Information will be available in seven languages.

The EU regulation is confirmation that mineral traceability systems are robust, mature, and here to stay.

5. Increased diversification in global production

It is widely held by tantalum industry analysts that by 2025 sources of production will have further diversified and the market share of central African production will have fallen relatively, if not absolutely. In particular, it is expected that production will increase from both Brazil and Australia.

In Brazil two important mines hope to increase production and a number of new players are investing in lithium mines which could produce tantalum as by-product. AMG Mineração’s Mibra mining operation has now been fully rehabilitated following a damaging fire in early 2017 and their expanded lithium production could generate additional tantalum units.

Meanwhile Mineração Taboca, the Brazilian subsidiary of the Peruvian mining giant Minsur, plans to expand activity at its Pitinga mine and increase its production of niobium-tantalum ferroalloy. Both Mibra and Pitinga also contain significant tin content. Some analysts are forecasting that by 2024 South American supply could have doubled its global market share, to over 30%.

In Australia rising demand for lithium to produce batteries for electric and hybrid vehicles is indirectly resulting in increased tantalum production. Traditionally most lithium has been produced from South American brines, but producing lithium from brine is slow and can’t easily be increased.
Australia has several world-class hard-rock lithium reserves and since 2014 investors have scrambled to bring mines into production. Many of these deposits also contain tantalum and global trade data has shown a significant growth in Australian tantalum concentrate exports since 2017. What impact might this tantalum by-product opportunity have on regional market shares of production going forward?

A forecast made by Roskill Information Services anticipated a relative reduction in artisanal mining from 43% of total new supply of tantalum in 2018 to 26% by 2023, through a combination of increased production from established industrial mines, increasing production as a by-product of lithium mining, and the (semi-)industrialisation of former ASM operations in central Africa.

Increased diversification in both where and how tantalum production occurs will contribute significantly to the stability of the tantalum supply chain over the long-term.

6. Conclusion

For the past decade, the tantalum industry has been a global leader in developing and operating mineral due diligence processes, resulting in stable, reliable production of conflict-free minerals from central Africa. Furthermore, with increasing tantalum production from both Brazil and Australia geographical diversity of production is also increasing. Over the last two decades risks of disruption to the tantalum supply chain have decreased considerably and, as a result, consumers of tantalum can have much greater confidence that their requirements will be provided for, both now and in the future.

Artisanal mining is highly responsive to market prices
Mineral traceability systems are mature and robust
The EU regulation will increase global due diligence

Tantalum minerals are found around the world
Brazil and Australia are increasing production
Tantalum supply is stable and reliable

Figure 10: Pilbara Minerals’ mine is just 5 years old. As of September 2018 the JORC Mineral Resource estimate was 226.0 Mt at 1.27% Li_2O and 116ppm Ta_2O_5 (photo: Pilbara Minerals)

Figure 11: Tantalum supply is becoming more stable and reliable

Further reading:
5. The ITSCI Programme, https://www.itsci.org/
9. AMG Mineração, http://amglithium.com/lithium-project/

Disclaimer: The information contained in this presentation is for general information purposes only. The information is provided by the T.I.C. and whilst we endeavour to present information that is correct, we make no representations or warranties of any kind, express or implied, about the completeness, accuracy, reliability, suitability or availability with respect to the information, products, services, or graphics contained in this essay.
A kilo of tantalum in my pocket

In Bulletin #178 an article asked the question “Is pure tantalum (Ta) radioactive?” and it was proven that the answer is a categorical “no”. However, the author of that article, docent Mark R. StJ. Foreman PhD ARCS BSc CChem MRSC, an associate professor in the department of Chemistry and Chemical Engineering at Chalmers University of Technology in Sweden, goes further to consider how much pure tantalum might generate a dangerous quantity of radioactivity.

Dr Foreman represents Chalmers University on the Tarantula Horizon 2020 program (with which T.I.C. is also involved). This article is for information only and all opinions it contains are those of the author and not the T.I.C.

Introduction

The International Atomic Energy Agency (IAEA) is the world’s central intergovernmental forum for scientific and technical co-operation in the nuclear field. It works for the safe, secure and peaceful uses of nuclear science and technology, and the safe transport of radioactive materials (under TRANSCC, where T.I.C. holds observer status).

Part of the IAEA’s effort to protect society from the harmful effects of radiation includes considering the question of “what is a dangerous quantity of a particular radioactive material?” (“D-values”).

They consider a series of scenarios which include the external exposures caused by someone putting non-dispersed radioactive material in their pocket for ten hours (the “pocket” scenario) or staying one meter from it for 100 hours (”room” scenario), the D1-values.

They also consider someone taking a given fraction of the radioactivity from dispersed material and either swallowing it, inhaling it, smearing it on your skin or immersing themselves in a cloud of the radioactivity (respectively, the “inhalation”, “ingestion”, “contamination” and “immersion” scenarios), giving D2-values.

Now before we get going it is important to keep in mind that the IAEA document is deadly serious, this is not a joking matter. The health outcomes which the IAEA consider range from death to injuries which would change your life forever.

These are bad situations; the sort of thing which I hope will only be encountered in a textbook or museum.

Tantalum metal and the “pocket” scenario

For the pocket scenario IAEA considers how much radioactivity would need to be carried in your pocket for ten hours to cause a radiation burn.

No value is offered for $^{180m}$Ta for either external exposure (D1) or exposure to a dispersed source (D2). But for $^{182}$Ta, an artificial radioisotope, they do set a D1 value of 60 GBq. They also have a mass limit of 500 grams for the object which goes in your pocket. They assume a dose of 25 Gy will be needed to cause a serious radiation burn.

Using D1 values for other radionuclides and some literature data it is possible to estimate what should be the D1 value for $^{180m}$Ta. I came up with 393 GBq.

Tantalum’s natural isotopes

Tantalum naturally occurs in the Earth’s crust in two stable isotopes, $^{181}$Ta (99.989%) and $^{180m}$Ta (0.0120%)$^6$. The $^{181}$Ta isotope is completely stable and non-radioactive.

The $^{180m}$Ta isotope is the rarest naturally occurring quasi-stable isotope and the longest-lived metastable state ever discovered. Its possible decay via the $\beta^−$ or the electron capture channel is so slow that it has never been observed and therefore natural tantalum emits negligible radioactivity (in fact less than table sugar, see Bulletin 178). The half-life of $^{180m}$Ta is longer than $4.5 \times 10^{16}$ years$^6$. 

$^6$They indicate no mention of the half-life of $^{180m}$Ta, which are very precise. The half-life of $^{180m}$Ta is longer than $4.5 \times 10^{16}$ years.
Now if we consider one kilo of tantalum metal which has been left outside to be exposed to cosmic ray neutrons before being rolled up into a ball. If you make this rather expensive ball, then it would contain 0.00044 Bq of $^{180m}$Ta and 0.92 Bq of $^{182}$Ta.

Based on these calculations I have calculated that the radioactivity in this kilo of tantalum is 65 thousand million times too small to cause the nasty radiation injury which the IAEA is concerned with.

For natural tantalum metal to reach the IAEA’s dangerous quantity (D1) limit you would need to have 65 million tonnes of it in your pocket.

The news gets even better, the IAEA document limits the mass of the radioactive material that a person might carry in their pocket to only 500 grams. They reason that this is a reasonable limit for what can be put in a pocket. When the amounts required to cause dangerous radiation levels in the pocket and room scenarios are above 500 grams and one tonne (t) the IAEA workers do not set a D1 value. This is because they believe it is impossible to reach dangerous levels of radiation in these scenarios.

But lets for a moment ignore the mass limit and see how much tantalum we would need to reach the D1 limit based on the pocket scenario. I would need to put 65 million tonnes of tantalum in my pocket. Now if I was to do this then the radiation level would never reach the dangerous level required to burn me, this would be because the vast majority of the radiation would be adsorbed in the gigantic bulk of the metal.

I am unsure of exactly how much tantalum exists in the Earth’s lithosphere, but I am sure that a single person collecting so much would cause a fluctuation of the price. Furthermore the lump would tear a hole in my clothing and would alter the shape of the ground below my feet.

I considered the idea of a mobile phone casing made from one kilo of tantalum. I calculate you would need to keep the phone in your pocket nonstop for about 7400 years to give the skin near it the equivalent to about one year’s worth of background radiation. I think that the take home message is that tantalum metal from a radiation point of view is safe to handle, and we should consider it as non-radioactive.

References:

i  EPR-D-values, Dangerous quantities of radioactive material (D-values), IAEA, 2006

ii J. R. de Laeter and N. Bukilic, Isotope abundance of $^{180m}$Ta and $p$-process nucleosynthesis, Phys. Rev. C 72, 2005
   https://journals.aps.org/prc/abstract/10.1103/PhysRevC.72.025801

iii B. Lehnert et al., Search for the decay of nature’s rarest isotope $^{180m}$Ta, Phys. Rev. C 95, 044306 (2017),
The ITSCI Programme aims to work within the OECD’s framework and to comply with the UN guidelines to create a system that assists companies with traceability, due diligence and audit requirements that arise from purchasing 3T minerals, particularly from the DRC, Burundi, Uganda and Rwanda. The T.I.C. and ITA (the tin association) sit on the ITSCI Governance Committee; there is also a third seat for a tungsten representative, but this is currently vacant. The ITSCI Programme has seen significant growth since the first pilot project was started in 2010; more mines to monitor, more processors to assist, more tags to monitor, more administration and inevitably more costs. Rigorous financial management is vital to the success of the Programme. Its costs are covered by four main sources of income: levies on minerals, ITSCI membership fees, donor payments (initially) and direct payments.

Although the day-to-day financial management, such as liaising with subcontracted field implementers, is the responsibility of the Secretariat (a role performed by ITA), the Governance Committee receives monthly budget updates and forecasts since several of its functions have a financial dimension, including the review of membership fees and mineral levies. ITSCI is financially separate from ITA, with its money held in trust. Both ITSCI and ITA are audited annually by Rayner Essex LLP, an independent chartered accountant. ITSCI publishes an annual financial summary and a statement from the auditor. This is a summary of the latest report, for the financial year January-December 2018. No external donor funding has been received by ITSCI since 2016.

### ITSCI in focus: lifting the lid on finances (2018 update)

![ITSCI logo]

The ITSCI Programme aims to work within the OECD’s framework and to comply with the UN guidelines to create a system that assists companies with traceability, due diligence and audit requirements that arise from purchasing 3T minerals, particularly from the DRC, Burundi, Uganda and Rwanda. The T.I.C. and ITA (the tin association) sit on the ITSCI Governance Committee; there is also a third seat for a tungsten representative, but this is currently vacant. The ITSCI Programme has seen significant growth since the first pilot project was started in 2010; more mines to monitor, more processors to assist, more tags to monitor, more administration and inevitably more costs. Rigorous financial management is vital to the success of the Programme. Its costs are covered by four main sources of income: levies on minerals, ITSCI membership fees, donor payments (initially) and direct payments.

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### Funding

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### Expenses

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<td><strong>7,996</strong></td>
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### Summary of annual funding and expenses (US$)

![Graph showing annual funding and expenses from 2010 to 2018]
Technology research on preparation of new type texture tantalum blank

Paper written by Li Zhao-Bo, NRMMI State Key Laboratory of Special Rare Metal Materials and Ningxia Orient Tantalum Industry Co. Ltd, and presented by Jiang Bin, Ningxia Orient Tantalum Industry Co. Ltd on October 16th 2018, as part of the Fifty-ninth General Assembly in Kigali, Rwanda. All views and opinions in this article are those of the authors and not the T.I.C.

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Ningxia Orient Tantalum Industry Co. Ltd, Shizuishan, Ningxia Province, China; www.otic.com.cn

Abstract

This article describes the influence of the texture component of a tantalum blank used for semiconductors on the sputtering rate of tantalum targets as it introduces a new type texture preparation of the tantalum blank. The research group used high purity tantalum ingot with a diameter of 330 mm. Through SPD forging, WR rolling and variable speed heating technology they produced 12-inch tantalum blanks. EBSD texture characterization was used to show the uniformity of the whole blank texture. The texture component of {100} or {111} is controlled in 25-35% and the texture component of {110} is less than 5%. The tantalum target with this new type texture blank ensures the uniformity of sputtering rate through target life and improves the sputtering efficiency. The blank can be used to produce 12-inch tantalum targets.

1. Introduction

With the fast development of semiconductor chip technology, integrated circuits began from a 10μm process, including 2300 transistors, and developed into 90 nm, 65 nm, 45 nm, 32 nm, 22 nm and 7 nm processes, including 6.9 billion transistors. The diameter of the wafer grew from 2 inches to the current 12 inches, and its integration level is much higher. For used material, it has much higher requirements. When development arrived at the 130 nm process, copper interconnection techniques combined with low dielectric coefficient media replaced aluminum interconnection step by step. Tantalum targets have been introduced into the semiconductor industry as thin-film material used for barrier layer preparation which prevent copper (Cu) atom diffusion into silicon (Si).

In preliminary stages for high purity tantalum metal blanks used for the 130 nm and 90 nm process, cold plasticity processing techniques are used with the primary technological level of internal structure is that the grain size is controlled within 75 um and the domination of recrystallization texture component is {111}/ND component. Currently, from the surface to mid-thickness, there exists a strong texture component {111} and gradual gradient. But because of line width of the 130 nm and 90 nm process is wide, the variation in the thin film thickness of the tantalum barrier layer has no obvious influence on electrical property of the entire film. During the tantalum blank sputtering process, sputtering speed conversion caused by the texture component and texture distribution gradient would be within the accepted limit.

When manufacturing technology of integrated circuit chips enters 65 nm and 45 nm processing, more advanced 32 nm, 28 nm, 22 nm, 14 nm and 7 nm processing has also entered volume production or research and development. As line width becomes more and more narrow, the variation in thin film thickness of tantalum barrier layers has obvious influences on the electrical properties of whole thin films and control of the variation in thin film thickness of the tantalum barrier layer is required. Tantalum blanks target texture component requirements are more reasonable, and distribution of texture component is more uniform. For target internal structure, it has new and more uniform requirements.

Currently, for high purity tantalum metal blanks of the 90 nm process, the texture component proportion and distribution are experiencing a non-uniformity problem. During the process of coating film, it appears that sputtering speed is not uniform, and will affect uniformity and consistency of film thickness. This will then affect the film electrical property thus not meeting the requirements of 65 nm and 45 nm technology node. Therefore, in order to meet requirements of tantalum barrier layer material of 65 nm/45 nm nodes and above, new processing methods need to be researched. For this reason, the need to adjust texture type and improve the uniformity of the texture component distribution becomes necessary.
Furthermore, considering selection of design parameters and thin film material properties of the tantalum barrier layer, and especially considering sputtering speed discrepancy of various kinds of texture component types, requirements for texture component type and proportion are put forward. It also is involved in texture type adjustment and control during the tantalum blank manufacturing process. In 130 nm and 90 nm processing, the dominant recrystallization texture component is \{111\}. A gradual gradient exists in thickness direction, but because its processing line width is broader than the 65 nm and 45 nm process, it still meets coating film requirements. But for the 65 nm and 45 nm processing, the dominant texture is \{100\} component, secondly is \{111\} component, and needs to be a homogeneous distribution without a gradual gradient. But because of tantalum’s characteristics, the slip system easily starts at \{111\} <111> and the dominant texture which is a easy to form \{111\} component with thickness direction gradient. Therefore, for the manufacture of barrier layer material having a dominant texture of \{100\} and \{111\} component, using the conventional cold plastic working method does not work and a new processing method needed to be developed.

As sputtering source material, the sputtering rate of tantalum blanks are different because of the different texture types based on the characteristics of tantalum crystal structure. The sputtering rate of \{110\} texture is the midrange with \{100\} being the highest and \{111\} the lowest. Meanwhile, uniformity and sputtering stability of tantalum blank texture is required to reduce deviation of film performance index and control consistency fluctuations throughout the life of the tantalum blank. So, the demand of tantalum blank texture component with reasonable proportion and distributive uniformity is needed in order to ensure uniformity, stability, sputtering yield, and reasonable sputtering efficiency during the sputtering process. Generally, the tantalum blank of a texture component of \{100\} or \{111\} controlled at 25-35% (and \{110\} to a lesser extent) can easily meet requirements of uniformity, stability and reasonable sputtering efficiency. OTIC has supplied 8-inch tantalum blanks for international and domestic target manufacturers for many years. In order to produce the most advanced 12-inch tantalum blank, change of texture type and improved texture distribution uniformity is required. OTIC uses the hot plastic processing method and introduced the SPD (Severe Plastic Deformation) method. OTIC also improved texture distribution uniformity by using the WR method and adjusting texture type to produce a new type 12-inch tantalum blank. EBSD texture characterization shows the uniformity of the whole blank texture. The texture component of \{100\} or \{111\} is controlled within 25-35% and the texture component of \{110\} is less than 5%.

**2. Texture control**

2.1 Influence of die SPD forging process on texture

EB smelting tantalum cast ingot is used because of the advantages of high purity, good densification, and is widely used as the raw material of tantalum barrier layer. EB tantalum cast ingot has large cast grain along its axial direction. Using 330 mm tantalum cast ingot as one example, the core section cast grain size can be achieved to 100 mm. The smaller cast grain size which is around core of the section also can be above 30 mm. (see Figure 1). This kind of coarse grain of EB tantalum cast ingot can cause issues with texture uniformity and component type control of tantalum barrier layer material. Although one large cast crystal can be broken into many small grains by the conventional cold forging method, its texture orientation distribution is very close. These many fine grains coming from this original large cast grain have close texture distribution. Therefore, it can cause a non-uniform texture distribution, although its grain size of tantalum blank is fine and small.

![Figure 1: Micro-metallography for cross section of tantalum cast ingot](image1)

![Figure 2: Sampling sketch map for tantalum blank after cold forging](image2)
For EB tantalum cast ingot, using the cold forging method due to tantalum metal characteristics, its atomic close-packed plane is \( \{110\} \), and atomic close-packed orientation is \(<111>\) and forms a slip system which is easily started at \( \{110\} <111> \). Other slip systems are not easy to start, and cause tantalum blank to easily form \( \{111\} \) texture component. Component occupied percentage can reach 70%. But in tantalum blank, the content of \( \{111\} \) texture component should be controlled strictly. Higher content can cause lower sputtering speed and then affect the electrical property of tantalum barrier layer thin film (see Figures 2, 3, 4).

Through changing the cold forging method, texture distribution uniformity is improved, and texture component type is adjusted. In the case of 8-inch tantalum blanks, 2 times cold forging method is used. Analyzing its texture grain and distribution, the results show the texture component is non-uniform but meets user requirements. In the case of 12-inch tantalum blanks, hot forging is used in order to improve texture uniformity and slightly adjust texture component type and adjust plastic deformation temperature. Heating the tantalum cast ingot to reduce deformation resistance of the tantalum cast ingot can increase forging permeability and start many more slip systems making the texture component more uniform. In the case of long-life type tantalum targets, in order to adjust distribution type of tantalum blank texture of the 12-inch tantalum blank, the die SPD technique was introduced (see Figures 5, 6). This technique helps to make tantalum cast ingot generate drastic plastic deformation and reduce slip deformation ratio during plastic deformation process.

It also increases the shear deformation ratio, improves grain interior sub-grain deformation ratio, makes different grain shift, changes grain orientation, and then reduces the content of the \( \{111\} \) texture component, and increases the content of \( \{100\} \) texture component. This makes for a good basis for further improving sputtering performance of tantalum barrier layer material (see Figures 7, 8, 9, 10).
Figure 7: IPF figures for forging structure and texture of tantalum blank

Figure 8: Sampling sketch map for tantalum blank after 2 times of hot die SPD forging

Figure 9: Statistical chart for tantalum blank texture component after 2 times of hot die SPD forging

Figure 10: 15° orientation figure of tantalum blank texture after 2 times of hot die SPD forging
2.2 WR process influence on texture

The rolling process is a key method of control of texture distribution uniformity and texture type during the process of tantalum blank preparation. The conventional process of using the flat cold rolling method can lead to huge rolling deformation resistance and cannot be rolled fully. On the rolling surface, it mainly is shear deformation, and the rolling center layer is mainly compressed slip deformation. From the surface to the center there exists a gradient distribution of slip and shear deformation causing non-uniform deformation mode along thickness direction. Because of cold rolling, the starting slip system mainly is \{110\} <111> causing non-uniform texture distributions of the tantalum blank along the thickness. The rolling center layer is \{111\} texture component, and \{111\} on the rolling surface. Sputtering speed is degenerative during the sputtering process. All of this will affect electrical properties of the tantalum barrier layer thin film (see Figures 11, 13, 15, 17).

Based on the 8-inch tantalum blank rolling process, the 12-inch long-life tantalum blanks process was changed to include hot WR rolling to improve texture distribution uniformity and adjust texture component type. Hot rolling reduces rolling deformation resistance, improves rolling permeability, and starts more slip systems. Changing flat rolling to WR rolling, (see Figures 11, 12), changes the form and stress state of the rolling deformation area and changes the mode of rolling deformation area while increasing the shear ratio to start more slip systems. It also can increase rolling permeability and make the deformation mode along the thickness direction more uniform. Finally, it makes texture distribution of tantalum blank along the thickness direction more uniform and reduces texture content, thus it meets the sputtering requirement of tantalum barrier layer material (see Figures 12, 14, 16, 18).

Figure 11: Pole figure and inverse pole figure of conventional rolled tantalum blank processed by cold forging

Figure 12: Pole figure and inverse pole figure of WR rolled tantalum blank processed by 2 times of hot die SPD forging
Figure 13: Statistical chart for conventional rolled tantalum blank processed by cold forging

Figure 14: Statistical chart for WR rolled tantalum blank processed by 2 times of hot die SPD forging

Figure 15: 15° orientation figure of conventional rolled tantalum blank processed by cold forging

Figure 16: 15° orientation figure of WR rolled tantalum blank processed by 2 times of hot die SPD forging
3. Texture control

The new type texture 12-inch tantalum blank produced by the research group has uniformly distributed whole blank texture. The texture component of (100) or (111) is controlled in 25-35% and the texture component of (110) is less than 5%. It ensures uniformity of sputtering rate and improvement of sputtering efficiency (see Figure 19).

4. Conclusion

(1) This forging technique makes tantalum cast ingots generate drastic and uniform plastic deformation. By the rolling technique, it can change deformation mode of rolling deformation area, reduce the texture content {111}, increase the texture content {100}. The texture component of (100) or (111) is controlled in 25-35% and the texture component of (110) is less than 5%.

(2) The new type texture 12-inch tantalum blank produced by the research group has uniformly distributed whole blank texture. It has the advantages of better uniformity, higher sputtering efficiency, longer life and lower cost.
The grave of Charles Hatchett gets renovated by the T.I.C.

In Bulletin #178 we described how in early June this year the T.I.C.’s Director, Roland Chavasse, and Technical Officer, David Knudson, had rediscovered the burial place of Charles Hatchett, at Saint Laurence’s Church in Upton-cum-Chalvey in Slough, a town about 30 km west of London in the UK.

Charles Hatchett (1766-1847) was an analytical chemist and the person who is credited for having first discovered niobium, albeit as an oxide. He lived almost his entire life in London and how he came to be buried in Upton-cum-Chalvey was something of a mystery which we will return to shortly.

During the July meeting of the T.I.C. Executive Committee, after all the main issues had been dealt with, conversation turned to Charles Hatchett and in particular the condition of his grave; there were cracks in the brick work, mortar that needed stabilising, plants growing up the side and decades of grime to be cleaned off. This didn’t seem an appropriate condition for the tomb of one held in such high regard by our industry and it was unanimously decided to commission restorative work to maintain the tomb for the future.

The team at St Laurence’s were immediately responsive and with the help of Allan and Julie James, and Reverend Alistair Stewart we were able to arrange for appropriate improvements to be made. Over the Summer permissions were arranged, specialist restorers contracted and the correct type of mortar established so that work could proceed (standard procedure when working in the grave yard of a 900-year old church).

The result has been a great success and will ensure that Charles Hatchett’s family tomb (the burial place of his mother, father and wife, as well as himself) can withstand the test of time for decades to come.

Charles Hatchett has been rediscovered by the parishioners and welcomed back to their community with gusto. To celebrate the restoration work on Charles Hatchett’s tomb the T.I.C. was invited to give a talk about Charles Hatchett at the church to interested members of the community. This was duly agreed and on September 15th the grave was unveiled to considerable local interest, with both the Mayoress of Slough and the local Member of Parliament in attendance. The church was full during the talk on Hatchett and there were many interested questions afterwards (including “why have I not heard of him before?”, good question, we are working on it!).

Photo: T.I.C.  … and after (September 2019)
Members of the congregation and local community celebrate Charles Hatchett at St Laurence’s Church, September 15th 2019.  
Left to right: Allan and Julie James, Roland Chavasse, Mayoress Councillor Harpreet Cheema, Rev. Alistair Stewart, Tan Dhesi MP, and friends (Photo: Eros Mungal)

How did Charles Hatchett come to be buried in Upton-cum-Chalvey?

Charles Hatchett was born, lived and died in London. He owned several large houses and workshops in London, two of which are still standing today. As a young man he spent several years travelling across Europe with his wife, selling his father’s luxury coaches to wealthy aristocrats (including to King George III of England, King Stanislaw II Augustus of Poland and the Empress Catherine the Great of Russia), but aside from those travels he appears to have lived exclusively in London, not Upton-cum-Chalvey. How then, did he come to be buried in a place some distance from his family and to where he appeared to have no family or commercial connections?

When we asked the vicar of St Laurence’s Church, Reverend Alistair Stewart, why Hatchett was in his graveyard he replied with a charming clerihew:

Charles Hatchett,
was not buried in Datchet*,
due to the opprobrium, of the discovery of niobium.

(*a small village next to Upton).

Further research was needed and with the help of Julie James we found the crucial clue; there was another famous 18th century scientist who was also buried at St Laurence’s Church, Frederick William Herschel (who, like many Germans living in Britain at the time, was known by his second given name).

William Herschel (1738-1822) was a German-born musician and astronomer who, working with his sister Caroline, constructed superb telescopes. In 1781 he discovered the planet Uranus, a revelation that made him famous overnight and saw him elected as a Fellow of the Royal Society, the elite British scientific association. Crucially, in 1782 Herschel was appointed to the position of Royal Astronomer, a position with a pension that was conditional on living near the royal castle at Windsor (just across the River Thames from Upton-cum-Chalvey).

Charles Hatchett, who joined the Royal Society in 1797, got to know Upton-cum-Chalvey through his visits to the Herschels and their observatory. The rural peace and quiet of the ancient church of St Laurence, with its idyllic views across the River Thames to Windsor, would have made a striking contrast to the noise, smells and bustle of life in London, and evidently made a big impression on Hatchett. Charles Hatchett’s parents were the first family members to be buried here, followed by his wife in 1837 and, finally, Charles himself in 1847.

If you would like to visit Charles Hatchett’s tomb, directions to St Laurence’s Church are available at www.saint-laurence.com or from the T.I.C. office.
Tantalum and niobium intellectual property update

Historically the T.I.C. reported those patents and papers that were relevant to the tantalum and niobium industries (2000-2007, available in the members’ area at www.TaNb.org). Information here is taken from the European Patent Office (www.epo.org) and similar institutions. Patents listed here were chosen because they mention “tantalum” and/or “niobium”. Some may be more relevant than others due to the practice by those filing patents of listing potential substitute materials. 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 contained here.

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<td>Tantalum powder, anode, and capacitor including same, and manufacturing methods thereof</td>
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<td>Carbon coated dual phase niobium metal oxide method of manufacturing the same and lithium ion battery having the same</td>
<td>KR20190091830 (A)</td>
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<td>Cesium-niobium-chalcogenide compounds and semiconductor devices including the same</td>
<td>WO2019156580 (A2)</td>
<td>QATAR FOUND [QA]; TRINITY COLLEGE DUBLIN [IE]</td>
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<td>Hybrid supercapacitor containing a niobium composite metal oxide as an anode active material</td>
<td>WO2019164561 (A1)</td>
<td>NANOTEK INSTRUMENTS INC [US]</td>
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Diary of forthcoming events to be attended by T.I.C. staff  
* correct at time of print

- RMI’s Annual Conference in Santa Clara, CA, USA, October 21st to 23rd 2019
- London Metals Week 2019 in London, UK, October 28th to 30th 2019
- IAEA’s 39th TRANSSC meeting in Vienna, Austria, October 30th to November 1st 2019
- EU Raw Materials Week, Brussels, Belgium, November 18th to 22nd 2019
- FORMNEXT, Frankfurt, Germany, November 18th to 21st 2019
- Mining Indaba, Cape Town, South Africa, February 3rd to 6th 2020
- Argus Metals Week, London, UK, February 17th to 19th 2020
- T.I.C.’s 61st General Assembly and AGM in Geneva, Switzerland, October 11th to 14th 2020

Member company and T.I.C. updates

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

- **Alliance Mineral Assets Ltd** has changed its name to **Alita Resources Ltd**. The email address for the delegate, Ms Claire O’Brien, is now claire.obrien@alitaresources.com.au and the website has changed to www.alitaresources.com.au.
- **Argus Media** has moved to Lacon House, 84 Theobald’s Road, London, WC1X 8NL, UK. All other details are unchanged.
- **B.W. Minerals (s) Pte Ltd** has moved to 9 Temasek Boulevard, 31/F, Tower 2, Suntec City, Singapore 038989. All other details are unchanged.
- **H.C. Starck Inc. / Fabricated Products** has changed its name to **H.C. Starck High Performance Metal Solutions (H.C. Starck Solutions)** and website to www.hcstarcksolutions.com. The company has also changed its delegate to Joseph Sheehan. His email is joseph.sheehan@hcstarcksolutions.com.
- **Imerys Ceramics France** has moved to 43 quai de Grenelle, 75015 Paris, France.
- **Mining Mineral Resources Sarl** has moved its office to 588 Route de Kipushi, Commune Annexe, Lubumbashi, Democratic Republic of Congo. All other details remain the same.
- **Mitsui Mining & Smelting Co. Ltd**: The delegate has changed to Mr Tatsuyoshi Sakada. He can be contacted on sakada@mitsui-kinzoku.com.
- **Roskill Information Services Ltd**: the contact email has changed to jessica@roskill.com.
- **ThreeArc Mining LLC** has a new delegate, Mr Dmitry Bovykin. His email is bovykin@gmail.com.
- **TVEL** has a new delegate, Mr Sergey Zernov. He can be contacted on zernov@tvel.ru.

Members of the Executive Committee of the T.I.C. 2018-2019

The Executive Committee is drawn from the membership and committee members may be, but need not also be, the delegates to the T.I.C. of member companies. The Executive Committee that was approved by the T.I.C. members at the Fifty-ninth General Assembly consists of (alphabetical by surname):

<table>
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<th>Name</th>
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<tr>
<td>Fabiano Costa</td>
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<td>John Crawley (President)</td>
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<td><a href="mailto:alexey.tsorayev@mail.ru">alexey.tsorayev@mail.ru</a></td>
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Of these twelve, Mr John Crawley was elected President of the T.I.C. until October 2019. The T.I.C. currently has the following subteams (chaired by): Marketing (Daniel Persico), Meetings (Candida Owens), Statistics (Alexey Tсораyев) and Supply Chain (John Crawley).
The Anders Gustaf Ekeberg Tantalum Prize

Winner 2019

The Anders Gustaf Ekeberg Tantalum Prize (‘Ekeberg Prize’), awarded annually for outstanding contribution to the advancement of the knowledge and understanding of the metallic element tantalum (Ta), has been awarded to Nicolas Soro and his co-authors for the paper ‘Evaluation of the mechanical compatibility of additively manufactured porous Ti–25Ta alloy for load-bearing implant applications’.

Nicolas Soro is studying for his PhD in ‘Additive Manufacturing of Porous Metals for Biomedical Applications’ within the group of Professor Matthew Dargusch at the Centre for Advanced Materials Processing and Manufacturing (http://ampam.mechmining.uq.edu.au/) of The University of Queensland, Australia.

The judges’ verdict

Announcing the winner, the independent judging panel stated that in choosing this paper, they took into consideration that the “advancement of knowledge and understanding of tantalum” should not be restricted to the scientific and research community but also benefit the general public.

The application of tantalum containing load-bearing implants, that can significantly improve the quality of life of recipients, was considered to have the greatest potential of all the submissions to enhance the reputation and recognition of the tantalum industry to the public.

The complete list of authors of the winning paper is Nicolas Soro, Hooyar Attar, Martin Veidt and Matthew Dargusch from the Centre for Advanced Materials Processing and Manufacturing (AMPAM) at The University of Queensland, Australia, and Erin Brodie and Andrey Molotnikov from the Department of Materials Science and Engineering at Monash University, Australia.

The panel wishes to congratulate all entrants whose papers are challenging the boundaries of current knowledge of tantalum, and which may well lead to significant breakthroughs into exciting new applications of the element.

The prize and award ceremony

The medal will be awarded at the T.I.C.’s annual conference, the 60th General Assembly, which will be held in Hong Kong, in October 2019.

The medals for the Ekeberg Prize have been manufactured by the Kazakhstan Mint from 100% pure tantalum metal refined by Ulba Metallurgical Plant JSC, Kazakhstan. For details of how to submit a publication for consideration for the 2020 Ekeberg Prize please visit the website www.TaNb.org.