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President’s Letter

Dear Fellow Members and Friends,

With autumn approaching, we are looking forward to welcoming you all to the 57th General Assembly in Toulouse. Participation rates so far are high, particularly for the special tour arranged at the nearby Airbus facility, where we’ve had to create a waiting list (although perhaps also due to the winery visit afterwards). Your Meetings Subteam, headed by David Gussack, has worked hard to develop a balanced line-up of interesting presentations and is planning ahead for next year’s meeting in Canada. Further details on the 58th General Assembly will be announced in France.

An important development in the last quarter has been the arrival of Dave Knudson as our new Technical Officer, reporting to Roland Chavasse. While further details on his appointment and background are available in a separate article, I would encourage our members to seek out Dave’s expertise and use his services for any technical queries you might have.

We want to demonstrate value for money to all our members and actively seek your input, suggestions, and criticisms please. Towards that end, after the presentation session ends, we will be having an open clinic for our members the afternoon of 18th October in Toulouse to meet with Roland, Emma and Dave, to listen to your concerns and recommendations.

It is gratifying to see that several individuals from some of our corporate members have recently announced their interest in standing as candidates for the Executive Committee at the General Assembly. Given a surplus of candidates versus available spaces, we will be having elections in Toulouse.

Also, at the General Assembly (or Annual General Meeting) and at a concurrent Extraordinary General Meeting, corporate members will be asked to vote on various other issues. These include such issues, for example, as an Anti-Trust Policy, ASM Code of Conduct (essentially replacing the Artisanal and Small-Scale Mining Policy adopted in Tallinn in 2009), and recommended changes to our Charter.

As your Executive Committee and T.I.C. staff have embarked on various initiatives and activities over the last year, further details are provided on some of these in this Quarterly Bulletin. In addition, more information will be given in Toulouse. As an example, as you'll see in one article below and to subsequently be expanded upon by Leah Butler at the General Assembly, some members of the T.I.C. have been working on a sub-team, led by EICC/CFSI, to improve and expand their definition of a tantalum smelter. Our objective is to have as broad a church as possible, so those of our members, who are interested, can be slotted into one of the categories for possible certification.

This is an example of the T.I.C.’s desire to build upon and establish better relationships with relevant partners— including companies involved in all parts of the Ta and Nb industries’ supply chains; various non-profit organizations, whether industry associations, such as EICC/CFSI and iTSCi/Pact, for example, or non-governmental organizations (or NGOs, such as Global Witness); various government agencies; and the trade press. We are also seeking to build the number of associate members we have, whether it's in the academic, government or NGO space.

The T.I.C. has embarked on a long-term education, promotion, and market development initiative for both tantalum and niobium. Our initial effort last winter was the Much Ado about Tantalum paper by Richard Burt, where we attempted to address a myth about location of Ta resources and its impact on future supply. Misinformation and myths about our industries can have a significant impact in a whole range of areas, whether it’s in circuit design to government stockpiling efforts to new mining projects, for example. As part of this effort on better information, we are seeking to improve our statistics, for instance.

I close by thanking the two members of our Executive Committee who will be stepping down in Toulouse, for all their good works and contributions over the years, whether it’s with their companies or for the T.I.C. Just as we had an interesting article on Dale Gwinnutt in our last Quarterly Bulletin, I trust you will find Bill Millman’s reflections in this issue to be thoughtful and illuminating.

Looking forward to seeing you all in Toulouse!

Best,

David Henderson

President
Director’s Letter

Dear T.I.C. Members,

Here at the T.I.C. there is a suggestion that Autumn is coming; tree leaves are turning yellow-red, schools have restarted and preparations for the 57th General Assembly are proceeding at full throttle and Emma Wickens deserves special mention here for doing vast amounts to keep our flagship event on course. Since the last Bulletin our new Technical Officer, David Knudson, has started and I am sure that you will join the Executive Committee and me in welcoming David to our community.

Those of you who are regular visitors to www.TaNb.org will have also noticed several changes over the Summer as we continue to build both the public and members-only sections of this resource. Recent additions in the members-only section have included:

- Photos from previous General Assemblies, including Penang, New York, York, Cape Town, Almaty and Lake Tahoe (see right).
- The agenda and supporting documents for the 57th General Assembly including the proposed Charter.
- Muito alarde sobre o Tântalo, a Portuguese translation by Breno Costa Rezende of Richard Burts. T.I.C. report Muito ad about tantalum. This is our first step in a strategy to educate and inform the world about our two elements in several languages.

Meanwhile, in the public section of our website we have added:

- Scanned copies of all 167 Bulletins dating back to issue #1 published in February 1975 (see left).
- A Frequently Asked Questions (FAQ) section, highlighting the services of our Technical Officers’ Q&A service.
- An application form for Associate Membership of the T.I.C., a category open to academic institutions, government bodies, non-governmental organisations (NGOs) and civil society. Several organisations have already applied and will be considered at the 57th General Assembly.

Our website is our public face to the world but it is far from being our only area of activity and of particular note is the proposed ASM Code of Conduct recently drafted by the Supply Chain Sub-team led by John Crawley. This document, together with the revised Charter and Antitrust Policy that are also for consideration at the 57th General Assembly, will ensure that the T.I.C. is equipped to serve its members and the wider tantalum and niobium industries for many years to come.

However, the T.I.C. is not just about the future and I note in passing that this year marks the 150th anniversary of Swiss chemist Jean Charles Galissard de Marignac’s discovery of a procedure to separate the oxides of niobium and tantalum employing their potassium double fluoride salts; potassium niobium oxyfluoride, K₂NbOF₇, has very high solubility compared to potassium tantalum fluoride, K₂TaF₇. Indirectly Marignac’s process demonstrated that columbium and niobium were the same element, thus starting a debate over naming-rights for element 41 that continues to this day.

I hope that you will agree that the Executive Committee’s strategy for growth, set out at the 56th General Assembly last year in Penang is starting to bear fruit and that you will share their vision of what this association is capable of achieving.

I am looking forward to meeting many of you in person in Toulouse. Always feel free to contact me.

Best wishes,
Roland Chavasse, Director

Open clinic at 57th General Assembly

During the 57th General Assembly the T.I.C. will be holding an informal drop-in clinic from 2.30pm to 4.30pm on Tuesday October 18th in meeting room "Bravo" of the conference hotel.

Please feel free to drop in for a cup of coffee and a chat about any aspect of the tantalum-niobium industry or simply to discuss the T.I.C.’s work and future plans.
Introducing David Knudson, T.I.C. Technical Officer

The Executive Committee and Director have great pleasure in introducing Mr David Knudson, the new Technical Officer to the T.I.C.

Mr Knudson has been in the tantalum industry for over 30 years, first spending 23 years at Cabot Corporation’s Boyertown plant (now part of Global Advanced Metals, GAM), before working for 8 years with Niotan Inc., and 3 years with KEMET Corporation. David began his career at Cabot as a Research and Development (R&D) Technician working on tantalum powder development projects.

After 13 years as an R&D technician, he was promoted to the position of Project Engineer, where he completed many projects associated with expanding plant capacity and automation of specific processes.

After two years as Project Engineer, he was then promoted to the position of Manufacturing Engineer responsible for the flake product line as well as the dry processing of tantalum powder products.

At Niotan, David was able to use his years of experience to commission the Niotan facility for production of capacitor grade tantalum powders. At Niotan, David held the positions of Project Engineer for 3 years, Engineering Manager for 3 years, and Vice President of Engineering for 2 years. After KEMET purchased the Niotan plant in 2011 David held the position of Engineering Manager. David joined the T.I.C. on July 29th 2016.

Technical services from the T.I.C.:

As a key member of the T.I.C. staff, the Technical Officer provides considerable behind-the-scenes support to the association in many ways, including by working closely with the Director and Executive Committee to execute the T.I.C.’s technically oriented goals and strategies.

The three key client-facing aspects to the Technical Officer’s role are:

- **Q&A**: To provide answers to technical questions from the members about (almost) any aspect of tantalum and niobium. If you have a question please feel free to ask the T.I.C.
- **Statistics**: To provide leadership in the development of global, credible, reportable industry statistics and trends in the tantalum and niobium supply chains.
- **Transport**: To promote a wider understanding regarding the transport of Naturally Occurring Radioactive Materials (NORM), including representing the association’s interests to stakeholders such as the International Atomic Energy Agency (IAEA) in Austria.

Mr Knudson will be based in Nevada, United States, and he can be reached on tech@tanb.org.

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**Statistics: new website update**


For Q3 the historical statistics forms will not be distributed and all T.I.C. members are requested to submit their data using the new system. If you have any questions at all please contact David Knudson, Technical Officer to the T.I.C., on tech@tanb.org.

Please do not send your trade statistics to the T.I.C. directly as we must have no access to members' data.

Members’ trade data is only seen by Miller Roskell Ltd, the 100% independent chartered certified accountant that has collected members’ statistics since 2015. Miller Roskell is a member of the Association of Chartered Certified Accountants, and is ruled by their client confidentiality rules.

Their website is www.millerroskell.co.uk.
Mr Millman has continuously served on the T.I.C. Executive Committee since the 36th General Assembly held in Goslar, Germany, in 1995. He has been President on three occasions (1998, 2005 and 2006) and will be stepping down from the Executive Committee this year at the 57th General Assembly in Toulouse, France.

Here Bill reflects on his career spanning over four decades in the tantalum and niobium industries.

My earliest recollection of encountering the exotic world of Tantalum and Niobium was upon my acceptance into a Student Apprenticeship with STC (Standard Telecommunications & Cable) in 1972, in Paignton, England. It was then that I learnt that tantalum was one of many forms of capacitors that were being manufactured at this site. This was in the era of vertical integration, where telecommunications companies felt the need to manufacture their own components. Thus, as telecoms were seen as national champions, and country monopolies, we had Ta capacitor makers running into the scores.

Then I was off to the wonderful world of academia at Plymouth University in south-west England, where I embarked upon a four-year degree course in new fangled Electronics - the market that was highlighted at the time as part of the 'white heat of technology'!

So newly minted as a freshly qualified fearless graduate I set out to learn everything I could around the world of capacitors. In the arrogance and inexperience of youth I felt that I would know everything worth knowing within two years, perhaps three at a stretch. Forty years later I can tell you I am still learning!

My first encounter with the T.I.C was when my then manager Robert (Bob) Franklin, a former T.I.C. President, informed us that we were to assist in a plant tour of the Paignton facility by delegates from all around the world, with interests in tantalum and niobium. This was 1980. So, the world was coming to our small town, as well as visits to tin mines in the next county, Cornwall. Then, as now, Tin and Tantalum were inexplicitly linked. The technical papers and presentation materials from this and other T.I.C. Technical Sessions were thrown like 'pearls to the swine' to us aspiring young engineers.

At this time, tantalum capacitors were limited to 'leaded' technology, that is parts with two solderable wire leads attached; specialist, high reliability types and very expensive, to be used in the growth industry of 'Telecommunications' as well as military parts, for which the technology emerged from Bell Labs in the late 1950s. Later something called the 'Personal Computer' was making waves and demand for components of all types was growing rapidly.

It was then, tantalum caught a lucky break, as it so often does - a new form of electronic component packaging, with solder attachment onto rather than into circuit boards had arrived - Surface Mount Technologies (SMT), where other, cheaper forms of capacitors could not withstand having their whole body experience the high reflow soldering temperatures. Ceramic and Tantalum capacitors were naturally capable of such thermal stresses over the most popular, lower cost, Aluminium types, which had a 'wet' electrolyte.

It was also at this time that I experienced the characteristic self-destructive nature of the industry in the face of great opportunity, Sprague and Kemet, then two of the largest tantalum capacitor makers, each decided to offer the world their own unique styles of SMD Ta capacitors.
By now AVX Corporation had acquired Ta capacitors to add to its MLCC offerings and through my role as the company’s T.I.C. delegate I was able to discuss this technology shift, from leaded to SMD, and what customers’ expectations were. Mr David Maguire, Kemet, told me forcefully, that their case sizes would be the world standard. I also spoke with Pete Maden, Sprague, who also was totally convinced that their own unique and different series of case sizes would become the world standard. This to me as a young, financially unwashed engineer was the wonder of the T.I.C. - I could actually meet and talk to these industry behemoths and gain insights unavailable anywhere else.

Of course STC (later to become I.T.T. and then AVX in 1987) also had its own, and different case sizes. Naturally, all of our Marketing ‘experts’ had surveyed their customers and understood what was required. It is great to be young, naive and ‘open’ to insights, so I convinced my seniors that a round of visits to the major consumers, including the automotive industry, would be useful. After many visits and discussions I then had to inform my management that not only were we backing the wrong horse but also the industry leaders were wrong.

This was a ‘career changing event! The message I received from our customers could not be clearer - we want one standard, multiple vendors and global supply. The only available source that met the first two criteria, at that time, was Japan. The Japanese capacitor makers had in a collaborate manner agreed a single standard.

Fortunately for me they did not shoot the messenger. Indeed, I then ended up owning the problem and changed our product offering to match this standard - named the TAJ series, the ‘J’ making it clear to all that it was ‘Japan’ sizes, and got a head start on the western competition.

I learnt very valuable lessons here - 1) Always listen to your customers and 2) NEVER believe Marketing folk and 3) Never fall into the trap that as the biggest you are always right. The adoption of a worldwide standard for SMD Ta capacitors took the brake off its adoption globally; demand exploded.

This brings us to the next big challenge of the tantalum capacitor industry - its cost relative to other capacitor forms. Here we had a coincidence of opportunities - if we could raise the CV/g of tantalum powders, we could reduce the amount of expensive tantalum required for a given capacitance and secondly we could shrink the anode size and offer smaller parts! - miniaturisation was becoming the ‘next big thing’ as electronics moved out of the office into the streets. Hand held electronics arrives.

I was informed by various ‘experts’ at that time that CV/g of tantalum powders could never exceed 10,000. Today we use powders in excess of 250,000. Here again, the Ta capacitor industry received another break - H. C. Starck development of the ‘Mg de-oxygenation’ process.
Again, all of these key players were active members and players within the T.I.C., enjoying the privilege of visiting their plants, talking to their engineers and scientists proved invaluable. AVX became a leader in the rapid adoption of these high purity and ultra-high CV powders, seeing its market share of tantalum capacitors rise from its original small 3% to today's number one single largest share by volume and value, boosted by huge investment in capacities world-wide to deliver SMD Tantalum and NbO parts.

Once again, the electronics industry came to the rescue of the Ta capacitor business. Something called the 'Internet', as old models of country teleco's champions were abandoned globally, led to boom in demand for Ta parts, leading to a peak demand during period 1999/2000 of 24 billion pieces annually. A number we have never approached again - due in large part to the other side in the 'cycle of life' in tantalum - its dysfunctional supply chain.

The scars left from the 'shortage' fears over the decades, still run deep even today. The other competitive forms of capacitors do not suffer from these fears - despite also having quite 'exotic' metals such as platinum, palladium and silver (remember the Bunker Hunt brothers?).

Concerns around availability, cost, and more recently 'conflict-free' status have relegated Ta in capacitors back to its lonely 1% share in volume terms in all capacitors consumed, albeit 15% by value. Use of Ta in all electronics has fallen from over 50% of total demand to now less than 40%. This is galling as it is as close to the ideal capacitor as nature allows. In 40 years I have seen four major booms and busts.

We can be all proud that the T.I.C. took head-on the challenges around 'responsible sourcing' of tantalite minerals even before US legislation around 'conflict-free' sourcing from central Africa was enacted.

Enlightened leadership from a number of T.I.C. Presidents, Executive Committee members and members led to the Ta industry being seen by the electronics/automotive and latterly aerospace industries, plus government entities, as progressive, proactive and engaged in solutions, when compared to Tungsten, Tin and Gold industries (the other 2'T's +Gold). Tantalum led from the front, particularly our smelter community and the tantalum capacitor makers, others followed.

Key to the strength of the industry is the T.I.C. and through it, its family of members, covering most if not all of its activities, and as with most families, its share of mad uncles/aunties, arguments, door slamming and reconciliations.

I have been immensely fortunate to have benefited from the long span of 'tribal elders' and a number of very special people, that like a moth to a flame are attracted to this crazy, unpredictable, maddening, fascinating industry. I love the term 'suffering from tantaliosis' - a self-confessed sufferer, from our President David Henderson. Most apt.

So after 40 years in the industry, nothing remains the same, but I now really understand the expression that 'the more things change the more they stay the same'.

Bill Millman
CFSI clarifies tantalum company types that are eligible for the CFSP audit

The Conflict-Free Sourcing Initiative (CFSI) has provided this clarification on the types of tantalum company that are eligible for audit under its Conflict-Free Smelter Program (CFSP). These clarifications were made with the direct input and review by the CFSI tantalum sub-team, consisting of CFSP tantalum smelters, CFSI members, and other companies in the tantalum supply chain. This is relevant to T.I.C. members since many of them already participate in the program and also because in the foreseeable future the Executive Committee plans to streamline members’ “activity” categories listed on the T.I.C.’s website to be in line with the CFSI’s definitions.

The CFSP audit program

The CFSP audit program is available to all Primary and Secondary tantalum smelters who voluntarily choose to participate. The CFSI has the discretion to determine whether a company is eligible to participate in a CFSP audit. All companies meeting the definition of Primary or Secondary smelter below are included within the scope of this audit. All smelting facilities within a group company that meets the definition of Primary or Secondary smelter will also be considered in scope for audit. Compliance to the CFSP protocol is determined at the level of the smelting facility.

Primary and Secondary tantalum smelters are defined as follows:

- **Primary smelter:**
  A company with one or more smelting facilities with the ability to convert any of the following into tantalum containing intermediates for direct sales or further processing into tantalum containing products:
  - tantalum containing ores (such as tantalite, columbite, etc.),
  - tantalum containing tin slags, and
  - tantalum concentrates (including synthetic concentrates)

- **Secondary smelter:**
  A company with one or more smelting sites with the ability to convert tantalum containing secondary materials into tantalum containing intermediate products, including synthetic concentrates, for direct sales or further processing into tantalum containing products.

Eligible companies may operate as either primary, secondary, or both types of smelters.

Companies outside the scope of the CFSP audit include but are not limited to:

- **Materials treatment specialists**
  Companies solely processing materials sent for external treatment are not within the scope of this audit. This form of external treatment must not include smelting or refining processes. For example, a materials treatment specialist might receive materials from, and return them to the smelter to remove hazardous waste contaminants (e.g. arsenic, radioactivity) as a service. Such materials, if continually owned by the smelter, will not require additional origin information on their return from such a company.

- **Trading companies**
  Companies trading in materials where there is no mechanical or heat treatment or other process performed, and the material traded is in the same chemical and physical state as received.

- **Tantalum intermediate processors**
  A company with one or more production sites with the ability to convert tantalum intermediate products (annex VI &VII) into tantalum containing products.

- **Recycler/Handler/Material recovery companies**
  A company with one or more sites with the ability to mechanically, but not thermally or chemically, process secondary materials using means such as shearing, cutting, sawing, shredding, briquetting/compacting, shot/sand blasting (wheel abrasive and pneumatic) and machining.

These categories are undergoing one final public review period before being finalized in the next version of the CFSP protocol. For further information, please contact CFSI staff at info@conflictfreesourcing.org.

**CFSI Annual Meeting and Conference 9th-10th November 2016**

This year the CFSI annual meeting and conference will take place at the Santa Clara Convention Center in Santa Clara, CA, United States on 9th-10th November. Register at [https://eicc.swoogo.com/cfspielac16/17716](https://eicc.swoogo.com/cfspielac16/17716).

This event brings together representatives of industry, government and civil society for updates, in-depth discussions and guidance on best practices to help companies make informed choices about conflict minerals in their supply chains. It will be held in the same venue as EICC Responsible Electronics 2016 (separate registration).
Letter from Rwanda: Progress by Ambition

Mirko Liebetrut of BGR, Germany’s Federal Institute for Geosciences and Natural Resources, explains the view from Rwanda of recent developments in supply chain of tantalum, tin and tungsten (3T) minerals. Mr Liebetrut is based in Kigali, Rwanda, and works closely with the Geology and Mine Department (GMD) of the Rwandan Natural Resources Agency (RNRA).

As commonly known, passing the Dodd-Frank Act section 1502 in the United States in 2010 put the world’s leading tantalite producing region under pressure; the DRC and neighbouring countries lost most of their western partners in tantalite trade, bringing the sector and reliant communities close to collapse. On the other hand, international attention was increased towards the Great Lakes Region (GLR) and its mining industry, creating various initiatives and different international cooperation. Even before Dodd-Frank, the UN Group of Experts on the DRC suggested developing a traceability system for 3T minerals in the GLR. Developments towards this were measurable, but little, lacking of a comprehensive region-wide chain of custody (CoC) scheme.

Rwanda is situated in the north-eastern part of the Kibaran belt, which hosts vast amounts of different metallic resources, mainly formed in granitic intrusions or pegmatites. Exports of 3T minerals provide a significant share to the nation's total export earnings (see figure 1). Hence, the country had a strong interest to implement a CoC systems and align with the OECD's Due Diligence Guidance, to meet international trade requirements. Rwanda's starting position was good; as well as the manageable size of the country and good infrastructure, its national legislation had already solved some issues like child labour and took further measures towards fair working conditions. Nevertheless, monitoring the mining sector and putting in place the necessary regulations has been rather a challenge and was managed outstandingly in regional context.

With strong partners, such as BGR, Partnership Africa Canada, and GIZ, the Ministry of Natural Resources (MINIRENA) was able to implement monitoring and inspection schemes. This was achieved through its Geology and Mine Department (GMD/RNRA), which followed protocols of the Regional Certification Mechanism (RCM) of the International Conference on the Great Lakes Region (ICGLR). More than 40% of ICGLR region-wide third-party audits have been carried out in Rwanda. On national level, GMD’s Certification Unit is supervising every 3T-shipment by exporters. Such shipments require the verification of tax payments and chain of custody documentation by the iTSCI Programme or Better Sourcing Program (BSP). The outcome of inspections by the Mining Regulation and Inspection Unit, which is capable of performing over 350 mine-inspections a year, is crucial to get permission to export. To enable this institutionalization of the certification process, an extensive amendment of national legislation has been performed.

Today, Rwanda’s mining sector is assumed to be the best monitored one in the GLR. Also, Rwanda is the only country where the 3T production is fully covered by chain of custody tracking schemes and all 3T exports must be accompanied by the conflict free mineral certificate of the ICGLR, which is OECD Due Diligence Guidance compliant. Even though tantalite exports have decreased slightly within the last years, a recovery and extension of the mining sector could be noted during first half of 2016 (see figure 2). National authorities are eager to develop the sector even further. The new mining standards, which are currently being developed, will excel international trade requirements and set focus on environmental sustainability and mine safety. Additionally, Rwanda’s deposit landscape is far from being fully explored, due to lack of high precision exploration activity. Some of the known deposits show extraordinary Columbite contents and are therefore not yet mined extensively. Regarding to the latest reports, an increasing interest of buyers in such niobium sources has already been noticed. With all this and the continuing international support, Rwanda will most probably stay a key player in supplying world’s tantalum and niobium consumption.

For further information visit the websites of BGR (www.bgr.bund.de/mineral-certification) or RNRA (www.mra.rw) or contact Mirko Liebetrut (Mirko.Liebetrut@bgr.de) or Joseph Kagabo (joseph.kagabo@mra.rw).
Superalloys: an introduction

T.I.C. member Roskill Information Services introduces the fundamentals of the superalloys industry.

Executive Briefing

Superalloys are a group of polymeric high performance alloys that possess very high melting temperatures, high strength, and considerable resistance to wear in corrosive and oxidizing environments. Because of their high strength and minimal thermal creep at high temperatures (above 800°C), the majority (85%) of superalloys find application as investment castings and directionally solidified and single crystal turbine blades in the hot sections of gas turbines used as aerospace engines and to generate industrial power (IGT). In 2015 the total superalloy sector consumed approximately 400t of tantalum and 2200t of niobium (excluding revert), some 20% and 4% of total production respectively.

Introduction

Superalloys are typically used in the hottest parts of gas turbines because they offer a unique combination of very high melting temperatures, high strength, and considerable resistance to wear in corrosive and oxidizing environments. They can be nickel-, cobalt- or iron-based alloys and will contain variable amounts of chromium, tungsten, molybdenum and a range of other elements, including tantalum and niobium. While the term superalloy is nominally reserved for those alloys which are used at service temperatures of above 800°C, in practice it is also used for alloys developed for corrosion and wear resistance. Superalloys were developed for applications where high tensile, thermal, vibratory and shock stresses are encountered and where oxidation resistance is frequently required, particularly in the aerospace and energy industries.

The development of superalloys has been driven by the requirements of gas turbine technology and still today the majority of superalloys are consumed in manufacturing gas turbine aero engines (55%) and industrial gas turbines (IGT) (30%), the latter used mostly for electricity generation, and also for powering terrestrial and marine vehicles (see Figure 1). The balance of superalloys are used by the petroleum and petrochemical industries as well as in space vehicles, nuclear reactors, and steam power plants. The superalloy market is a global one, with particular concentrations in North America, Europe, Russia, China, Japan and Australia. The USA and the EU produce most superalloys and also consume the greatest quantities, estimated at between two-thirds and three-quarters of total demand.

![Figure 1: World: Total consumption of all superalloys by end-use, 2014 (%)](Image)

Source: Roskill 2015
Development of superalloys

The gas turbine engine, with its high temperatures and stresses, is the dominant application that drives the superalloy industry, continually demanding that the performance boundaries be advanced.

The perpetual drive to create improved superalloys has seen a number of developments in both manufacturing techniques and elemental composition. Superalloys with the highest heat tolerance are used to manufacture turbine blades (rotating) and vanes (static) used in the high-pressure turbine (HPT) located in the hot section of a gas turbine engine, adjacent to the combustion zone (see Figure 2 for a stylized representation of a turbojet gas turbine).

When the first experimental jet engines were manufactured in the 1930s the turbine blades were manufactured using stainless steels such as Rex-78. Although these steels were relatively advanced it was known from the Brayton Cycle6 (a thermodynamic framework on the relationship between temperature, pressure, and volume) that greater fuel efficiency, and therefore engine power, would result from operating at higher temperatures.

Consequently, metallurgists worked with gas turbine engineers to create a new breed of metals capable of operating at higher temperatures. Within a decade stainless steel blades had been superseded by nickel-chrome alloys, including Nimonic 75, a 80% Ni -20% Cr alloy said to be first superalloy. By the early 1950s superalloys such as Nimonic 80 had been developed which had additions of aluminium and titanium, found to produce strengthening gamma precipitation (see inset). Additions of cobalt and molybdenum in later alloys gave higher operating temperatures and increased solid solution strengthening respectively.

In the USA, the Inconel 600 series of nickel-chromium-iron alloys was developed based on aluminium and titanium additions for gamma strengthening. Additions of niobium (Inconel X750 and 713C) produced additional strengthening by precipitating the gamma (Ni3Nb) phase.

Metallurgy #1: Gamma (γ) and Gamma Prime (γ')

By W. Parry-Jones, AMC plc, 2013

On a metallurgical level nickel-base superalloys derive their properties primarily due to the relationship between two phases; Gamma (γ) and Gamma Prime (γ'). A phase is a region that differs in structure and/or composition from an other region, and is defined by the atomic bonding and arrangement of elements in a material. For a simple alloy of metal A and metal B the phases could consist of: Pure A, Pure B, and many permutations of A+B.

When the microstructure of a nickel-base superalloy is viewed under a x13,000 electron microscope (see left) one sees a dark background which is the Gamma phase or matrix, within which the light grey Gamma prime phase precipitates are distributed.
Inconel X750 was originally used for turbine blades, but is now only used for discs. Of all the many superalloy compositions that use niobium there is one that stands out, Inconel 718, which contains 5-5.2% niobium. Since its first application in the early 1960s, 718 has become the most widely used superalloy in the world and large quantities have been and continue to be widely used in the aerospace, IGT and petrochemical industries. In 2008 SMR estimated that out of the total quantity of superalloys used by General Electric and Rolls-Royce 718 accounted for 65% and 50% respectively.

Tantalum, with its high melting point, is an effective strengthenner and also helps castability and freedom from freckling. Tantalum has been used extensively in superalloys since the late 1960s (TRW-NASA VIA superalloy).

Today superalloys employ a wide range of elements and their functions are summarised in Table 1 and Figure 3. As certain impurities can be deleterious, producers of superalloys have tight constraints on material selection. The term ‘superalloy-grade’ is often used to describe grades of rhenium and other minor metals that meet tight superalloy consumer specifications (typically levels of oxygen, O, and nitrogen, N, must be particularly low for superalloy manufacturers).

**Table 1: Elements and their function in superalloys**

<table>
<thead>
<tr>
<th>Function \ Element</th>
<th>Ni</th>
<th>Co</th>
<th>Fe</th>
<th>Cr</th>
<th>Mo</th>
<th>W</th>
<th>Re</th>
<th>Nb</th>
<th>Ti</th>
<th>Al</th>
<th>C</th>
<th>B</th>
<th>Zr</th>
<th>Hf</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Gamma prime</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Grain boundary</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carbide</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td></td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oxide scale</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>X</td>
</tr>
</tbody>
</table>

Source: Roskill after Ross & Catherall Ltd (Luigi La Braca & N. Gravili).

Tantalum and niobium are typically added to the superalloy melt as high-purity virgin or scrap metal or as revert, the latter either as foundry revert (pre-consumer scrap) or engine revert (processed end-of-life post-consumer scrap). Niobium can also be added in the form of vacuum-grade NiNb or FeNb masteralloys.

**Figure 3: Elemental effects of minor metals**

Source: Firth Rixon (elements except K), Rolls-Royce, Roskill (K')
Notes: 1 - Potassium (K) acts as a crystallisation centre.
Grains within cast superalloys

While nickel-based alloys offered a paradigm shift over stainless steels, early turbine blades were still conventionally cast, resulting in polycrystalline structure with distinct grain boundaries. The grains (crystals) that formed within conventionally cast blades are randomly shaped and, since they grew in all directions at once, they are called ‘equiaxed’. When these blades are operated at high temperature sliding at and diffusion along the grain boundaries result in creep, which over time causes the blade to elongate and risks damaging the engine. Conventionally cast turbine blades therefore had rigid operating temperature parameters.

Directionally solidified (DS) turbine blades, first developed by Pratt & Whitney, part of United Technologies Company, in the late 1960s, were a major step forward in casting technology. In DS blades columnar grains can be grown within the blade by carefully controlling the conditions of solidification after casting (see circular inserts in Figure 4). A DS blade contains no grain boundaries transverse to the stress direction, thus virtually eliminating grain boundary sliding and allowing DS blades to operate at higher temperature parameters than conventionally cast blades. The addition of rhenium to 2nd generation alloys for DS turbine blades such as PWA 1426 increased the melting temperature of the alloy further while hafnium prevented grain boundary cracking during solidification (see table 2).

The next paradigm shift, again led by Pratt & Whitney, was the development of single crystal turbine blades (SX or monocristalline) in the 1970s. In an SX turbine blade there are no crystal boundaries and so grain boundary sliding is impossible. This development negated the need for carbon, boron, zirconium and hafnium in the superalloy and allowed for higher levels of tungsten, molybdenum, tantalum and rhenium. SX turbine blades allowed engine operating temperatures to increase significantly again and by the 1990s they were applied extensively by engine designers and new 3rd, 4th and 5th generation superalloys were developed with steadily increasing percentages of rhenium.

Figure 4: The development history of superalloys and the associated casting technology

![Graph showing the development history of superalloys and associated casting technology](image)

Source: Firth Rixson, after NASA.

Note: The circular inserts illustrate the crystal boundaries found in (from left to right) conventionally cast, DS and single crystal blades.

However, with hindsight, considerable effort appears to have been spent on investing in a technological dead end; demand for rhenium in superalloys doubled between 2000 and 2008 and supply could not keep up as rhenium is a by-product of molybdenum production, itself a by-product of copper production. Despite a surge in superalloy recycling rhenium spot prices spiked between 2008 and 2012. The impact of the rhenium price spike was felt in two ways; increased use of end-of-life scrap (engine revert) and the development of low- and no-rhenium alloys.
### Table 2: A selection of superalloys with a focus on those containing niobium (Nb) and tantalum (Ta)

#### Nickel-base polycrystalline (equiaxed)

<table>
<thead>
<tr>
<th>Alloy \ Element</th>
<th>Ni</th>
<th>C</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Nb</th>
<th>Ta</th>
<th>Ti</th>
<th>Al</th>
<th>B</th>
<th>Hf</th>
<th>Re</th>
<th>other</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inconel 625</td>
<td>base</td>
<td>0.2</td>
<td>21.6</td>
<td>-</td>
<td>8.7</td>
<td>-</td>
<td>3.9</td>
<td>-</td>
<td>0.2</td>
<td>0.2</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Inconel 706</td>
<td>base</td>
<td>0.04</td>
<td>16</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>2.9</td>
<td>-</td>
<td>1.8</td>
<td>0.2</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>37 Fe</td>
</tr>
<tr>
<td>Inconel 713 C</td>
<td>base</td>
<td>0.1</td>
<td>13.5</td>
<td>-</td>
<td>4.5</td>
<td>-</td>
<td>2</td>
<td>-</td>
<td>0.8</td>
<td>6</td>
<td>0.01</td>
<td>-</td>
<td>0.06 Zr</td>
<td></td>
</tr>
<tr>
<td>Inconel 718</td>
<td>base</td>
<td>0.04</td>
<td>18</td>
<td>-</td>
<td>3</td>
<td>-</td>
<td>5.2</td>
<td>-</td>
<td>0.9</td>
<td>0.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>18.5 Fe</td>
</tr>
<tr>
<td>Inconel 738 C</td>
<td>base</td>
<td>0.17</td>
<td>16</td>
<td>8.5</td>
<td>1.7</td>
<td>2.5</td>
<td>0.8</td>
<td>1.7</td>
<td>3.5</td>
<td>3.5</td>
<td>0.01</td>
<td>-</td>
<td>0.1 Zr</td>
<td></td>
</tr>
<tr>
<td>Nimonic 75</td>
<td>base</td>
<td>0.1</td>
<td>20</td>
<td>5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.3</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.5 Cu, 5 Fe</td>
<td></td>
</tr>
<tr>
<td>Nimonic 80A</td>
<td>base</td>
<td>19.5</td>
<td>1.1</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>2.5</td>
<td>1.3</td>
<td>0.06</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Rene’ 95</td>
<td>base</td>
<td>0.15</td>
<td>14</td>
<td>8</td>
<td>3.5</td>
<td>3.5</td>
<td>3.5</td>
<td>-</td>
<td>2.5</td>
<td>3.5</td>
<td>0.01</td>
<td>-</td>
<td>0.05 Zr</td>
<td></td>
</tr>
</tbody>
</table>

#### Nickel-base directionally solidified (DS)

<table>
<thead>
<tr>
<th>Alloy \ Element</th>
<th>Ni</th>
<th>C</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Nb</th>
<th>Ta</th>
<th>Ti</th>
<th>Al</th>
<th>B</th>
<th>Hf</th>
<th>Re</th>
<th>other</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mar M 200+Hf</td>
<td>base</td>
<td>0.14</td>
<td>9</td>
<td>10</td>
<td>-</td>
<td>12</td>
<td>1</td>
<td>-</td>
<td>2</td>
<td>5</td>
<td>0.015</td>
<td>0.8-1.9</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Mar M 247</td>
<td>base</td>
<td>0.16</td>
<td>8.2</td>
<td>10</td>
<td>0.6</td>
<td>10</td>
<td>-</td>
<td>3</td>
<td>1</td>
<td>5.5</td>
<td>0.015</td>
<td>1.5</td>
<td>-</td>
<td>0.05 Zr</td>
</tr>
<tr>
<td>PWA 1426</td>
<td>base</td>
<td>0.1</td>
<td>6.5</td>
<td>12</td>
<td>2</td>
<td>6</td>
<td>-</td>
<td>4</td>
<td>-</td>
<td>6</td>
<td>0.015</td>
<td>1.5</td>
<td>3</td>
<td>0.03 Zr</td>
</tr>
<tr>
<td>Rene’ 142</td>
<td>base</td>
<td>0.12</td>
<td>6.8</td>
<td>12</td>
<td>2</td>
<td>5</td>
<td>-</td>
<td>6</td>
<td>-</td>
<td>6.2</td>
<td>0.015</td>
<td>1.5</td>
<td>3</td>
<td>0.02 Zr</td>
</tr>
</tbody>
</table>

#### Nickel-base single crystal (SX)

<table>
<thead>
<tr>
<th>Alloy \ Element</th>
<th>Ni</th>
<th>C</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Nb</th>
<th>Ta</th>
<th>Ti</th>
<th>Al</th>
<th>B</th>
<th>Hf</th>
<th>Re</th>
<th>other</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMSX-4</td>
<td>base</td>
<td>-</td>
<td>6.5</td>
<td>9.6</td>
<td>0.6</td>
<td>6.4</td>
<td>-</td>
<td>6.5</td>
<td>1.0</td>
<td>5.6</td>
<td>-</td>
<td>0.1</td>
<td>3.0</td>
<td></td>
</tr>
<tr>
<td>DD6</td>
<td>base</td>
<td>-</td>
<td>4.3</td>
<td>9.0</td>
<td>2.0</td>
<td>8.0</td>
<td>0.5</td>
<td>7.5</td>
<td>-</td>
<td>5.6</td>
<td>-</td>
<td>0.1</td>
<td>2.0</td>
<td></td>
</tr>
<tr>
<td>PWA 1480</td>
<td>base</td>
<td>-</td>
<td>10.0</td>
<td>5.0</td>
<td>-</td>
<td>4.0</td>
<td>-</td>
<td>12.0</td>
<td>1.5</td>
<td>5</td>
<td>0.003</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>PWA 1484</td>
<td>base</td>
<td>-</td>
<td>5.0</td>
<td>10.0</td>
<td>1.9</td>
<td>5.9</td>
<td>-</td>
<td>8.7</td>
<td>-</td>
<td>5.7</td>
<td>-</td>
<td>0.1</td>
<td>3.0</td>
<td>0.004 B</td>
</tr>
<tr>
<td>Rene’ N5</td>
<td>base</td>
<td>-</td>
<td>7.0</td>
<td>8.0</td>
<td>2.0</td>
<td>5.0</td>
<td>-</td>
<td>6.0</td>
<td>-</td>
<td>6.2</td>
<td>-</td>
<td>0.2</td>
<td>3.0</td>
<td></td>
</tr>
<tr>
<td>Rene’ N6</td>
<td>base</td>
<td>-</td>
<td>4.0</td>
<td>12.0</td>
<td>1.0</td>
<td>6.0</td>
<td>-</td>
<td>7.0</td>
<td>-</td>
<td>5.8</td>
<td>-</td>
<td>0.2</td>
<td>5.0</td>
<td></td>
</tr>
<tr>
<td>TMS 196</td>
<td>base</td>
<td>-</td>
<td>4.6</td>
<td>5.6</td>
<td>2.4</td>
<td>5.0</td>
<td>-</td>
<td>5.6</td>
<td>-</td>
<td>5.8</td>
<td>-</td>
<td>0.1</td>
<td>6.4</td>
<td>5 Ru</td>
</tr>
<tr>
<td>ZhS32-VI</td>
<td>base</td>
<td>-</td>
<td>4.9</td>
<td>9.3</td>
<td>1.1</td>
<td>8.7</td>
<td>1.6</td>
<td>4.0</td>
<td>-</td>
<td>6.0</td>
<td>-</td>
<td>-</td>
<td>2.0</td>
<td></td>
</tr>
</tbody>
</table>

#### Cobalt-base

<table>
<thead>
<tr>
<th>Alloy \ Element</th>
<th>Ni</th>
<th>C</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Nb</th>
<th>Ta</th>
<th>Ti</th>
<th>Al</th>
<th>B</th>
<th>Hf</th>
<th>Re</th>
<th>other</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mar-M-509</td>
<td>10.0</td>
<td>0.6</td>
<td>23.0</td>
<td>base</td>
<td>-</td>
<td>7.0</td>
<td>3.5</td>
<td>0.2</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.5 Zr</td>
</tr>
</tbody>
</table>

Source: Cannon-Muskegon (http://cannonmuskegon.com), except: Aubert & Duval (Inconel 706 and 718, Nimonic 75 and 80A alloys); All-Russian Institute of Aviation Materials (VIAM), Russia (ZhS32-VI alloy); Beijing Institute of Aeronautical Materials, China (DD6 alloy).

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### Metallurgy #2: Metal carbides in superalloys

Metal carbides (MC) form in superalloys by the combination of carbon (usually added in 0.02-0.2wt%) with reactive elements such as titanium, tantalum, hafnium, and niobium. MCs often precipitate at grain boundaries in nickel alloys, whereas in cobalt- and iron-based superalloys, intergranular sites are common. MC formation is usually considered deleterious although in nickel alloys precipitation of MCs at grain boundaries reduces grain boundary sliding, preventing creep and therefore extending service life. In cobalt-base superalloys MCs provide precipitation hardening, but decrease low-temperature ductility. With the advent of DS and SX techniques, however, grain boundaries are eliminated thus MCs are unnecessary in single crystal superalloys. This can be seen in the absence of niobium from most DS and SX superalloys.
Firstly, it stimulated a new generation of low-rhenium and no-rhenium alloys to be developed, principally led by General Electric and Cannon-Muskegon. This has had a generally positive impact on tantalum since the new alloys typically included more tantalum and other elements to make up for the reduction in rhenium. For example, CMSX-4 is a 2nd generation alloy created by Cannon-Muskegon, a PCC company, and is the most produced superalloy for SX turbine blades. CMSX-10K was developed as a 3rd generation superalloy but contained a prohibitive 6% rhenium and so CMSX-4 PLUS was developed as an alloy with properties approaching CMSX-10K, but with only 4.8% rhenium (see table 3).

<table>
<thead>
<tr>
<th>Alloy \ Element</th>
<th>Ni</th>
<th>Co</th>
<th>Cr</th>
<th>Mo</th>
<th>W</th>
<th>Re</th>
<th>Nb</th>
<th>Ta</th>
<th>Ti</th>
<th>Al</th>
<th>Hf</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMSX-4</td>
<td>base</td>
<td>9.6</td>
<td>6.5</td>
<td>0.6</td>
<td>6.4</td>
<td>3.0</td>
<td>-</td>
<td>6.5</td>
<td>1.0</td>
<td>5.6</td>
<td>0.1</td>
</tr>
<tr>
<td>CMSX-10K</td>
<td>base</td>
<td>3.0</td>
<td>2.0</td>
<td>0.4</td>
<td>5.0</td>
<td>6.0</td>
<td>0.1</td>
<td>8.0</td>
<td>0.2</td>
<td>5.7</td>
<td>0.03</td>
</tr>
<tr>
<td>CMSX-4 PLUS</td>
<td>base</td>
<td>10.0</td>
<td>3.5</td>
<td>0.6</td>
<td>6.0</td>
<td>4.8</td>
<td>-</td>
<td>8.0</td>
<td>0.85</td>
<td>5.7</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Source: Cannon-Muskegon Corporation

Secondly, there was a surge in the processing and use of engine reverté a high-quality post-consumer scrap produced from end-of-life gas turbine parts. Once scrapped engine parts have been checked for chemical uniformity, cleaned of their zirconia- or alumina-based heat-resistant coating and shot-blasted, one is left with pieces of 100% homogenous superalloy metal ready to be remelted. Estimates vary, but industry sources claim that in 2015 engine revert supplied around 20% of all superalloy feedstock.

These two recent trends will inevitably have had an impact on demand for tantalum and niobium, but the most significant trend in the superalloy market is the rise in production of gas turbines for aero-engines.

End-of-life superalloy scrap that has been cleaned and is ready for reuse as engine revert. IGT turbine blades (left) of IN738 alloy (1.75% Ta, 0.8% Nb) and aerospace compressor rings (right) of 718 alloy (5.2% Nb, no Ta) (Photos T.I.C.).

**Outlook for superalloy demand**

The high temperature capabilities of single crystal superalloys are not currently substitutable in modern aero engines, although in the longer-term ceramics may become feasible alternatives. Demand for new aero engines tracks that for new aircraft and the outlook for this industry is for strong growth for the foreseeable future; Boeing’s 2015 Market Outlook estimates long term growth at ~5% per year through to 2034 while Airline Monitor is more optimistic still (see Figure 5).

What is more certain is that in the foreseeable future several engine programmes are expected to reach full production simultaneously. These include CFM’s LEAP engine (formerly called LEAP-X), Pratt and Whitney’s PW1100G Geared Turbofan engine, Rolls-Royce’s Trent XWB and 1000 engines, and several military programmes too.
The impact of so many new aircraft and aero-engines will on balance see an increase in demand for tantalum and niobium. Roskill expects tantalum demand from the superalloy sector to grow at 8% per year through 2020, a higher rate than for other sectors of tantalum demand. However, what proportion will be sourced from engine revert and what from high-purity virgin metal or scrap is almost impossible to predict given the opacity of the market. To further complicate any forecast General Electric is spending billions of dollars in developing ceramic matrix composite gas-turbine parts (CMCs) which may become substitutes for superalloys, even though this is unlikely to have a significant impact on superalloy consumption before at least 2020.

One further influence on the future of superalloy consumption will be additive layer manufacturing (ALM). ALM is in general the opposite of traditional subtractive manufacturing, where material is removed from a crudely cast shape to reach the final desired dimensions. In ALM parts are built up in successive layers of material under computer control. At first ALM was largely for rapid prototyping but manufacturers, especially General Electric, have realized the endless capabilities of this new fabrication process and as production of parts switches to ALM there will be an increase in superalloy powder production and a significant decrease in pre-consumer scrap (foundry revert).

Consumption of superalloys in industrial gas turbines and other non-aerospace applications is probably more closely linked to GDP growth levels and market sources suggest growth in this market to be in the region of 2-3%.

For further information on how superalloy trends will influence niobium and tantalum supply and demand in the foreseeable future please contact T.I.C. member Roskill Information Services.

Roskill Information Services has released its new tantalum market report with forecasts out to 2020. It is essential reading for anyone needing a comprehensive overview of this rapidly evolving industry.


For further information, contact us on:
Tel: +44 20 8417 0087. Fax: +44 20 8417 1308.
Email: info@roskill.com Web: www.roskill.com
Applications and properties of Metalysis FFC-produced spherical tantalum powder

Paper written by Ian Margerison & Dr Ian Mellor and presented by Ian Margerison on October 27th 2015, as part of the Fifty-sixth General Assembly held in Penang, Malaysia.

Metalysis Ltd, Unit 2, Farfield Park, Manvers Way, Wath Upon Deame, Rotherham S63 5DB, United Kingdom
email ian.margerison@metalysis.com, www: http://www.metalysis.com

Abstract

The presentation will demonstrate the applications of Metalysis FFC tantalum spherical powder with regards to selective laser melting (SLM) manufacturing. SLM is an additive manufacturing process by which a layer of tantalum powder is deposited onto a substrate and is spread uniformly by a wiper and then a high power density fibre laser fully melts the pre-deposited powder layer according to a specific computer generated two dimensional pattern. The melted particles fuse and solidify to form a layer of the tantalum component. The part retracts vertically and powder is deposited on top, the process is repeated until the three dimensional tantalum part is completed.

The Cambridge FFC Process

The Cambridge FCC process was developed by Prof Derek Fray, Dr Tom Farthing and Dr George Chen at Cambridge University in the UK. At its essence the process starts when metal oxide powder is pressed into pellets. The pellets are reduced in a molten salt bath by electrolytic process, before being extracted from the solution as a metal sponge. The sponge is milled to the required size particles before being washed and dried. The electrolyte is calcium chloride, a low toxicity salt used to melt road ice in cold climates.

Figure 1: The Metalysis FFC Process
The technology has wide applicability and is suitable and attractive for a wide range of metals with stable oxides. By October 2015 the Cambridge FFC process has been demonstrated on 14 pure elements, although the company is currently focusing on titanium and tantalum. Metalysis’ FCC process is also capable of producing metal powder from pure oxides and is also capable of co-reducing mixed oxides to form alloys. By real-time monitoring of the flue gasses emitted from the reduction cell it is possible to know precisely when the process has peaked and remove the reduced product for post processing.

![Figure 2: Metalysis FFC Reduction Cell](image)

**Metalysis FFC tantalum powders for additive manufacturing**

The technology is currently designed to produce capacitor- and metallurgical-grade (dinet) tantalum powders. The dendritic-shaped powder is used as feedstock to create spherical powders.

![Figure 3: FFC Met Grade Tantalum Powder](image)
There are several techniques used to generate spherical powders and Metalysis chose a technique that gave the best parameters for the tantalum feedstock. When powder is used in additive manufacturing it is critical that the properties of the powder are such that no satellites form (particles are not welded together) since these will result in unheterogenous melting characteristics during the additive manufacturing process.

A key advantage of using spherical powder in additive manufacturing is the high flowability of the powder that will give high uniformity of layers as the additive manufacturing process takes place. Thus leading to a high density part (99.9%).

The particle size distribution of the spherical powder is correlated to the particle size of the metallurgical-grade powder that is used as feedstock. The spherical powder is a high quality material and the apparent density and tap density are important considerations in additive manufacturing.
Additive manufacturing build trials at TWI

Metalysis has worked closely with The Welding Institute (TWI, http://www.theweldinginstitute.com) to develop a body of knowledge concerning the use of tantalum in additive manufacturing.

TWI used a selective laser melting (SLM) machine called the Realizer SLM 100 which possesses the following characteristics:

- Build volume: 125(x) * 125(y) * 180(z) mm
- 200W fibre laser
- 20-100 μm layer thickness
- 20 μm diameter beam spot
- Pre-heating capability
- Inert atmosphere (Argon)

The Realizer SLM 100 has a base of around 5kg and is commonly used to construct medical components, a critically important market for tantalum additive manufacturing.

During the SLM process a layer of tantalum powder is deposited onto a substrate and is spread uniformly by a wiper and then a high power density fibre laser fully melts the pre-deposited powder layer according to a specific computer generated two dimensional pattern.

The melted particles fuse and solidify to form a layer of the tantalum component. The part retracts vertically and powder is deposited on top, the process is repeated until the three dimensional tantalum part is completed.
The first iteration trial explored the optimum conditions for tantalum as a feedstock and created hollow cubes with internal bracing. The goal was to find the right laser parameters for the optimum build.

![Image](image_url)

**Figure 7: AM 1st Iteration Trial**

The second iteration trial focused on creating solid blocks of tantalum metal.

![Image](image_url)

**Figure 8: AM 2nd Iteration Trial**

Analysis of a tantalum block made by the Realizer SLM 100 revealed 99.9% dense parameters.

A third iteration of tests was to build a 7mm tantalum lattice build to demonstrate the potential for complex geometrical shapes constructed by SLM using tantalum powders.
Tantalum parts manufactured by additive manufacturing offer several advantages, not least in allowing tantalum to be used in a highly selective and cost-efficient way. This is particularly relevant to products for the nuclear industry where historically tantalum has often been overlooked due to its relatively high cost and perceived difficulties in obtaining material.

Summary
Spherical powders offer additive manufacturing considerable advantages over dendritic powders, most notably by forming highly uniform layers which is advantageous in the additive manufacturing process. There are very few organisations that are capable of producing spherical tantalum powders that are appropriate and an advantage of Metalysis process is that producing spherical powder using plasma methods acts as an additional purification process.

Metalysis owns the commercial rights to the FFC Process and is working with a leading additive manufacturing company, to commercialise the use of spherical tantalum powders in additive manufacturing. The aims of this partnership are as follows:

- The production of spherical particles without satellites
- The process allows control and purification of the chemistry
- The spheroidisation process can be applied to Metalysis FFC produced alloys
- Applied to additive manufacturing this technology provides:
  - Fully spherical particles (10-45 m and 45-100 m) depending on feedstock PSD
  - Increase in flowability
  - A reduction in Oxygen levels
  - Increased packing density

For more information please contact Ian Margerison at ian.margerison@metalysis.com.
Member company updates

Changes in member contact details

AS International Corporation Ltd (formerly A.S. Metallurgy (Liverpool) Ltd) has relocated to Unit 2b Olympic Way, Sefton Business Park, Aintree, Liverpool L30 1RD, United Kingdom.

Avon Specialty Metals Ltd has relocated to Empire Way, Gloucester, GL2 5HY, United Kingdom.


CBMM: The new representative at CBMM has been confirmed as Mr Yuri Bugarin Woiski Miranda. To contact Mr Miranda telephone +55 (11) 3371-9222 or e-mail yuri.miranda@cbmm.com.br.

Duoluoshan Sapphire Rare Metal Co. Ltd of Zhaoqing has a new website www.zqdlis.com.

ECO White Comercio de Sucatas Ltda has relocated its office to Rua Barretos, 1191, Vila Elisa i Ribeirão Preto, SP, Zip Code: 14075-000, Brazil. The telephone lines are +55 16 3626-4034 and +55 16 3626-2921.

Global Advanced Metals has changed its nominated address to 880 Winter Street, Waltham, MA 02451, United States. All other contact details remain the same.

Mineração Taboca S.A. has changed its designated contact address to Vila Pitinga s/n, Zona Rural, Presidente Figueiredo, 69735-000, AM, Brazil.

Molycorp Silmet has changed its name to NPM Silmet AS. The representative, Mr David O'Brien, has a new email address, d.obrock@neomaterials.com, and the company's new website is www.neomaterials.com.

NAC Kazatomprom JSC: Mr Talgat Nurtazayev has become the contact person for NAC Kazatomprom in the place of Mr Anel Mendigali. Mr Nurtazayev can be contacted on nurtazayev@kazatomprom.kz. The delegate remains Mr Askar Zhumagaliev.

Roskill Information Services Ltd: Patrick Stratton has a new email address patrick@roskill.com.

Shalina Resources Limited: Mr Uday Shetty is the new nominated delegate to the T.I.C. in place of Mr Abbas Virji. Mr Shetty can be contacted on uday.shetty@shalina.com.

Tantaline has a new contact email address sales@tantaline.com.

Members of the Executive Committee of the T.I.C. 2015-2016

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

Conor Broughton
John Crawley
David Gussack
Dale Gwinnutt
David Henderson (President)
Marc Hjelle
Jiang Bin
William Millman
David O'Brien
Candida Owens
Daniel Persico
Alexey Tsrayev
conor@amgroup.uk.com
jcrawley@mmc.com.hk
david@exotech.com
dalegwinnutt@elitematerial.com
dhenderson@rittenhouseir.com
marc.hueppeler@hcstarck.com
jiangb_nniec@otic.com.cn
bill.millman@avx.com
d.obrock@neomaterials.com
candida.owens@btinternet.com
danielpersico-rc@ne-cotkin.com
tsrayevaa@ulba.kz

On Monday October 17th 2016 at the General Assembly of the T.I.C. in Toulouse, France, there will be elections to select the members of the Executive Committee that will stand for the period until the next General Assembly in October 2017. This year there are 15 nominations for the 12 places permitted by our Charter, so a secret ballot will be held to determine who is elected. The results of the election will be reported in Bulletin #168 (January 2017).
And finally… new light on niobium

Brazilian designer Claudia Moreira Salles has created a collection of limited-edition niobium lamps for her solo design exhibition Fine Tuning at Espasso in New York City, NY, United States. Fashioned using niobium supported by copper and reclaimed wood, Ms Salles’ new fixtures are billed as niobium’s first use in designer lighting fixtures.

Ms Salles said that niobium presented uncharted territory for her, not least in the methods for achieving the desired colours. In order to achieve the [lamps] distinct spectrum of colours, we had to develop a very complex process, including submerging the niobium in water and acid and then applying an electric current to it, she recalled.

The line of limited-edition lamps is called Ñintonia Fina” and for further information visit http://www.espasso.com. Ms Salles graduated in 1978 at the Escola Superior de Desenho Industrial (ESDI) in Rio de Janeiro, Brazil. A video showing how the lamps were created is at https://youtu.be/wH4cQMKBH7k

Photos are by Andres Otero for the book: "Sintonia Fina, Luminárias/lamps" by Claudia Moreira Salles.

…but still in the dark about tantalum-180m

Deep underground in northern Belgium an experiment has ended that ran for six months but failed to record any activity at all in its test sample of tantalum.

Tantalum naturally occurs in the Earth’s crust in two stable isotopes, $^{181}$Ta (99.99%) and $^{180}$Ta (0.01%), and the experiment attempted to measure the decay rate of tantalum-180m. The "m" denotes that the isotope is a metastable state, which means that its nucleons exist in an excited state. Tantalum-180m is also exceptional because it has the longest known lifetime of a metastable nucleus and it was this that attracted the team at the HADES laboratory. HADES is located 225m under the town of Mol, near Antwerp, and is named after the god of the underworld in ancient Greek mythology.

The lifetime of tantalum-180m is so long that no decays have ever been recorded. The latest attempt, led by Björn Lehnert and his colleagues at Technical University Dresden in Germany and the Joint Research Centre in Geel, Belgium, placed samples of high-purity tantalum with natural isotopic abundances in the Sandwich detector system at HADES for 176 days of continuous observation and measurement. Sandwich comprises two high-purity germanium radiation detectors. However, at the end of the experiment the team reported that they had recorded zero evidence for the decay of tantalum-180m.

However, in science even no result is a result and following the HADES-Sandwich experiment the lower limit on the half-life of the isotope has revised to 4.5x10$^{16}$ years. The research is described in a preprint on arXiv at http://arxiv.org/abs/1609.03725.

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